

# **Review of Durability Test Methods and Standards for Assessing Long Term Performance of Adhesive Joints**

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## **ABSTRACT**

This report provides a critical appraisal of test methods and related standards used for the assessment of durability of adhesive joints under combined cyclic or creep loading and hostile environments. Accelerated ageing methods/schemes and durability data for design requirements are included in the assessment. The test methods are assessed in terms of material compatibility, data generated, environmental and service conditions, long term performance, costs of implementation, ease of use, consistency of data, data reduction requirements and stress uniformity. Consideration is given to the practicality of using the test method in an industrial environment, in terms of ensuring "fitness for purpose". The review is based on information compiled from previous government funded research programmes, a survey of industrial practices and requirements, and general literature.

The results of the review will contribute to the formulation of a subsequent experimental programme, assisting in identifying test methods that can be used to measure the parameters required for long-term performance predictions and enable a prioritised listing of service conditions. These test methods and environmental conditioning procedures are to be developed and validated in the experimental programme.

The report was prepared as part of the research undertaken for the Department of Trade and Industry funded project on "Performance of Adhesive Joints - Combined Cyclic Loading and Hostile Environments".

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Approved on behalf of Managing Director, NPL, by Dr M K Hossain,  
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## 1. INTRODUCTION

There is a general consensus within UK industry that the lack of reliable test methods and accelerated ageing procedures, quantitative data and predictive analysis for determining the durability of structural adhesive joints subjected to the combination of applied stresses and hostile environments has impeded the wider use of adhesive technology. This applies for both metals and fibre-reinforced plastics, particularly with regard to long-term strength retention and life prediction.

The current status for testing and environmental conditioning of adhesive joints is less than satisfactory with little or no evidence of harmonisation between existing methods. There is a tendency for many manufacturers to develop and work to their own in-house test procedures. This may be due to a lack of knowledge about appropriate standards, or because the durability standard fails to represent actual service conditions. The major disadvantage with many tests is that the test piece is conditioned unstressed in the required environment and then tested. Failure to account for the combined effect of stress and environment will frequently result in an overestimation in strength and fatigue life. Standard methods, therefore, need to have the widest possible applicability and be capable of representing actual service conditions.

One of the main objectives of the Department of Trade and Industry funded project on "Performance of Adhesive Joints - Combined Cyclic Loading and Hostile Environments" is to develop and validate test methods and environmental conditioning procedures that can be used to measure the parameters required for long-term performance predictions. This project is one of three technical projects forming the programme on "Performance of Adhesive Joints - A Programme in Support of Test Methods". The programme is targeted at supporting the infrastructural needs of UK industry for the development, validation and standardisation of test methods to measure and predict the performance of adhesive joints. The new programme is a direct continuation from the previous programme, extending to new materials and test methods. To meet this objective it was necessary to establish the current status of durability test methods, associated standards and data generated.

This report provides a critical appraisal of test methods and related standards used for the assessment of durability of adhesive joints under combined cyclic or creep loading and hostile environments. Accelerated ageing methods/schemes and durability data requirements for design requirements are included in the assessment. The review is based on information compiled from previous government funded research programmes, a survey of industrial practices and requirements, and general literature.

The test methods are assessed in terms of material compatibility, data generated, environmental and service conditions, long term performance, costs of implementation, ease of use, consistency of data, data reduction requirements and stress uniformity. Consideration is given to the practicality of using the test method in an industrial environment, in terms of ensuring "fitness for purpose". The results of the review will provide the basis for the subsequent experimental programme directed towards the development and validation of mechanical and environmental test methods for quantitative measurements required for long term performance prediction.

## 2. SCOPE OF REVIEW

The main requirement of the review is to identify test methods and environmental conditioning procedures that can be used to measure the parameters required for long-term performance predictions. The review provides:

- (i) A critical assessment of available tensile, compressive, shear, peel and fracture toughness test methods and corresponding standards (ie national and international), to determine suitability of use under combined cyclic or creep loading and hostile environments.
- (ii) A compilation of environmental conditioning and accelerated ageing procedures, and corresponding standards.

Each mechanical test method is examined in terms of:

- (a) Suitability to various adherends and adhesive systems.
- (b) Properties obtained.
- (c) Test specimen geometry, size and alignment.
- (d) Test specimen preparation (including machining and loading attachments).
- (e) Fixture and testing equipment requirements.
- (f) Strain measurement techniques.
- (g) Problems associated with load introduction and free edges.
- (h) Test data analysis and accuracy.
- (i) Failure criteria, failure modes and associated problems.
- (j) Level of standardisation.

Particular consideration is given to the practicality of using the test method in an industrial environment taking into account costs, and applicability of the test method for fatigue and environmental testing. Several additional issues have been considered, including the degree of stress uniformity in the adhesive layer, non-linear behaviour and problems associated with manufacture (eg residual stresses).

The report is divided into five sections (including the Introduction and Scope of Review (Section 2)). Section 3 covers bulk and adhesive joint test methods (ie tension, peel, cleavage, shear and fracture toughness). The results of the review of test methods are summarised at the end of this section, and in Tables 1 to 4. Section 4 covers environmental conditioning and accelerated ageing procedures. Conclusions are given in Section 5. Appendix I provides a listing of durability standards and Appendix II describes the content of standards relating to environmental conditioning.

### 3. TEST METHODS

Stress analysis of adhesive joints requires a database of basic engineering properties of the adhesive, adherend and the joint geometry. Properties required are listed below [1]:

- Adhesive shear modulus
- Adhesive elastic tensile or compressive modulus
- Effective adhesive transverse modulus
- Characteristic adhesive shear strength
- Characteristic adhesive tensile or compressive strength
- Adhesive and adherend elastic/plastic shear stress and strain
- Adherend tensile or compressive modulus
- Adherend Poisson's ratio
- Characteristic adherend through-thickness tensile strength
- Adhesive layer thickness
- Thickness of adherends
- Joint efficiency (ie ratio of joint strength to adherend strength)

Numerous test methods exist for characterising adhesives and bonded joints, and may be used to determine fatigue resistance, environmental durability and stress relaxation resistance. Adhesive tests can be divided into those methods that provide mechanical property data for the adhesive, which aids the selection of adhesives, and those methods which can be used to determine the quality of adhesively bonded structures, and thus aid the design process of adhesive joints. Although an extensive range of test methods is available as national and international standards, most of these tests can only be used for qualitative measurements, providing a means of checking the effectiveness of different surface preparations and comparing mechanical properties of different adhesive systems (ie ranking adhesive formulations). A limited number of test methods are suitable for generating engineering data, particularly for determining structural integrity of adhesively bonded structures subjected to static, cyclic and environmental effects. A list of standards issued by the American Society for Testing and Materials (ASTM), British Standards Institution (BS series) and International Standards Organisation (ISO) is presented in Appendix I.

Most of the commonly used test methods are incapable of providing reliable engineering data because the test geometry induces a complex state of stress in the adhesive layer, thus invalidating the results. Two approaches have been adopted in order to overcome this problem. The first and direct approach is to measure the properties of bulk adhesive specimens. Bulk adhesive specimens are cast and machined to the required shape (eg dumbbell tensile specimens). Many liquid adhesives can be easily cast into bulk specimens without the need for machining. To minimise the deleterious effect of surface scratches which may cause premature failure, the edges and faces of the specimen are carefully polished to remove any surface defects.

Although bulk adhesive specimens are relatively straightforward to test, there are a number of problems associated with casting bulk specimens. For example, exothermic reactions can occur when casting thermoset materials, resulting in material degradation through overheating. The problem is exacerbated with increasing thickness. Porosity, in the form of entrapped air and volatiles, is a common cause of premature failure. In many cases it is

virtually impossible to produce void free specimens, particularly for materials with a high viscosity. In addition, residual thermal stresses may be generated, as a result of non-uniform (rapid) cooling. Residual stresses, which are typically compressive on the surface and tensile in the interior, are frozen into the material. This is an undesirable situation, as tensional strain at the surface enhances environmental stress cracking.

The second approach for determining engineering properties of adhesives is to use specially designed joint geometries with a thin bond line, often referred to as *in-situ* testing. In order for these test geometries to produce reliable engineering data, the test geometry should provide a pure state of stress, uniformly distributed across the contact surface and through the bond line, free of stress concentrations. The surface treatment should be sufficient to ensure cohesive failure in the adhesive layer. Ideally, the test method should employ simple and easily prepared specimens. Failure force should in principle remain constant with fluctuations in the failure force being attributed to variations in adhesive strength.

There are a number of problems associated with adhesive joint configurations. These are listed below:

- a) The stress distribution within the bondline tends to be non-uniform in a majority of test joint configurations with stress concentrations existing at the bondline ends. Premature failure will often occur as a result of these stress concentrations.
- b) Generally, the time taken for environmental effects to become apparent increases with joint size, thus test joints with small bonded areas, or with large bondline perimeters compared with the bonded area are preferred. Evidence suggests that small joints exposed to water may initially exhibit a short-term increase in joint strength, due to plasticisation of the bondline perimeter, reducing the stiffness and relaxing internal stresses. In the longer term, joint strength diminishes.
- c) The accuracy and reliability of displacement measurements are often in question, as the magnitude of displacements is often small.

The four main loading modes of bonded joints are?[1] (see also Figure 1):

- Peel loads produced by out-of-plane loads acting on thin adherends.
- Shear stresses produced by tensile, torsional or pure shear loads imposed on adherends.
- Tensile stresses produced by out-of-plane tensile loads.
- Cleavage loads produced by out-of-plane tensile loads acting on stiff and thick adherends at the end of the joints.

NB. Bonded joints are subjected to simultaneous combinations of all four loading modes.

### 3.1 TENSILE TEST METHODS

Transfer of load to an adhesively bonded structure by tension, either directly or indirectly (peel), represents the most severe form of loading, since the strength of the joint relies on the tensile strength of the adhesive, which is low. It is good design practice, therefore, to ensure that the load is transferred by shear or compression and that direct or induced tensile stresses are minimised. Tensile stresses are virtually impossible to avoid, however there is a need for reliable test methods to measure the tensile properties of the adhesive.

This section examines the tensile butt joint, which is used to evaluate the tensile properties of adhesive joints (see also Table 1). Test methods that can be used to measure the static, fatigue and creep behaviour of engineering adhesives, in bulk form, will not be covered in this section. The reader should refer to the ISO standards relating to the testing of polymeric materials [2, 3].

### 3.1.1 Tensile Butt Joint

The cylindrical butt joint shown in Figure 2 is commonly used to test thin bondline specimens in tension, torsion and compression. Adhesive suppliers use tensile butt joint tests to provide comparative properties on different adhesives. The test provides data on the moduli of rigidity and elasticity, and Poisson's ratio. The average strength is taken as the applied load at failure divided by the bond area. Variations of this test configuration have been included in ASTM D 897 [4] and ASTM D 2095 [5], and in BS EN 26922 (ISO 6922) [6]. Specimens may be designed with either a circular or square cross-section. Circular cross-section specimens are usually used to facilitate manufacture and maintain symmetry. Both circular and square cross-sections, 10 to 50 mm wide, are accommodated within BS EN 26922.

In a tensile butt joint, the adhesive is restrained in the radial and circumferential directions by the adherends. In the absence of this restraint, the adhesive is free to undergo radial contraction with respect to the stiffer adherend. The presence of the adherends has the effect of inducing radial and circumferential stresses, thereby increasing the effective stiffness of the joint. The apparent Young's modulus  $E^*$  is given by [7]:

$$E^* = \frac{E(1-\nu)}{(1+\nu)(1-2\nu)} \quad (1)$$

where ( $E$ ) is the Young's modulus and ( $\nu$ ) is the Poisson's ratio of the adhesive. This analysis assumes that the adhesive has a much lower stiffness than the adherends (ie zero contraction of adherends).

Numerical analysis [7, 8] of butt joints in tension has revealed that the tensile stresses in the central region of the adhesive layer are uniformly distributed and there are no shear stresses present. In marked contrast, the stress state in the outer edges is highly complex, varying through the thickness of the adhesive layer. The stress state at the overlap edges (ie adhesive-adherend interface) is complicated by through-thickness shear stresses and the presence of stress singularities. Large, indefinable, stress concentrations at the periphery of the adhesive layer can be expected to render the strength data meaningless. Their removal can be facilitated by rounding the adherend butt edges to a diameter equal to the adhesive thickness. This effectively increases the bondline thickness at the outer edges of the bondline, and as a result non-linear stress effects may result, especially at elevated temperatures and exposure to high humidity environments [7, 8].

Significant bending can be induced due to misalignment of the adherends, resulting in premature failure. Care needs to be taken to ensure good alignment during specimen preparation (ie bonding of adherends) and testing. Small misalignment can severely reduce strength data. A further consideration, relates to the removal of the spew fillet (ie excessive adhesive) around the outer edges of the joint. The spew is able to transmit some of the applied load, resulting in a significant reduction in the level of axial stresses within the adhesive joint, and thus contributing to the uncertainty in moduli and strength data.

**It is difficult to envisage using the butt joint for assessing long-term performance of**

**adhesive joints under combined cyclic and hostile environments, considering the difficulties associated with this configuration (inc. monitoring of strain, which is relatively small).** Tensile strength values based on average stress measurements (ie failure load divided by the bond area) are often misleading. Hence, there is little evidence to support the recommendation of this test procedure for either static or cyclic loading conditions.

### 3.2 PEEL TESTS

Since the inception of structural bonding, peel tests have been an integral part of adhesive performance specifications, and have played an important role in the development of adhesives [7, 9]. The emphasis has been on the development of higher peel strengths, often at the expense of environmental resistance. Peel resistance is defined as the average force per unit test specimen width, measured along the bond line that is required to separate progressively two adherend members of a bonded joint. The incessant demand for higher peel stresses has provided an impetus for the development of suitable peel tests. Engineers and designers now have a wide selection of peel tests and associated standards at their disposal (see Appendix 1).

Peel stresses arise if one or both adherends are flexible, and hence the test methods are suitable for use in determining peel resistance of thin metallic adherends (eg thin aluminium alloy sheet). The popularity of these methods can be attributed to ease of use and a physical resemblance to actual in-service debonding problems. Specimen fabrication, testing and data reduction are relatively straightforward. Adhesion is measured by peeling a thin adhering layer from a rigid substrate or from an identical layer. The peel force is a measure of fracture energy, although the data generated has little use in the stress analysis of a bonded joint [8]. Peel tests are normally used to compare adhesives.

Peel strength has been observed to increase with both adherend and adhesive thickness, but decreases with increasing adhesive stiffness [8]. Steel adherends are known to give higher peel strengths than aluminium adherends for identical thicknesses. This is because the higher modulus material is less likely to deform out-of-plane, thus reducing peel stresses. A major disadvantage of peel tests is that excessive extension of flexible adherends contributes to failure at the adhesive/adherend interface.

In this section, three popular peel tests will be described (see also Table 1). These are listed below:

- T-peel
- Climbing drum peel
- Floating roller method

#### 3.2.1 T-Peel Test

The primary purpose of this test method is to determine the relative peel resistance of adhesive bonds between flexible adherends by means of a T-type specimen (Figure 3). The term flexible refers to the ability of the adherend to bend through 90° without breaking or cracking. This test geometry has been adopted by most standards bodies and is widely used by industry to evaluate environmental durability of adhesively bonded systems [10-12]. The T-peel test is particularly suited to testing thin aluminium alloys (eg 2024-T3 or 5251). Specimens are typically 25 mm wide, have a minimum bonded length of 150 mm, and 50 mm long arms [11]. The recommended thickness is 0.5 mm for steel and 0.7 mm for aluminium. Adhesive layer thickness is not specified. The cost of producing T-peel

specimens is relatively low.

Testing is straightforward and requires no special fixture. The specimen can be readily loaded using standard tension/compression mechanical test equipment. It is important to ensure that the bonded portion of the specimen remains perpendicular to the applied load. The specimen is bent backwards 180° and peeled. Tests are normally conducted at displacement rates of 100 mm/min for metals and 10 mm/min for other adherends. The average peeling load per unit length/width is used to define the peel strength. This is taken from the force-displacement curve after the initial peak reading. At least five to ten specimens should be tested from each batch of material, depending on the standard used. T-peel tests are only suitable for use with thin flexible adherends. Adherends are generally thick enough to detach without undergoing significant deformation (ie stretching and bending). The analysis should account for the possibility of adherend deformation. The test has been shown to discriminate between various combinations of pretreatment and adhesives, although coefficients of variation are typically 20 to 30%, or higher [13]. Correlation between service and laboratory data has proved to be reasonably reliable, although qualitative. Unstable behaviour can cause difficulties in interpretation of test data and in ranking of environmental resistance.

### 3.2.2 Climbing Drum Peel Test

The climbing drum peel test was developed to determine the peel resistance of adhesive bonds between [14, 15]:

- a) A relatively flexible adherend and a rigid adherend; and
- b) The relatively flexible facing (or skin) of a sandwich structure and its core (Figure 4).

The test consists of peeling a thin strip of metal from a thick strip by winding the thin strip around a drum [6]. Torque is applied to the drum by pulling down on straps wrapped around the drum. The thin strip of metal is wound on a the drum at a smaller radius than the straps. The difference in radius (ie moment arm) results in a large torque being applied to the drum compared with that applied on the thin strip. The resultant upward motion causes the thin strip to peel from the thicker strip. The peel torque,  $T$ , can be calculated using [15]:

$$T = \frac{[(R_0 - R_1)(F_p - F_0)]}{b} \quad (2)$$

where ( $R_0$ ) is the flange radius, ( $R_1$ ) is the flange radius, ( $F_p$ ) is the load required to bend and peel adherend (inc. load required to overcome the resistance of the drum), ( $F_0$ ) is the load required to bend adherend (inc. load required to overcome the resistance of the drum) and ( $b$ ) is the specimen width. Both  $R_0$  and  $R_1$  account for one half the loading strap thickness. Additional tests are required to determine the resisting torque of the drum, clamp and counterweight, and to compensate for the bending of the adherend.

The upward motion of the drum causes the thin strip to be peeled from the thicker adherend, thus resulting in bond failure. This test is often used for qualifying skin-to-core bond strength of sandwich structures, and is particularly sensitive to adherend surface preparation. This method provides comparative data of adhesion and is particularly suited to process control. Direct comparison of different adhesive systems and processes can only be made when the specimen configuration and test conditions are identical [9].

The climbing drum peel test is applicable to the testing of joints between an outer metal

sheet and the core of sandwich assemblies (eg honeycomb). The test specimen for a sheet metal assembly consists of two 25 mm wide adherends, one 300 mm long and the other 240 mm long [14]. The longer adherend has a 30 mm tail at either end. BSI recommend that the thickness for the longer adherend and shorter adherend be 0.5 mm and  $\geq 5$  mm, respectively. The test specimen for honeycomb structures should be 75 mm wide. The skins (facings or adherends) of the sandwich structure should be 0.5 mm thick, and the core separating the skins should be at least 12 mm thick. The adherend lengths should be 300 mm and 240 mm. When testing laminated assemblies, the test specimens are typically 25 mm wide. The short adherend should be 240 mm long and the long adherend at least 254 mm long [15]. BSI recommends that sandwich structures be 75 mm wide and 300 mm long, including a 25 mm overhang of one skin at each end of the specimen (Figure 4).

It is important to ensure that the core thickness/stiffness is high enough to prevent bending of the sandwich specimen. For aluminium alloy 2024-T3, an adherend thickness of 0.51 mm and core thickness of 12.7 mm has been found to produce satisfactory results [14]. Tests are conducted at 25 mm/min.

Specimen preparation and testing is relatively straightforward, although the cost of specimen preparation can be expensive, particularly for honeycomb sandwich structures. A special fixture is required, which can be purchased from a test machine manufacturer or produced in-house, but at a substantial cost. Commercial fixtures are available. **The climbing drum test is not particularly suited to operating in hostile environments, and is definitely incompatible with cyclic loading conditions.**

### 3.2.3 Floating Roller Method

The floating roller peel test has been developed to determine the relative peel strength of adhesive bonds between one rigid adherend and one flexible adherend. This test method is considered suitable for acceptance and process control testing. The asymmetric specimen is typically 25 mm wide and consists of a 250 mm long flexible adherend bonded to a 200 mm long rigid adherend. The unbonded end of the flexible adherend is bent perpendicular to the rigid adherend and clamped in the grip of the testing machine. The applied load peels the flexible adherend from the rigid substrate. A schematic of the floating roller drum loading configuration is shown in Figure 5. ASTM D 3167 [16] specifies 0.63 mm thick flexible adherend and 1.63 mm thick rigid adherend, whereas the British Standards Institution (BSI) [17] recommends corresponding values of 0.5 mm and 1.6 mm.

Tests are normally conducted at displacement rates of 100 mm/min or higher. As with the T-peel test, the average peeling load per unit area is used to define the peel strength. This is taken from the force-displacement curve after the initial peak reading. Failure analysis does not allow for deformation of the flexible adherend. It is recommended that at least five specimens be tested from each batch of material [17].

The BSI standard [17] was developed to assess pretreatments for aluminium, which could differentiate between pickling pretreatments, complete chromic acid anodisation (CAA)/phosphoric acid anodisation (PAA) and incomplete CAA/PAA processes. In the wet peel test, several drops of water containing a wetting agent (eg 0.5 to 1.0% detergent) is applied to the crack opening after peeling 75 mm of the bonded length. The introduction of the liquid changes the mode of failure from cohesive (dry joint) to quasi-adhesive failure. **The test is not suitable for long-term durability prediction.**

Specimen preparation and testing is relatively straightforward. Associated costs of specimen fabrication and preparation can be considered reasonable. A relatively high cost fixture is required to test specimens. Commercial fixtures are available. The floating roller test is considered the more reliable method of the three techniques. **The floating roller**

**method is not particularly suited to operating in hostile environments, and is definitely incompatible with cyclic loading conditions.**

### 3.3 CLEAVAGE TESTS

This section will examine loading configurations that measure adhesive resistance to cleavage (see also Table 2). Mode I (crack opening) fracture toughness test methods, such as the double cantilever beam (DCB) and the tapered DCB, will be discussed in Section 3.5.

#### 3.3.1 Wedge Cleavage Test

Cleavage stresses (essentially concentrated tensile stresses applied perpendicular to the bondline) represent the most severe form of loading that can be experienced by a bonded joint. The Wedge cleavage test [13, 18], often referred to as the Boeing wedge test, was developed in order to simulate, although in qualitative manner, the forces and effects of cleavage stresses in an adhesively bonded joint at metal-adhesive/primer interface (Figure 6). **This test method is widely used throughout the aerospace/defence industry and has proved highly reliable in determining and predicting the environmental durability of adherend surface preparations** [19]. Although, the test was developed specifically for aluminium alloy joints, the test is applicable to metal-metal, metal-composite and composite-composite joints.

The test involves forcing a wedge into the bondline of a flat-bonded specimen (ie double cantilever beam configuration), thereby creating cleavage stresses in the region of the resulting crack tip. The wedge imposes a fixed displacement to the adherends and the energy stored in bending the adherends (ie arms) provides the driving force for crack growth. The stressed specimen is exposed to an aqueous environment, usually at an elevated temperature, or an appropriate environment representative of service condition. The resulting crack length is monitored with time, and fracture energies,  $G$ , are determined as follows [20]:

where ( $E$ ) is the Young's modulus of adherends, ( $w$ ) is the displacement caused by the wedge, ( $h$ ) is adherend thickness and ( $a$ ) is the crack length. Measurement of crack length can be performed reasonably accurately using a travelling microscope. Variations in surface condition are easily observed by completely separating the two adherends and examining the surface under an optical microscope [21].

$$G = \frac{Ew^2h^3[3(a+0.6h)^2 + h^2]}{16[(a+0.6h)^3 + ah^2]^2} \quad (3)$$

Specimens are nominally 25 mm wide and 200 mm long, with an adherend thickness of approximately 2 mm. The bonded joints can be made either separately or sectioned from a bonded panel (150 mm square). An aluminium or steel wedge, 25 mm square and with a 14° taper is specified in ASTM D 3762 [18]. Testing and specimen fabrication is relatively straightforward and economic.

The wedge test is more severe than conventional lap-shear or peel tests, because water is present at the crack tip, a region of high tensile stress, whereas moisture has to diffuse into the bondline from the edges of the lap-shear and peel tests. **The test is not suitable for generating quantitative fracture strength data or for short-term testing.** If the bonded joint is sensitive to the test environment, then significant crack growth can be expected to occur within one hour of exposure [18]. A crack growth limit will often be reached within 10 days for most systems, enabling rapid comparative durability measurements [13]. The general accuracy of the wedge test is compromised when testing toughened adhesives and flexible adherends. It is therefore important to ensure that plastic deformation of the adherends is

either prevented or at least minimised when inserting the wedge. The effectiveness and sensitivity of the technique is dependent on the ageing environment.

### 3.3.2 Cleavage strength (Compact Tension) Test

An alternative approach is to use the cleavage strength (ie compact tension) test shown in Figure 7 [22, 23]. In this method, a rigid joint is subjected to cleavage stress by the application of a tensile force at one edge of the joint. The test was developed for determining the cleavage strength of metal-to-metal adhesive bonds, with steel being the preferable material of construction. Specimens are easily fabricated by bonding the flat adherends together. The spew fillet present at the loading end of the specimen should be carefully removed, avoiding cracks and scratches, which will considerably reduce cleavage strength of the adhesive bond. Specimens are typically 25 mm wide, with a bondline length of 25 mm. The depth of the adherend is 12 mm along the bondline. The BSI standard may be used for assessing compliance of an adhesive with a specification and for quality control.

A special fixture is required to test the specimens. **Although, the test was designed for the determination of comparative cleavage properties under static loading conditions, the use of the procedure could possibly be extended to fatigue testing.**

## 3.4 SHEAR TESTS

The transfer of load between adherends is most effectively achieved by shear stress transfer. A quick perusal of the ASTM and ISO standards (Appendix 1) will quickly reveal a multitude of shear test geometries, each with the objective of producing a state of pure uniform shear in the adhesive layer. This section critically evaluates the following test configurations listed below (see also Tables 3 and 4):

- Lap joints (single, double and modified)
- V-notched beam (Iosipescu)
- Arcan
- Plate-twist
- Torsion of cylindrical butt joint
- Thick adherend
- Napkin ring
- Skin doubler

### 3.4.1 Lap Joints

#### a) Single-lap joint

Despite all its obvious weaknesses, the lap-shear test (Figure 8) is the most widely used method for producing *in-situ* shear strength data of an adhesively bonded joint. The test consists essentially of two rectangular sections, typically 25 mm wide, 100 mm long and 1.6 mm thick, bonded together, with an overlap length of 25 mm [24]. Variations of this test method are included in both national and international standards [24-26] (see Appendix 1). The single-lap specimen is easy to prepare and test. A fixture is used to ensure correct overlap and accurate alignment of the adherend. This may include control of the fillet. Testing can be conducted using standard tension/compression mechanical test equipment. The lap-shear strength ( $\tau$ ) is given by:

$$t = \frac{P}{bL} \quad (4)$$

where (P) is the maximum load, (b) is the joint width and (L) is the joint length.

The analysis assumes the adherends are rigid, and that the adhesive only deforms in shear. In fact, the resultant stress distribution, across and along the bond length is very complex. The eccentricity of the load path causes out-of-plane bending moments, resulting in high peel stresses and non-uniform shear stresses in the adhesive layer. This effectively reduces the structural efficiency of the joint [7]. The structural efficiency is defined as the ratio of the direct membrane stress outside the joint to the sum of the stretching and bending stresses at the ends of the bonded overlap [7]. For BS EN 1465 [24] and ASTM D 1002 [25], the structural efficiency is approximately 0.33. The solution is to increase the overlap length-to-thickness (L/t) ratio from 8:1 to 80:1, which is obviously not a practical remedy for coupon tests, but will alleviate the problem for structural joints.

Efforts to reduce stress concentrations formed at the bondline ends have included the use of tapered or bevelled external scarf and radius fillets at the bondline ends (NB. The use of absolutely rigid adherends will not prevent the formation of stress concentrations at the bondline ends). Tapering the ends can prevent premature failure, but the shear strength of the joint remains unaltered. Significant increases in the "apparent" shear strength of single-lap joint, compared with square-ended bondlines, can be achieved through the formation of a spew fillet at the overlap ends [27]. Further increases in strength may be achieved by rounding the ends of the adherend.

The main problem with the single-lap shear test is that the average shear strength determined using this method does not correspond to a unique material property of the adhesive, and therefore cannot be used as a design parameter. In fact, this quantity is strongly dependent on the joint geometry.

### Cyclic and Environmental Testing

The rapid and continuous changes that occur to the shear and normal stress distributions within a single-lap shear specimen with increasing monotonic loading raises doubts about tests involving creep or fatigue. Exposure to elevated temperatures or hostile environments can only compound this uncertainty. The test, however, is widely employed to evaluate problems associated with service conditions, providing the user with comparative data on long-term performance of adhesive joints. Both BS EN ISO 9664 [28] and ASTM D 3166 [29] specify a method, based on the single-lap joint, for the measurement of fatigue strength. Environmental tests often involve a train of standard or miniaturised joints with or without the application of external loading. The factors that influence the tolerance of the single-lap shear joint to either static or cyclic loading include on lap length (ideally 30 times the joint thickness) adhesive and adherend elastic moduli and thickness and bondline thickness [19].

In practice, a large number of bonded systems may need to be evaluated in relatively short time spans. The test specimen needs to be simple and quick to prepare and sensitive to environmental effects. For these reasons, a perforated short diffusion path configuration of the single-lap joint (Figure 9) has been adopted by a number of industries [31]. Specimens are typically 20 mm wide and 120 mm long. The overlap length is 10 mm. The short diffusion path is provided by drill holes 3 mm or 4 mm in diameter located within the bonded area. The possibility of the joints failing by yielding and fracture of the material between the drilled holes is reduced by using the smaller diameter holes. These specimens are often prestressed in self-stressing fixtures and exposed to either natural weathering or accelerated ageing conditions representing in-service conditions. A number of specimens

(typically six) may be loaded in a single train with failed joints replaced with spacers.

#### b) Double-Lap Joint

Attempts to eliminate eccentric loading, responsible for bending of the adherends and rotation of the bonded region, have resulted in the development of a symmetric variant of the single-lap shear test, known as the double-lap joint (Figure 10). However, bending of the outer adherends is unavoidable, since the load is applied to the outer adherends via the adhesive, away from the neutral axis. The bending moment introduces tensile stresses across the adhesive layer at the free end of the overlap and compressive stresses at the other end. The centre adherend is free from the net bending moment.

Double-lap joints are twice as strong as their single-lap counterparts. For practical design purposes, the optimum  $L/t$  ratio is 30. Beyond this limit, any additional increase in the value of  $L/t$  is ineffective in reducing peak adhesive shear and peel stresses. This limit is known as the **ineffective** length. The joint efficiency of double-lap joints is considered good. Various constructions of this test configuration are included in both national and international standards [26, 28, 30] (see Appendix 1). Adherend dimensions are identical to those employed for single-lap shear.

#### c) Modified Lap Joints

A number of design modifications have been recommended to reduce/remove peel stresses associated with single-lap and double-lap joints, and thus increase the strength of the bonded joint [1, 7, 19, 60]. An example is the double-butt strap lap joint with and without tapering (internal and external) as shown in Figure 10. Tapering the adherends, minimises peel stresses, increases bond efficiency (ie strength) and alters the failure mode from peel to shear. The peel stresses virtually disappear when using external tapers with 30° fillets [7, 19]. Both configurations (ie 30° and 90° fillets) shown in Figure 10 are suitable for determining shear behaviour under fatigue, creep and environmental assessment of shear behaviour, with butt separation monitored using extensometers. The cost and time involved in fabricating specimens with internal and external tapers are well in excess of the double-strap joint.

In practice, a large number of bonded systems may need to be evaluated in relatively short time spans. The test specimen needs to be simple and quick to prepare and sensitive to environmental effects. For these reasons a perforated short diffusion path configuration of the single-lap joint geometry (Figure 11) has been adopted by a number of industries [31]. Specimens are typically 20 mm wide and 120 mm long with either three 3 mm or 4 mm diameter holes drilled through the bonded section of the specimen. The possibility of the joints failing by yielding and fracture of the material between the drilled holes is reduced by using the smaller diameter holes.

### 3.4.2 V-Notched Beam (Iosipescu) Test

#### Bulk Resin Tests

The V-notched beam test was originally developed by Iosipescu for measuring the shear strength of metals and welds [32]. The original specimens were cylinders, with a 90° circumferential notch located at the specimen centre. The notch was introduced to promote a uniform state of shear at the specimen centre, and to ensure failure occurred within the test-section. Walrath and Adams [33], subsequently modified the specimen geometry and loading configuration in order to determine the shear characteristics (ie shear modulus and

shear strength) of fibre-reinforced plastic composites. The modified method, now standardised as ASTM D 5379 [34], is used widely for characterising the shear properties of polymeric materials, including adhesives [35-39].

The ASTM method employs a double edge-notched, flat, rectangular specimen (Figure 12) with two 90° angle notches cut at the edge mid-length with faces orientated at ±45° to the longitudinal axis. The notches extend to a depth of 20% of the specimen width (ie 4 mm). ASTM D 5379 recommends a notch root radius of 1.3 mm in order to minimise the shear stress concentration at the notch roots, and thus promote a more uniform shear stress distribution along the notch-root axis. The specimen has a length of 76 mm and a width of 20 mm with a specimen thickness between 3 and 4 mm. Specimens with a thickness less than 3 mm require adhesively bonded tabs (typically 1.5 mm thick) to prevent out-of-plane bending or twisting which could lead to premature failure. Local crushing, which can occur near the inner loading regions, can also be avoided by the use of tabs. Test specimen dimensions may be reduced if required.

In principle, this procedure induces a state of pure shear stress at the mid-length of an isotropic specimen, by the application of two force couples. A state of constant shear force is induced through the mid-section of the test specimen, with the induced moments cancelling exactly at the mid-length, thereby producing a state of pure shear at this location.

Numerical and experimental studies of the V-notched beam method have shown that the shear stress distribution in the test-section of both isotropic and anisotropic specimens is not uniform [36, 37]. Furthermore, shear and normal stress distributions have been shown to be highly dependent on the loading boundary conditions, the notch geometry and the material anisotropy [5]. The use of a large notch root radius (ie 1.3 mm), as recommended in ASTM D 5379, is not entirely successful in promoting a uniform shear stress distribution. Tensile stresses near the notch roots can lead to premature tensile failure of brittle isotropic materials.

Shear strain is measured by bonding two biaxial strain gauges (one on each opposite face of the specimen) to the centre of the specimen in the area between notches. The strain gauges have a gauge-length of 1 mm or 2 mm, to keep within the region of uniform stress, and are aligned at ±45° to the longitudinal axis of the specimen. The average shear strength ( $S_{xy}$ ) and shear modulus ( $G_{xy}$ ) can be calculated by the following equations:

$$S_{xy} = \frac{P_{\max}}{wh} \quad (5)$$

$$G_{xy} = \frac{\Delta t_{xy}}{\Delta g_{xy}} = \frac{\Delta P}{wh\Delta(e_{+45} - e_{-45})} \quad (6)$$

where ( $P_{\max}$ ) is the ultimate failure load, ( $w$ ) is the notch width and ( $h$ ) is the specimen thickness. Changes in applied load ( $\Delta P$ ) and +45° or -45° normal strains, ( $\Delta \epsilon_{45}$ ) and ( $\Delta \epsilon_{-45}$ ) in Equation (6) are obtained from the initial linear region of the stress-strain curve. The shear-stress strain curve exhibits some nonlinearity close to fracture.

Although a special test fixture is required, testing is relatively straightforward. To minimise potential effects of out-of-plane movement or twisting of the specimen, it is recommended that the strain data used for determining shear modulus be the average of the indicated strains from each side of the specimen. A number of investigators [40, 41] have modified the recommended ASTM fixture in an attempt to prevent misalignment of the two fixture halves.

The failure process is highly dependent on the microstructure of the material. Figure 13 shows typical failure modes recorded for thermoset and thermoplastic materials. Tensile

failure is characteristic of brittle polymers (eg untoughened epoxy and acrylic resins). Failure initiates in the vicinity of the notch roots in a region of high tensile stress with cracks propagating in an unstable manner along the plane of the principal tensile stress. For these materials, ultimate failure stress does not correlate with shear strength. Thermoplastic polymers (eg polycarbonate and polypropylene) tested at room-temperature fail as a result of shear yielding along the notch root axis. Ductile materials can redistribute the tensile stress through plastic deformation, and therefore strain data can be obtained well beyond the shear yield stress of the material. Localised high compressive stress gradients may also produce plastic deformation in the vicinity.

### Adhesive Joint Tests

Work has been conducted using the V-notched beam test for characterising the shear properties of adhesive joints [39, 42, 43]. The test specimen consists of two shaped metal adherends, bonded together, with the bondline forming the test section between the notch roots (Figure 14). Shear strain was measured via a strain gauge based shear extensometer. Tests conducted on a flexible adhesive showed reasonable repeatability for the stress-strain response, however, no shear moduli data was supplied. Specimens can be expected to fail at the adhesive-metal interface due to high transverse tensile stresses present at the bondline ends. This mode of failure has been observed for Arcan joint specimens [38], and there is no indication that V-notched beam specimens should respond differently. Strain gauges and shear extensometry are unsuitable for use with adhesive joints specimens with thin bondlines.

### Cyclic and Environmental Testing

The V-notched beam test has limited fatigue testing capabilities. The loading fixture would probably need to be modified in order to minimise frictional effects and prevent fretting at the loading points. This method is suitable for creep and environmental testing. The formation of ice within the moving parts of the test fixture (Figure 12) could cause problems at sub-zero temperatures. Despite this, the test method has been used successfully over the range -40°C and 180°C.

### **3.4.3 Arcan Test**

#### Bulk Resin Tests

The Arcan test method employs a compact butterfly specimen (Figure 15) with two symmetrical notches cut at  $\pm 45^\circ$  to the loading axis [38, 39]. This specimen is gripped in a loading frame with the load applied via the faces to avoid the potential problem of instability associated with edge loading [39]. This method has been used to measure the shear properties of fibre-reinforced plastic composites and adhesives [44-49]. The original Arcan specimen consisted of a diametrically loaded circular disc containing asymmetric cut-outs with the mid-section designed to establish a state of uniform stress. Depending on the loading angle, various biaxial stress states can be produced. In principle, a uniform state of pure shear should exist when the loading axis and the notch axis are aligned.

Specimens are typically 40 mm wide, 50 mm long with a 12 mm distance between notches. Modulus measurements obtained from bulk adhesive specimens have been shown to be consistent for both 2 mm and 4 mm thick specimens. A notch radius of 1.5 mm is used to ensure a relatively uniform shear stress state exists within the specimen gauge-section [39]. Specimens are clamped in a specially designed fixture, with the four bolt holes shown in Figure 15 serving to supply the necessary pressure to the jaws. The specimen is loaded

along the faces and by the bolts. Alignment is critical with small misalignments resulting in a prestressed specimen, which could lead to premature failure.

Shear strain is measured by either bonding two biaxial strain gauges, one on each opposite face of the specimen, to the centre of the specimen, in the area between notches, or by using a shear extensometer [39]. The strain gauges have a gauge-length of 1 mm or 2 mm, to keep within the region of uniform stress, and are aligned at  $\pm 45^\circ$  to the longitudinal axis of the specimen. The extensometer is suitable for measuring shear modulus, but is not satisfactory for determining failure strains. Measurements of shear modulus have been found to correlate well with other techniques, whereas the shear strength value tends to be low. This is because failure occurs as a result of high transverse tensile stresses on the cutout boundary, away from the gauge-section.

High compressive stresses also exist near the notch roots, and as a result localised buckling may occur. Compliant materials, such as polyurethane, are prone to deform at high stresses. Measurements are invalidated unless constraints are applied the specimen clamps to maintain alignment.

### Adhesive Joint Tests

Arcan joint specimens consist of two symmetrical metal adherends, each forming half of the test specimen as shown in Figure 16. Each adherend is 19.75 mm wide to allow for a 0.5 mm bondline thickness. The adherends are typically 6 mm thick. Bondline length may range from 5 to 10 mm, without producing significant differences in test data. The two halves are bonded together in a clamping fixture. This fixture ensures good specimen alignment and maintains suitable pressure on the specimen during cure.

Specimen alignment is particularly critical as small misalignments can result in premature failure of the specimen. Rigid adhesives (eg epoxy) are particularly sensitive. An approach that has proved successful has been to use a clamping fixture for specimen manufacture that replicates the actual loading fixture [39].

The shear stress distribution at the specimen centre is uniform, however there are shear stress concentrations at the notch roots. Numerical analysis [39] has shown that tensile and compressive peel stress concentrations occur near the ends of the bonded region, at the interface between adhesive and adherend. The use of concave fillets at the bondline ends lowers the shear and peel stresses in these regions, preventing premature failure of the specimen. The shear displacement is measured using an extensometer with the average shear stress given by Equation (5), as with the V-notched beam test. Strain gauges are unsuitable for this application. The method is particularly suitable for measuring the shear moduli of compliant materials (ie  $G < 1$  GPa), such as polypropylene, polyurethane and acrylic.

The application of shear extensometry to adhesively bonded Arcan specimens proved more difficult than for bulk adhesive specimens. Due to the hardness of the metal adherends, it is difficult to achieve an acceptable contact between the extensometer needles and the adherend surface. This approach is unsuitable for fatigue testing, as the extensometer needs would be unable to maintain satisfactory contact with the specimen.

### Cyclic and Environmental Testing

Provided the necessary precautions are taken, the Arcan test is probably suitable for long

term testing under cyclic loading conditions. There will probably be a tendency under cyclic loading for slippage within the grips of the test fixture, resulting in the loading bolts directly bearing onto the specimen. This would cause bulk adhesive specimens to fail within the gripping regions. Although this may not be a serious problem for adhesive joint specimens, concern must be expressed as to the validity of the strength data, since these specimens invariably undergo interfacial failure as result of the presence of high transverse tensile stresses at the bondline ends. This method is expected to be suitable for creep and environmental testing, provided the measurements are restricted to the measurement of shear modulus.

### 3.4.4 Plate-Twist Test

The plate-twist method, which was initially developed to measure the shear modulus of plywood (ASTM D3044-76 [50]), has proved satisfactory for measuring shear moduli ranging from 0.29 GPa (chopped glass-fibre reinforced polyurethane) to 88.2 GPa (steel). The test method is unsuitable for determining in-plane shear strength [51].

In the plate-twist test, a square plate is supported on the two corners of one diagonal and load is applied at a constant rate to the corners of the opposite diagonal (Figure 17). The stress state induced in the plate is essentially pure shear. The total load is recorded as a function of the resultant displacement. The plate should be square or rectangular in shape with the diagonals being of equal length. The plate length to thickness ratio should be  $\geq 35$  to minimise through-thickness shear effects. This test method is unsuitable for materials that are not transversely isotropic or homogeneous through the section (eg multidirectional laminates including  $0^\circ/90^\circ$  cross-ply). For these materials, the shear modulus produced under flexural loads is no longer equivalent to the in-plane shear modulus, but equivalent to the effective flexural shear modulus [51].

The shear modulus ( $G_{12}$ ) can be determined from the displacement of the loading points ( $\Delta w_p$ ):

$$G_{12} = \frac{3\Delta PabK}{4\Delta w_p h^3} \quad (7)$$

and from the displacement at the plate centre ( $\Delta w_c$ ):

$$G_{12} = \frac{3\Delta PabK}{8\Delta w_c h^3} \quad (8)$$

where (a) and (b) are the plate edge dimensions, (h) is the plate thickness and ( $\Delta P$ ) is the change in load (total) for a change in displacement (w). A practical difficulty with the test has been the positioning of the loading points, which are normally in-board, rather than at the actual corner; as assumed in the analysis. This mis-positioning leads to an error of several percent in the shear modulus value. A correction factor (K) has been introduced into Equations (7) and (8) to account for in-board loading [52]:

$$K(r) = 3r^2 - 2r - 2(1-r)^2 \ln(1-r) \quad (9)$$

where (r) is the ratio of (S) the test span diagonal length and (D) the plate diagonal length. The correction factor (K) is relatively insensitive to variations in the Poisson's ratio ( $\nu$ ) over the range 0.25 to 0.4016 [51]. The analytical solution as given by Equation (9) produces (K) values almost identical to those calculated by Sims et al. [51] using a polynomial line fit to finite difference data (ie less than 1% for  $r > 0.8$ ).

A draft test procedure ISO/CD 15310 [53] is currently under consideration for international standardisation. The procedure, supported by a round-robin trial run by the National

Physical Laboratory, recommends a standard plate specimen 150 mm x 150 mm, and an in-board ratio of 0.95, with other plate dimensions being optional. The maximum thickness requirement for non-standard plate specimens should meet the requirement of  $35h \leq a \leq b$ . The modulus is determined over the displacement range of 0.1h to 0.3h, with a maximum allowable plate deflection of 0.5 mm.

There are many advantages in the use of a plate specimen, particularly for moulded plastics and fibre-reinforced plastic composites. For these materials, plate specimens more closely represent the properties of actual products than moulded-to-size dumbbell and beam specimens or hoop-wound tubes. As the technique is non-destructive (most shear methods result in the destruction of the test material during testing), a plate-twist specimen can also be sectioned into tensile or compression coupon specimens along the principal material directions to assess the materials anisotropy. This guarantees that the shear modulus relates directly to any other property measured from the plate. An additional bonus is that the test results represent the shear response over a relatively large area, which means variations in microstructure across the plate are averaged.

### **3.4.5 Torsion Method**

#### Bulk Resin Tests

Torsion loading of a circular rod is mechanically one of the most accurate methods for characterising the shear properties of bulk resin [54]. Specimens may be moulded or machined directly from rods. A typical specimen is shown in Figure 18 [55]. This specimen has an overall length of 135 mm with a circular gauge-section, 10 mm in diameter and 85 mm long. The specimen ends are 12.7 mm square with a gripping length of 25 mm. The ends of the gauge-section are filleted (6 mm radius) to minimise stress concentrations present at these locations.

The high degree of machining required exposes this specimen geometry to a high risk of machine-induced damage. This is compounded by difficulties in producing castings free of voids and residual stresses place considerable limitations on the materials that can be evaluated using the torsion method. This test may be inappropriate for polymeric materials that have a rapid exothermic cure reaction (eg acrylic) or exhibit considerable flexibility (eg polyurethane). Accurate machining of flexible materials can be a problem.

Torsion testing of solid circular rods is relatively straightforward to perform, provided suitable mechanical test equipment (ie torsion facility) is available. Many laboratories however do not have access to suitable equipment. The equipment is relatively complex and requires that no axial loads are applied to the test specimen during test set-up or during the test. Servo-hydraulic torsion + tension/compression test machines are more costly than servo-hydraulic tension/compression machines. Costs can be reduced by using a torsion gear box attached to a screw-driven test frame.

Surface shear strain is measured using either strain gauges or by contacting extensometers. Strain gauges are adhesively bonded to the specimen surface at the centre of the gauge-section. The gauges are oriented at  $\pm 45^\circ$  to the longitudinal axis of the specimen. An additional axially aligned strain gauge is recommended to monitor longitudinal strain. Tensile or compressive axial strains must not be present throughout the test duration. The shear modulus is determined from the linear region of the stress-strain curve.

Simple data reduction procedures are available for both linear and non-linear stress-strain response of circular rods in torsion. The analysis procedure developed by Nadai [56] for

metals can be used to correct for the non-uniform shear stress distribution through the specimen cross-section, which occurs during non-linear stress-strain behaviour. The accuracy and repeatability of strain measurements, resulting from the large gauge-length, is relatively high compared with other test methods.

As with all bulk test specimens, the strength data is dependent on the quality of the specimen. Internal stress concentrations in the form of entrapped air introduced during processing can result in premature failure.

### Adhesive Joint Tests

The butt torsion test is a suitable method for all types of adhesives and is capable of generating reliable shear modulus values and stress-strain curves. A typical butt torsion specimen is shown in Figure 19 [55]. This specimen has a 15 mm diameter circular bonded region. Contact extensometers are used to measure the angular rotation across the adhesive. A major disadvantage of this method is the difficulty of obtaining high precision strain measurements, due to the very low angular displacements (1-2 degrees) associated with this test configuration [55].

Shear modulus (G) can be determined from the following equation:

$$G = \frac{2b}{p R^4} \left( \frac{T}{q} \right) \quad (10)$$

where (T) is the applied torque, (R) is the radius, (b) the adhesive layer thickness and ( $\theta$ ) the angular displacement.

The stiffness of the adherends needs to be considerably higher than the adhesive stiffness, in order to minimise the effect of adherend deformation on the stress-strain response. Strain measurements need to be corrected for the effect of adherend deformation and non-linear stress-strain behaviour (ie non-uniform shear stress distribution across the bond area). The shear distribution is high on the external surface and lower in the centre. From a structural design perspective, butt joints should be avoided.

### Cyclic and Environmental Testing

This test in either bulk or joint form is compatible to cyclic, creep and environmental loading conditions. Environmental equipment can be adapted to meet the requirements of torsion equipment. Concern must be expressed with regard to materials, such as polyurethane, where the behaviour is highly rate dependent. For polyurethanes the glass-to-rubber relaxation transition occurs at ambient temperatures. The corrections for non-linear behaviour may prove inadequate.

#### **3.4.6 Thick Adherend Test**

An alternative approach for determining the shear properties of an adhesive is to apply uniaxial tensile or compressive load to a specimen consisting of thick, rigid adherends, with a short overlap length (Figure 20) [57]. This test geometry was developed to overcome the inherent weaknesses of the double-lap shear test. Various thick adherend geometries have been suggested, including those specified in ISO 11003 - 2 [58] and ASTM D 3165 [59]. The specimen can be produced by either:

- i) Bonding two pre-shaped bars together; or
- ii) Bonding two sheets together and then milling two parallel slots.

ISO 11003 - 2 specifies a specimen with an overall length of 110 mm, a width of 25 mm and an overlap length of 5 mm. The adherend thickness is 6 mm and the bondline is typically 0.5 mm thick. Slots should be 1.5 mm wide.

Load is introduced to the specimen via two 12.7 mm diameter bolt holes. The hole centres are 80 mm apart. Care is needed to ensure that the holes are accurately drilled in the centre of each adherend, since small misalignments can result in unwanted rotation and uneven loading of the joint, thus compromising the test data. Testing can be conducted using a standard mechanical test machine.

It is important to ensure that no coolants are used when machining the two parallel slots, as the coolant may react with the adhesive or penetrate the adherend-adhesive interface. Localised increases in temperature may also result in thermal degradation of the adhesive properties. The slots must be accurately machined through only one adherend.

In principle, the entire load is transmitted by shear forces, distributed along the bondline between the slots or notches. Finite element analysis [57] has shown that the shear stress distribution along the overlap length is predominantly shear and that large peel stresses are present at the corners of the overlap. The large peel stresses may cause premature failure. The high concentration of peel stresses may be reduced by adding a fillet to the end of the bondline. The average shear stress,  $\tau$ , is given by [57]:

$$\tau = \frac{P}{bL} \quad (11)$$

where (P) is the applied force, (L) is the overlap length and (b) the specimen width.

Strain is measured using two extensometers in order to eliminate errors from unsymmetrical specimens, the results are averaged. The procedure used is given in references [57] and [58]. The shear modulus is obtained by calculating the slope of the linear region of the stress-strain diagram. If the strength results are to be used for design of bonded structures, then only cohesive failure data can be used.

The thick adherend test specimen could also be loaded in compression using a similar fixture to that specified in ASTM D 3846 [60]. In the ASTM standard, a composite specimen is supported along its entire length to minimise out-of-plane deformation. Specimens are nominally 79.5 mm in length and 12.7 mm wide for equivalence with the short beam method. The two parallel notches are 6.4 mm (0.25 in) apart and between 1 to 1.6 mm wide. Test specimen geometry and loading configuration are given in Figure 21. The method specified in ASTM 3846 is relatively straightforward to perform, requiring a support fixture of moderate cost.

### Cyclic and Environmental Testing

The thick-adherend test should be suitable for characterising the shear properties of adhesive joints subjected to cyclic or creep loading, and/or hostile environments. The simplicity of the test geometry ensures that the thick-adherend method provides no particular loading problems under these conditions, although difficulties are envisaged in monitoring shear strains with the contact extensometers for cyclic loads and in corrosive environments. Clip gauges, used for crack opening displacement measurements, may prove satisfactory.

### 3.4.7 Napkin Ring Test

The napkin ring test (Figure 22) produces a relatively uniform shear stress within an adhesive layer (ie 3% variation across the adhesive ring), and hence the method has been used for producing shear modulus and shear strength data for adhesives [61, 62]. The test consists of applying equal and opposite torques to the tubular adherends. Shear stress is a maximum at the outer radius. Significant stress concentrations are present at the inner and outer bondline perimeters. Bevelling the inner and outer adherend edges reduces these stress concentrations. A number of researchers have preferred to use a solid butt joint in preference to the napkin ring simply because it was easier to fabricate, and only the outer spew fillet needed to be removed [7].

The test consists of applying equal and opposite torque to two tubular adherends butt joined by an adhesive ring. This configuration is used to generate shear modulus and shear strength of adhesive films restrained by relatively higher modulus adherends [61].

$$t_{z\theta} = \frac{2TR}{p(R_o^4 - R_i^4)} \quad (12)$$

The shear stress ( $\tau_{z\theta}$ ) at a radial distance (R) from the central axis is [7]:

where (T) is the applied torque, ( $R_o$ ) is the outer radial boundary and ( $R_i$ ) is the inner radial boundary of the cylinder.

As with all torsional loading configurations, there is a need for specialised test equipment and loading fixtures. Contact extensometers can be used to measure the angular rotation across the adhesive, although it may be difficult in obtaining high precision strain measurements, due to the very low angular displacements (1-2 degrees) associated with this test configuration [53]. The little evidence to support the use of the napkin ring specimen in determining fatigue resistance under shear loading conditions (see also Section 3.4.5).

### 3.4.8 Skin-Doubler Test

As previously mentioned, a number of factors render most of the test methods unsuitable for fatigue testing of structural adhesives, particularly when combined with prolonged exposure to a hostile environment. The tendency is for engineers to conduct fatigue tests on actual components or on complex specimens related to a specific design. Krieger [7] suggests that the data generated by loading a skin-doubler specimen (Figure 22) in tension is probably more pertinent to actual engineering structures, providing static, fatigue and creep design allowables. Testing can be conducted using standard tension/compression mechanical test equipment. Specimen fabrication is relatively straightforward and economic. Stress-strain response can be measured in both the linear and nonlinear regions of the stress-strain curve using a KGR-1 extensometer (Figure 22) [7]. Unfortunately, there is insufficient comparative data to support the use of this technique for determining allowables for adhesives.

### 3.5 FRACTURE TOUGHNESS TESTS

A major consideration in the design of adhesively bonded structures is the possibility of crack growth, either within the adhesive or at the adhesive-adherend interface. Crack propagation can be catastrophic when the fracture toughness ( $G$ ) of the adhesive-adherend system has been exceeded. Debonds or delaminations are probably the most life-limiting defects that occur in layered or laminated structures, and may arise during processing or subsequent service. Common structural features, such as thickness changes, bolt holes or bonded joints, generate through-thickness stress concentrations, which may initiate debonding under static or cyclic loading conditions. The situation is exacerbated at elevated temperatures and in the presence of aggressive environments (eg water vapour or solvents).

Crack initiation and growth usually occurs under mixed-mode conditions, a combination of mode I (crack-opening), mode II (forward-shear) and mode III (scissor-shear) [62]. As a result, fracture toughness tests have been developed to quantify resistance to crack initiation and growth under tensile and shear loading conditions. These test methods are used to assess the influence of adherend surface condition, adhesive, adhesive-adherend interactions, primers, etc., on the fracture strength. Efforts in recent years have been directed towards generating reliable test methods for determining fracture toughness under mixed-mode loading conditions.

Serious doubts have been raised as to the relevance and reliability of employing a fracture mechanics approach for the design and analysis of adhesively bonded structures. It is recognised, however, that fracture mechanics has proved a valuable tool for understanding the fundamental mechanisms of adhesion. Kinloch et al [63] have shown that fracture toughness data obtained from mode I fatigue tests can be used to predict the fatigue life of adhesively bonded joints. Good agreement was obtained between the theoretical predictions and experimental results for the fatigue behaviour of single-lap joints. This section evaluates a number of mode I, mode II and mixed-mode I/II test methods.

#### 3.5.1 Mode I

The crack growth in adhesive bond specimens can proceed in two ways [64]:

- (i) Slow-stable extension where the crack is dictated by the displacement (ie cross-head) rate (eg rubber modified film adhesives tested at sub-zero temperatures).
- (ii) Run-arrest extension where a stationary crack abruptly grows at a rate that exceeds the cross-head speed (eg modified epoxies at 73°C).

Terms associated with run-arrest crack growth are given below:

**Opening mode fracture toughness,  $G_{IC}$ :** The value of ( $G_I$ ) just prior to the onset of rapid fracture is determined from the load required to initiate crack growth.

**Opening mode crack arrest toughness,  $G_{Ia}$ :** The value of ( $G_I$ ) just after arrest of a run-arrest segment of crack extension is determined from the load required for crack arrest.

**Double Cantilever Beam (DCB):** Although, this test has attracted considerable academic interest, it is not widely used in industry. The test is used to measure the initiation and propagation values of ( $G_I$ ) under static and cyclic loading conditions. A tensile load is applied to a specimen with an embedded through-width insert (ie debond) at the specimen mid-plane (Figure 23). The tensile force acts in a direction normal to the crack surface.

Specimens are typically 25 mm wide and 356 mm long. The adherend thickness is typically 6.35 mm (0.25 inches).

The critical strain-energy release rate or fracture toughness ( $G_I$ ) is calculated as follows [64]:

$$G_{Ic} = \frac{4P^2(3a^2 + h^2)}{Eb^2h^3} \quad (13)$$

where (P) is applied load, (E) is the Young's modulus of adherend, (b) is the specimen width, (a) is the crack length and (h) is the adherend thickness. To determine ( $G_{Ic}$ ) and ( $G_{Ia}$ ) values, the corresponding applied load values ( $P_{max}$ ) and ( $P_{min}$ ) are substituted into Equation (13).

Non-linear load-displacement response may result from inelastic material behaviour and/or subcritical damage formation in front of the planar crack (ie micro-cracking and extensive deformation) or by large beam deflection. The latter can be associated with materials with low flexural stiffness or loss in flexural stiffness resulting from substantial crack growth. The analysis shown in Equation (13) assumes linear elastic behaviour and no large displacement effects. Williams [65] using finite displacement analysis developed correction factors to account for the shortening of moment arms and the tilting of the loading blocks.

Fabrication and testing of DCB specimens is straightforward and relatively inexpensive. Testing can be conducted using standard mechanical test frames. Specimen fabrication is identical to that employed for wedge cleavage specimens. Reusable aluminium loading blocks are recommended for polymer composites (Figure 24). **Both static and fatigue loading can be used with these specimens. Tests may also be conducted under simulated service environments such as hot humid environments.**

Crack growth rate ( $da/dN$ ) can be related to the total strain energy release rate ( $G_{max}$ ) by a form of the Paris Equation [62, 66]:

$$\frac{da}{dN} = C(G_{max})^n \quad (14)$$

where C and n are material constants. As with static tests, the crack extension is measured using either a travelling microscope, a crack gauge or video camera. The use of a crack gauge enables crack measurement to be automated. For static tests, the coefficient of variation is typically 20% or higher. The uncertainty is far greater for fatigue tests where the power-law relationship (Equation (14)) is sensitive to small errors in applied load and crack length.

**Tapered Double-Cantilever Beam (TDCB):** The purpose of this test geometry is to make the measurement of fracture toughness ( $G_I$ ) independent of the crack length (ie constant compliance). The specimen design is well suited to tests where the crack length (a) is difficult to measure, especially environmental testing. To develop a linear compliance specimen, the height is varied so that the quantity  $3a^2/h^3 + 1/h$  is constant.

The critical strain-energy release rate or fracture toughness ( $G_{Ic}$ ) is calculated as follows [64]:

$$G_{Ic} = \frac{4P^2}{Eb^2} \left( \frac{3a^2}{h^3} + \frac{1}{h} \right) \quad (15)$$

where (P) is applied load, (E) is the Young's modulus of adherend, (b) is the specimen width, (a) is the crack length and (h) is the adherend thickness. A schematic of the TDCB specimen is shown in Figure 25.

**The TDCB test specimen has been used to determine the rate of crack growth under**

**various cyclic loading and environmental conditions** [7, 62]. The main disadvantage is the relatively high cost associated with specimen fabrication. Specimens are typically 241 mm long and 25 mm wide. Adherend thickness has a nominal maximum thickness of 32 mm [64]. Variations of TCDB test are being developed to test thin metal sheet or composite laminates.

### 3.5.2 Mode II

**End-Notched Flexure (ENF):** This test has emerged as the most commonly used test method for quantifying ( $G_{IIc}$ ) for metallic and laminated polymer composite adherends. The ENF test is essentially a DCB specimen loaded in three-point flexure (Figure 26). The dimensions are similar to those employed for the DCB specimen. The test is straightforward and can be conducted using a standard mechanical test equipment. A flexure fixture is required, which can be purchased from a test machine manufacturer or produced in-house. Commercial fixtures are available.

**Both static and fatigue loading can be used with these specimens. Tests may also be conducted under simulated service environments such as hot humid environments.** Analysis is straightforward, although there is concern as to the method of precracking.

The critical strain-energy release rate or fracture toughness ( $G_{II}$ ) is calculated as follows [64]:

$$G_{Ic} = \frac{9P^2 a^2}{16Eb^2h^3} \quad (16)$$

where (P) is applied load, (E) is the Young's modulus of adherend, (b) is the specimen width, (a) is the crack length and (h) is the adherend thickness. The values of ( $G_{IIc}$ ) determined using Equation (16) need to be corrected for transverse shear deformation and large displacement effects. Crack growth rate ( $da/dN$ ) can be related to the total strain energy release rate ( $G_{max}$ ) using an empirical power-law relationship (ie Paris Equation).

### 3.5.3 Mixed-Mode I/II

The problems associated with mode I and mode II loading configurations, such as inelastic material behaviour, large displacement effects, frictional forces in the form of abrasion between upper and lower fracture surfaces are just as pertinent, if not more so, to mixed-mode configurations. The work on the following test geometries can be considered still in its infancy, despite the claims of their authors. Further research into measurement techniques for monitoring the onset and progress of delamination is required. This comment also applies to precracking with particular emphasis on quantifying geometrical nonlinearities.

**Mixed-Mode Flexure (MMF):** This test consists of clamping one end of a DCB specimen and applying load to one of the adherends. The ratios of modes ( $G_I/G_{II}$ ) is varied by using adherends of different bending stiffnesses (ie altering the thickness ratio of the two adherends). A schematic of the loading configuration is shown in Figure 27. Test specimen dimensions and fabrication are similar to those used for DCB and ENF specimens. A special fixture is required.

**Crack Lap Shear (CLS):** This method enables the magnitude of the shear and peel stress components to be varied, by changing the relative thickness of strap and lap adherends (Figure 27). Load is applied axially. Failure is mode II dominated with ( $G_I/G_{II}$ ) ratios ranging from 20 to 35% [7]. The specimen can be loaded in either tension or flexure (four-point bend). Mode I component increases when the CLS specimen is subjected to pure

bending, also the energy release rate is independent of crack length.

### 3.6 SUMMARY

#### 3.6.1 Tension

##### Butt Joint

| Advantages                                                                                                                                                                                                                                     | Disadvantages                                                                                                                                                                                                                                                                                                            |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields tension, torsion and compression<br>- stiffness / strength<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ISO / ASTM standards<br>Suitable for cyclic / environmental testing<br>(QA only) | Qualitative adhesive property data only<br>Strain measurements difficult<br>High shear and peel stresses at bondline edges<br>Strength sensitive to spew fillet<br>Sensitive to specimen misalignment<br>Limited to thick and rigid adherends<br>Failure attributed to peel stresses<br>Special glueing fixture required |

#### 3.6.2 Peel

##### T-Peel

| Advantages                                                                                                                                                                                                                                                                            | Disadvantages                                                                                                                                                       |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields peel strength<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>Discriminates between pretreatments and adhesives<br>ISO / ASTM standards<br>Suitable for in-situ environmental testing<br>(QA only)<br>Suitable for fatigue testing | Limited to thin flexible adherends<br>Not suitable for generating design data<br>Large uncertainties in measurements<br>No allowance for large adherend deformation |

##### Climbing Drum

| Advantages                                                                                                                                                                                                        | Disadvantages                                                                                                                                                                                                                                                                                       |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields<br>- peel torque<br>- skin (facing) stiffness of honeycomb sandwiches<br>Laminated or honeycomb sandwich structures<br>Straightforward / economic<br>- testing<br>- data reduction<br>ISO / ASTM standards | Special test fixture required<br>Limited to<br>- laminated or honeycomb sandwich structures<br>- low stiffness skins (facings)<br>Large and expensive specimens<br>Comparative data / service correlation unknown<br>Unsuitable for fatigue testing<br>Unsuitable for in-situ environmental testing |

### Floating Roller Method

| Advantages                                                                                                                                                                               | Disadvantages                                                                                                                                                                                                                                              |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields peel strength<br>Compatible with metals and PMCs<br>Straightforward / economic<br>- specimen fabrication (moderate cost)<br>- testing<br>- data reduction<br>ISO / ASTM standards | Special test fixture required<br>Limited to rigid-to-flexible adherends<br>No allowance for large adherend deformation<br>Comparative data / service correlation unknown<br>Unsuitable for fatigue testing<br>Unsuitable for in-situ environmental testing |

### 3.6.3 Cleavage

#### Wedge Cleavage (Boeing Wedge)

| Advantages                                                                                                                                                                                                                                                                         | Disadvantages                                                                                                                                                                       |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Self-stressed<br>Yields fracture strength<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>Accurate and highly reproducible data<br>Sensitive to environmental effects<br>ASTM standard<br>Suitable for environmental testing (QA only) | Limited to rigid materials<br>Not suitable for generating design data<br>Limited to rigid adherends<br>No allowance for large adherend deformation<br>Unsuitable for cyclic loading |

#### Cleavage Strength (Compact Tension)

| Advantages                                                                                                                                                                                                                                      | Disadvantages                                                                                                                                              |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields fracture strength<br>Compatible with metals and PMCs<br>Straightforward / economic<br>- specimen fabrication (moderate cost)<br>- testing<br>- data reduction<br>ASTM standard<br>Suitable for environmental / fatigue testing (QA only) | Special test fixture required (moderate cost)<br>Special glueing fixture required<br>Limited to rigid adherends<br>Not suitable for generating design data |

## 3.6.4 Shear

## Single/Double-Lap Joints

| Advantages                                                                                                                                                                                                                                                 | Disadvantages                                                                                                                                                                                                                                                                                          |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields "apparent" shear strength<br>Compatible with metals, plastics and PMCs<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ISO / ASTM standards<br>Suitable for cyclic / environmental testing<br>(QA only) | Geometry dependent<br>Limited to rigid adherends<br>Not suitable for generating design data<br>Elevated shear and peel stresses at bondline ends<br>Moderate to high bending moments<br>Failure attributed to peel stresses<br>Special glueing fixture required<br>Large uncertainties in measurements |

## V-Notched Beam

| Advantages                                                                                                                                                                                                                                                                                                                                                                   | Disadvantages                                                                                                                                                                                                                                                                                                                                                                                                  |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <u>Bulk Resin</u><br>Shear modulus attainable for all polymeric materials<br>Shear strength attainable for thermoplastics<br><u>Adhesive Joints</u><br>Yields shear strength<br><u>Additional Points</u><br>Low material requirements<br>Data reduction is straightforward<br>Suitable for use under environmental conditions<br>Suitable for creep testing<br>ASTM standard | <u>Bulk Resin</u><br>Strain gauges (2 biaxial rosettes) required<br>Brittle polymers - tensile failure occurs<br><u>Adhesive Joints</u><br>Special glueing fixture required<br>Interfacial tensile failure occurs<br>Non-uniform shear stress state<br><u>Additional Points</u><br>Accurate specimen machining required<br>Special test fixture required<br>Unsuitable for use under cyclic loading conditions |

## Arcan

| Advantages                                                                                                                                                                                                                                                                                                                                                                                     | Disadvantages                                                                                                                                                                                                                                                                                                                                                                                                             |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <u>Bulk Resin</u><br>Shear modulus attainable for all polymeric materials<br>Shear strength attainable for thermoplastics<br><u>Adhesive Joints</u><br>Yields shear modulus<br>Suitable for compliant adhesives<br><u>Additional Points</u><br>Low material requirements<br>Data reduction is straightforward<br>Suitable for use under environmental conditions<br>Suitable for creep testing | <u>Bulk Resin</u><br>Strain gauges / extensometer required<br>Brittle polymers - tensile failure<br><u>Adhesive Joints</u><br>Special glueing fixture required<br>Interfacial tensile failure occurs<br>Non-uniform shear stress state<br><u>Additional Points</u><br>Accurate specimen machining required<br>Special test fixture required<br>Unsuitable for use under cyclic loading conditions<br>No existing standard |

**Plate Twist**

| <b>Advantages</b>                                                                                                                                                                                                                                                                                              | <b>Disadvantages</b>                                                                                           |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------|
| Yields shear modulus<br>Compatible with all materials<br>Non-destructive<br>Stress state relatively uniform over entire specimen<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>Currently being assessed as an ISO standard<br>Suitable for environmental testing | No strength data<br>Large specimens required<br>Not suitable for adhesive joints<br>Limited fatigue capability |

**Torsion Rod/Butt**

| <b>Advantages</b>                                                                                                                                                                                                                                                                                                                          | <b>Disadvantages</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <u>Bulk Resin</u><br>Shear modulus attainable<br>Shear strength attainable for thermoplastics<br><u>Adhesive Joints</u><br>Yields shear strength and shear modulus<br>Compatible with metals and PMCs<br><u>Additional Points</u><br>Suitable for use under environmental conditions<br>Suitable for cyclic/creep testing<br>ASTM standard | <u>Bulk Resin</u><br>Strain gauges / extensometers required<br>Brittle polymers - tensile failure occurs<br>Axial strains need to be monitored<br><u>Adhesive Joints</u><br>Special glueing fixture required<br>Non-uniform shear stress state<br>Small strains - difficult to measure accurately<br><u>Additional Points</u><br>Accurate specimen machining required<br>Torsion facility required<br>Stress concentrations present at end grips<br>Complex data reduction (non-linear stress / strain) |

**Thick-Adherend**

| <b>Advantages</b>                                                                                                                                                                                                                                                                                                | <b>Disadvantages</b>                                                                                                                                                                                                                        |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields shear strength and shear modulus<br>Compatible with all metals, plastics and PMCs<br>Stress state relatively uniform over bondline<br>Straightforward / economic<br>- specimen fabrication (moderate cost)<br>- testing<br>- data reduction<br>ISO / ASTM standards<br>Suitable for environmental testing | Complex / expensive extensometers (2 off) required<br>Special glueing fixture required<br>Accurate specimen machining required<br>Stress concentrations present at bondline ends<br>Stress analysis difficult<br>Limited fatigue capability |

**Napkin Ring**

| <b>Advantages</b>                                                                                                                                                                                                                                                                    | <b>Disadvantages</b>                                                                                                                                                                                                                                                        |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields shear strength and shear modulus<br>Compatible with metals and PMCs<br>Stress state relatively uniform over bondline<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ASTM standard<br>Suitable for cyclic / environmental testing | Extensometer required<br>Accurate specimen machining required<br>Small strains - difficult to measure<br>Torsion facility required<br>Special glueing fixture required<br>Bondline thickness difficult to control<br>Stress analysis difficult (non-linear stress / strain) |

**Skin-Doubler**

| <b>Advantages</b>                                                                                                                                                                                                                                                                                                                           | <b>Disadvantages</b>                                                                                                                                                                                              |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields shear strength and shear modulus<br>Compatible with all metals, plastics and PMCs<br>Stress state relatively uniform over bondline<br>Straightforward / economic<br>- specimen fabrication (moderate cost)<br>- testing<br>- data reduction<br>Representative of actual structures<br>Suitable for environmental and fatigue testing | Complex / expensive extensometers (2 off) required<br>Geometry dependent<br>Large specimens required<br>Limited to rigid adherends<br>Elevated shear and peel stresses at bondline ends<br>No standards available |

### 3.6.5 Mode I Fracture Toughness

#### Double Cantilever Beam (DCB)

| Advantages                                                                                                                                                                                                                             | Disadvantages                                                                                                                                                                                                                                                                                                                                   |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields mode I fracture toughness<br>Compatible with metals and PMCs<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ASTM standard<br>Suitable for cyclic / environmental testing (QA only) | Limited to rigid adherends<br>Loading tabs and test fixture required<br>Limited ability for generating design data<br>Analysis required to account for<br>- end (loading) block tilting<br>- large beam deflection<br>Non-linear load-displacement<br>Crack extension measurements difficult<br>Moderate to large uncertainties in measurements |

#### Tapered Double Cantilever Beam (TDCB)

| Advantages                                                                                                                                                                                                                                                                          | Disadvantages                                                                                                                                                                                                             |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields mode I fracture toughness<br>Compatible with metals<br>Adaptable to thin adherends<br>Constant compliance<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ASTM standard<br>Suitable for cyclic / environmental testing (QA only) | Limited to rigid adherends<br>Large specimens required<br>Not suitable for generating design data<br>Special test fixture required<br>Special glueing fixture required<br>Moderate to large uncertainties in measurements |

### 3.6.6 Mode II Fracture Toughness

#### End Notched Flexure (ENF)

| Advantages                                                                                                                                                                                                                                       | Disadvantages                                                                                                                                                                                                                                                                                                                                                           |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Yields mode II fracture toughness<br>Compatible with metals and PMCs<br>Straightforward / economic<br>- specimen fabrication<br>- testing<br>- data reduction<br>ASTM standard for PMCs<br>Suitable for cyclic / environmental testing (QA only) | Limited to rigid adherends and brittle adhesives<br>Flexural test fixture required<br>Unsuitable for generating design data<br>Analysis required to account for<br>- shear deformation<br>- large beam deflection<br>Non-linear load-displacement<br>Precracking difficult<br>Crack extension measurements difficult<br>Moderate to large uncertainties in measurements |

## 4. ENVIRONMENTAL CONDITIONING

In order to determine the effectiveness of different adhesive systems, processing variables and surface pretreatments, it is necessary to expose adhesively bonded joints to various environmental and loading conditions that simulate actual service conditions. The resistance of the bonded structure to degradation agents often becomes apparent within a short period. In some circumstances only a few hours of exposure may lead to catastrophic failure or seriously compromise the structural integrity of the joint. Environmental considerations are paramount in the design of adhesively bonded structures. The two predominant factors in climatic exposure are humidity and temperature. The severity of these two factors will depend on geographical location, and need to be taken into account in assessing strength allowables. A list of service environmental conditions are listed below:

- Static heat ageing or sub-zero exposure.
- Humidity (inc. hot / wet) exposures.
- Complete immersion in water at ambient and elevated temperatures.
- Freeze / thaw and dry / wet cyclic conditions.
- Continuous or intermittent saltwater immersion or spray.
- Combined load (ie stress) and environmental exposures.
- Solvents (inc. paint strippers).
- Acid and alkali solutions.
- Diesel and engine oils.

Engineering structures are exposed to various combinations of these environments in service, often resulting in complex synergistic degradation of the material. A listing of fatigue, creep and environmental standards can be found in Appendix I. Appendix II provides a comprehensive summary of environmental conditioning procedures, covering the scope of the standard, relevant materials, environmental conditions and specimen requirements. The reader is directed towards reference [7], which provides an in-depth review of chemical and radiation effects on adhesive joint performance. This section will briefly examine the effect of elevated temperature and moisture on adhesive joints, and accelerated ageing methods. Further information on moisture and chemical resistance of adhesives and adherends is provided in Sections 6 and 7, respectively.

### 4.1 DEGRADATION AGENTS

#### 4.1.1 Temperature

Prolonged, or even short term, exposure to elevated temperatures will often produce irreversible chemical and physical changes within adhesives. As the temperature increases, the bond strength decreases. All adhesives degrade at elevated temperatures. The rate of degradation increases with the amount of oxygen present. In adhesively bonded joints, the primary path for oxygen diffusion is through the adhesive, which can be relatively rapid at elevated temperatures. Oxidation is the primary degradation process at elevated temperatures. Metallic adherends are impermeable to oxygen, thereby providing a barrier to the gas. In contrast, polymer matrix composites (PMCs) are permeable to atmospheric gases, and hence the rate of degradation can be expected to be higher.

The use of anti-oxidant additives in adhesives slows the degradation process by scavenging any free radicals. High temperature resistant adhesives (eg epoxy-phenolic) were developed

to resist thermal oxidation. The use of anti-oxidant additives in adhesives slows the degradation process by scavenging any free radicals. Elevated temperatures also depolymerise adhesives and degrade the bondline interface. Differences in coefficients of thermal expansion between adhesive and adherend can induce residual stresses, thereby reducing bond efficiency.

#### **4.1.2 Moisture**

Moisture (water) degradation is probably the major cause of in-service failure in bonded structures. The ubiquitous nature of water combined with the ability to penetrate into the adhesive structure poses considerable problems. With good design it is possible to prevent moisture diffusing to the adhesive/adherend interfaces. This problem is further exacerbated at elevated temperatures and mechanical stress. Hot and humid environments can often cause rapid loss of strength in metal-epoxy joints within a short duration (ie 2 years) with catastrophic consequences. Failure invariably occurring at the adhesive/adherend interface. The major cause of strength loss in adhesively bonded joints is associated with interfacial degradation through water-substrate interaction rather than weakening of the adhesive. Water can degrade the strength of adhesive joints through hydration of metal oxide layers. Corrosion products, such as rust, at the interface is considered a post-failure phenomenon. The presence of water in epoxy adhesives results in plasticisation (essentially softening) of the polymeric material. At temperatures below the glass transition temperature ( $T_G$ ), polymer property reduction is reversible upon dehydration, whereas above  $T_G$ , matrix properties are permanently degraded. Surface treatments, such as phosphoric acid anodisation and organosilane primer coatings will bestow joints with improved moisture resistance.

### **4.2 ACCELERATED CONDITIONING**

The degree of degradation that occurs in adhesive structures when in service is linked directly with the amount of moisture absorbed. The moisture absorption kinetics of polymer systems differ widely and also change with physical ageing. The approach being adopted, is to define a constant relative humidity environment that will produce a moisture level that is representative of an bonded structure that has been exposed to a real life environment at a particular geographical location. The variability of a natural environment, that is daily, monthly or seasonal changes in temperature and humidity are known to a major factor in determining both the final moisture equilibrium level in the materials bulk and the distribution of moisture in the outer surface layers. The military consider the worst worldwide environment to be represented by a relative humidity of 85% and a temperature of 60°C.

The natural process of moisture absorption in adhesive structures is normally very slow, and this makes it very difficult to reach an adequate degree of degradation in a structural test element in practical timescales. It has been found necessary, therefore, to speed up the moisture diffusion process by employing an accelerated conditioning technique that can ensure a representative level of degradation in a significantly reduced time.

The usual approach used to accelerate moisture uptake is to increase the diffusivity of the adhesive by elevating the temperature of the conditioning environment [67]. The diffusion coefficient of Ciba Geigy 914 epoxy resin system increases by a factor of 5 for an increase in temperature from 20°C to 60°C. This increase in diffusion rate is still slow, and ideally an increase in temperature to further accelerate the conditioning would be an advantage. However, there is a temperature limit to which most epoxy resins can be raised without

affecting a change in the mechanism by which moisture is absorbed. Above this critical temperature, there is a strong possibility that the degradation mechanism is altered. At high temperatures and high humidity, the Fickian model is no longer valid. A maximum safe temperature exists above which moisture diffusion deviates from the classical single phase Fickian model, to one that is a two-phase Langmuir type.

An alternative approach to attempting to reach an equilibrium condition, involves altering the acceptance criteria to a given percentage of the chosen equilibrium condition. Conditioning the material to 95% of the full equilibrium state takes a relatively shorter time to reach than the full (100%) equilibrium condition. The time required to obtain the last 5% can take longer than the time taken to reach the 95% level. Clearly a very large saving in time is possible if a 95% of equilibrium can be justified in terms of a non-significant change in the structural strength and stiffness properties.

Further acceleration can be obtained by increasing the relative humidity to the maximum that can be readily maintained (ie 96%). It is required that the acceleration method takes full advantage of the high rates of diffusion given by temperature and humidity and still achieve a reasonably realistic moisture absorption. It is not satisfactory to precondition at 96% RH, so that the equilibrium level in the outer surface layers exceeds the bulk equilibrium level obtained due to exposure in service.

### **Three-stage conditioning**

The approach is to expose the material to the maximum practical humidity level that is available (ie 96% RH) until the moisture level in the bulk reaches the bulk equilibrium level at the lower humidity / temperature condition. The second stage is to expose the material to a lower humidity level (eg 15% RH), a drying stage, to remove excess moisture in the outer surface layers. The third stage uses an RH that will give the desired final moisture level (ie 84% RH). The distribution flattens out across the entire material thickness. The effective use of this technique relies upon mathematically modelling the moisture diffusion for each stage of conditioning.

Accelerated ageing, is however, an unknown phenomenon and there is no agreement upon a satisfactory accelerated test. Predictive models relating to accelerated ageing are covered in reference [68].

## **5. CONCLUSIONS**

The two most important criteria when selecting a test method are the availability of a standard test method and the ability of the test method to produce consistent and reliable engineering data for a range of service conditions. Most of the test methods assessed in this report have been standardised at either national or international level. These standards generally apply to standard laboratory conditions (ie 23<sup>o</sup>, 50% RH) and are usually limited to providing comparative data on adhesive systems and surface pretreatments, and are unsuitable for generating design data. There is the added problem of insufficient data of reliable pedigree required to determine the degree of uncertainty and the sensitivity of the these techniques. In many cases, an improvement in specimen design would improve performance and versatility, reduce measurement uncertainty and allow the methods to be applied to a wider range of adherend materials.

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Table 1 Tensile and Peel Test Methods

| Test Method                                 | Tensile Butt Joint                         | T-Peel                                                                     | Climbing Drum                                                                     | Floating Roller Method                                                                |
|---------------------------------------------|--------------------------------------------|----------------------------------------------------------------------------|-----------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|
| Shear Properties Obtained                   | Tensile strength/modulus                   | Peel strength                                                              | Peel strength/skin stiffness                                                      | Peel strength                                                                         |
| Material Quantity Requirements per specimen | Low                                        | Low                                                                        | High                                                                              | Low                                                                                   |
| Typical Specimen Dimensions (mm)            | Diameter 15-25<br>Adherend thickness 12-15 | Bond length 150<br>Width 25<br>Adherend thickness 0.5-1.0<br>Arm length 50 | Long adherend 300<br>Short adherend 240<br>Width 25<br>Adherend thickness 0.5-5.0 | Flexible adherend 250<br>Rigid adherend 200<br>Width 25<br>Adherend thickness 0.5-1.6 |
| Materials Suitable for Testing              | 1 - 6                                      | 1, 4 and 6<br>Flexible-flexible adherend                                   | Flexible-rigid adherend<br>1 - 6 + sandwich structures                            | Flexible-rigid adherend<br>1 - 6                                                      |
| Cost of Specimen Fabrication/Preparation    | Low                                        | Low-moderate                                                               | High                                                                              | Low - moderate                                                                        |
| Cost of Testing/Specimen                    | Low                                        | Low                                                                        | Low - moderate                                                                    | Low - moderate                                                                        |
| Specimen Fabrication Equipment Requirements | Surface preparation                        | Surface preparation<br>Bonding+Bonding jig                                 | Surface preparation<br>Bonding+Bonding jig                                        | Surface preparation<br>Bonding+Bonding jig                                            |
| Specimen Instrumentation Requirements       | Extensometer                               | None                                                                       | None                                                                              | Extensometer (2 off)                                                                  |
| Test Equipment and Fixture Requirements     | Universal test machine +<br>end grips +    | Universal test machine +<br>end grips                                      | Special test fixture<br>Universal test machine +<br>end grips                     | Special test<br>fixture<br>Universal test<br>machine + end grips                      |
| Fatigue performance                         | Limited                                    | unsuitable                                                                 | Unsuitable                                                                        | Unsuitable                                                                            |
| Creep Performance                           | Suitable                                   | Possibly                                                                   | Unsuitable                                                                        | Unsuitable                                                                            |
| Environmental suitability                   | Suitable                                   | Suitable                                                                   | Unsuitable                                                                        | Unsuitable                                                                            |
| Data Reduction                              | Straightforward                            | Straightforward                                                            | Straightforward                                                                   | Straightforward                                                                       |
| Accuracy (Estimated)                        | To be determined                           | Large uncertainty (> 30%)                                                  | To be determined                                                                  | To be determined                                                                      |
| Standard Test Methods Available             | ASTM D 897<br>ASTM D 2095<br>BS EN 26922   | ISO 8510: Part 2<br>ISO 11339<br>ASTM 1876                                 | BS 5350: Part C13<br>ASTM D 3167                                                  | DD ENV 1967:1996<br>ASTM D 3167                                                       |

1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

Table 2 Cleavage and Mode I Fracture Toughness Test Methods

| Test Method                                 | Wedge Cleavage                                 | Compact Tension                                | DCB                                               | TDCB                                              |
|---------------------------------------------|------------------------------------------------|------------------------------------------------|---------------------------------------------------|---------------------------------------------------|
| Shear Properties Obtained                   | Fracture energy                                | Cleavage strength                              | Mode I fracture toughness                         | Mode I fracture toughness                         |
| Material Quantity Requirements/Specimen     | Low                                            | high                                           | Low                                               | High                                              |
| Typical Specimen Dimensions (mm)            | Length 200<br>Width 25<br>Adherend thickness 2 | Length 25<br>Width 25<br>Adherend thickness 12 | Length 356<br>Width 25<br>Adherend thickness 6.35 | Length 240<br>Width 25<br>Adherend thickness 12.8 |
| Materials Suitable for Testing              | 1                                              | 1 - 6                                          | 1 - 6                                             | 1 and 6                                           |
| Cost of Specimen Fabrication/Preparation    | Low                                            | Low-moderate                                   | Low                                               | Moderate - High                                   |
| Cost of Testing/Specimen                    | Low                                            | Low-moderate                                   | Low-moderate                                      | Low-moderate                                      |
| Specimen Fabrication Equipment Requirements | Surface preparation                            | Surface preparation<br>Bonding + bonding jig   | Surface preparation                               | Surface preparation<br>Bonding + Bonding jig      |
| Specimen Instrumentation Requirements       | Travelling microscope or<br>video camera       | Extensometer for crack<br>opening displacement | Travelling microscope or<br>video camera          | Travelling microscope or<br>video camera          |
| Test Equipment and Fixture Requirements     | Self-stressed                                  | Universal test machine +<br>loading fixture    | Universal test machine +<br>loading fixture       | Universal test machine +<br>loading fixture       |
| Fatigue performance                         | Not suitable                                   | Suitable                                       | Suitable                                          | Suitable                                          |
| Creep Performance                           | Possibly                                       | Suitable                                       | Unsuitable                                        | Unsuitable                                        |
| Environmental suitability                   | Suitable                                       | Suitable                                       | Suitable                                          | Suitable                                          |
| Data Reduction                              | Straightforward                                | Straightforward                                | Straightforward                                   | Straightforward                                   |
| Accuracy (Estimated)                        | To be determined                               | To be determined                               | Large uncertainty                                 | Moderate                                          |
| Standard Test Methods Available             | ASTM D 3762                                    | BS 5350: Part C1<br>ASTM D 1062                | ASTM D 3433                                       | ASTM D 3433                                       |

1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

Table 3 Shear Test Methods

| Test Method                                 | Single-Lap                                                                     | Double-Lap                                                           | V-Notched Beam                                                  | Arcan                                                           |
|---------------------------------------------|--------------------------------------------------------------------------------|----------------------------------------------------------------------|-----------------------------------------------------------------|-----------------------------------------------------------------|
| Shear Properties Obtained                   | Shear strength                                                                 | Shear strength                                                       | Shear strength/modulus                                          | Shear strength/modulus                                          |
| Material Quantity Requirements/Specimen     | Low                                                                            | Low                                                                  | Low                                                             | Low                                                             |
| Typical Specimen Dimensions (mm)            | Length 100<br>Width 25<br>Adherend thickness 2<br>Overlap length 25            | Length 100<br>Width 25<br>Adherend thickness 12<br>Overlap length 25 | Length 76<br>Width 20<br>Notch width 12<br>Adherend thickness 5 | Length 52<br>Width 40<br>Notch width 12<br>Adherend thickness 6 |
| Materials Suitable for Testing              | 1, 3 and 6                                                                     | 1, 3 and 6                                                           | 1, 3 and 6                                                      | 1, 3 and 6                                                      |
| Cost of Specimen Fabrication/Preparation    | Low                                                                            | Low-moderate                                                         | Moderate                                                        | Moderate                                                        |
| Cost of Testing/Specimen                    | Low                                                                            | Low-moderate                                                         | Low-moderate                                                    | Low-moderate                                                    |
| Specimen Fabrication Equipment Requirements | Surface preparation<br>Bonding + bonding jig                                   | Surface preparation<br>Bonding + bonding jig                         | Surface preparation<br>Bonding + Bonding jig                    | Surface preparation<br>Bonding + Bonding jig                    |
| Specimen Instrumentation Requirements       | None                                                                           | None                                                                 | Shear extensometer<br>Strain gauges                             | Shear extensometer<br>Strain gauges                             |
| Test Equipment and Fixture Requirements     | Universal test machine +<br>loading grips                                      | Universal test machine +<br>loading grips                            | Universal test machine +<br>loading fixture                     | Universal test machine +<br>loading fixture                     |
| Fatigue performance                         | Limited                                                                        | Suitable                                                             | Unsuitable                                                      | Suitable                                                        |
| Creep Performance                           | Suitable                                                                       | Possibly                                                             | Unsuitable                                                      | Unsuitable                                                      |
| Environmental suitability                   | Suitable                                                                       | Suitable                                                             | Suitable                                                        | Suitable                                                        |
| Data Reduction                              | Straightforward                                                                | Straightforward                                                      | Straightforward                                                 | Straightforward                                                 |
| Accuracy (Estimated)                        | Moderate                                                                       | Low (30%)                                                            | Low - moderate (10-20%)                                         | Low - moderate (10-20%)                                         |
| Standard Test Methods Available             | BS 5350: Part C5<br>BS EN 1465<br>BS EN ISO 9664<br>ASTM D 1002<br>ASTM D 3166 | BS 5350: Part C5<br>BS EN ISO 9664<br>ASTM D 1002<br>ASTM D 3166     | No adhesive standard<br>ASTM D 5379 -PMCs                       | None                                                            |

1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

Table 4 Shear and Mode II Fracture Toughness Test Methods

| Test Method                                 | Thick Adherend                                                     | Torsion Butt Joint                           | Napkin Ring                                  | ENF                                            |
|---------------------------------------------|--------------------------------------------------------------------|----------------------------------------------|----------------------------------------------|------------------------------------------------|
| Shear Properties Obtained                   | Shear strength/modulus                                             | Shear strength/modulus                       | Shear strength                               | Mode II fracture energies                      |
| Material Quantity Requirements/Specimen     | Low                                                                | Low                                          | Low                                          | Low                                            |
| Typical Specimen Dimensions (mm)            | Length 110<br>Width 5<br>Adherend thickness 26<br>Overlap length 5 | Diameter 15-25<br>Adherend thickness 12-15   |                                              | Length 356<br>Width 25<br>Adherend thickness 6 |
| Materials Suitable for Testing              | 1-6                                                                | 1-6                                          | 1-6                                          | 1 and 6                                        |
| Cost of Specimen Fabrication/Preparation    | Low-moderate                                                       | Low-moderate                                 | Moderate-high                                | Low                                            |
| Cost of Testing/Specimen                    | Low                                                                | Low-moderate                                 | Low-moderate                                 | Low-moderate                                   |
| Specimen Fabrication Equipment Requirements | Surface preparation<br>Bonding = bonding jig                       | Surface preparation<br>Bonding + bonding jig | Surface preparation<br>Bonding + bonding jig | Surface preparation<br>Bonding + Bonding jig   |
| Specimen Instrumentation Requirements       | Extensometers (2 off)                                              | Rotary extensometer                          | Rotary extensometer                          | Travelling microscope or<br>video camera       |
| Test Equipment and Fixture Requirements     | Self-stressed                                                      | Universal test machine +<br>loading fixture  | Torsion test machine +<br>loading fixture    | Universal test machine +<br>flexure fixture    |
| Fatigue performance                         | Not suitable                                                       | Suitable                                     | Suitable                                     | Suitable                                       |
| Creep Performance                           | Possibly                                                           | Suitable                                     | Unsuitable                                   | Unsuitable                                     |
| Environmental suitability                   | Suitable                                                           | Suitable                                     | Suitable                                     | Suitable                                       |
| Data Reduction                              | Straightforward                                                    | Straightforward                              | Straightforward                              | Straightforward                                |
| Accuracy (Estimated)                        | To be determined                                                   | To be determined                             | To be determined                             | Large uncertainty (30%)                        |
| Standard Test Methods Available             | ISO 11003<br>ASTM D 3165                                           | None                                         | ASTM E 229                                   | None                                           |

1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

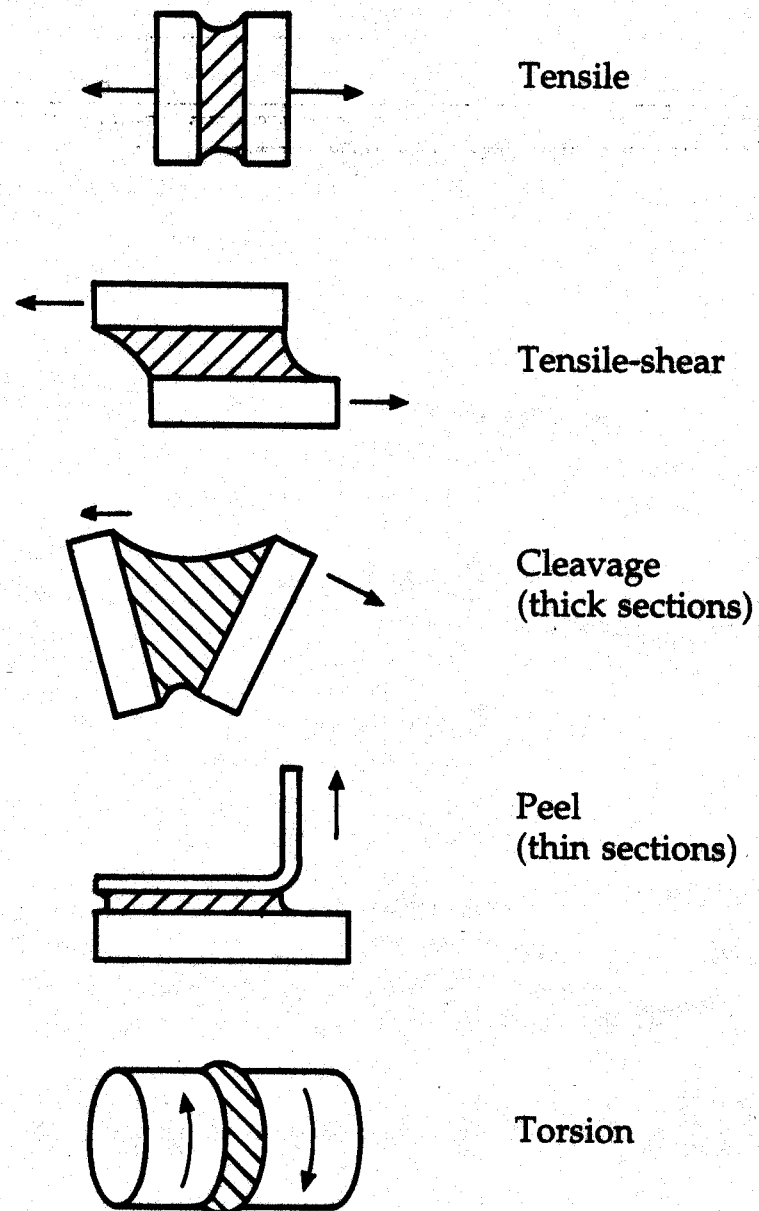


Figure 1 Basic loading modes experienced by adhesive joints.

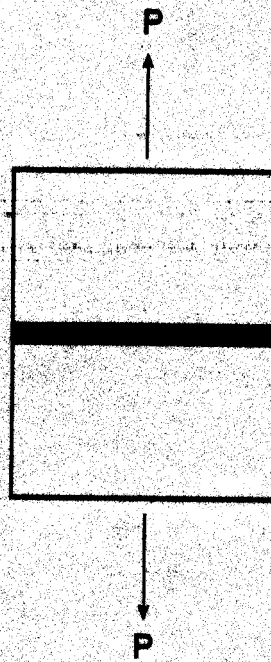


Figure 2 Tensile butt joint.

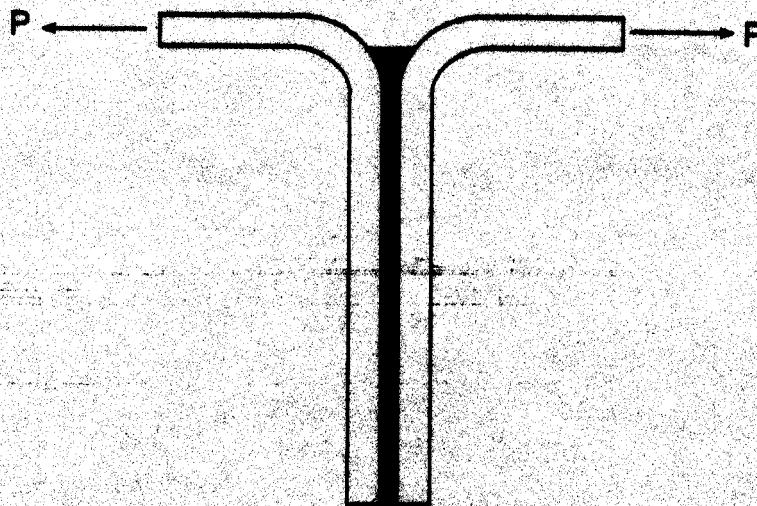


Figure 3 T-peel test.

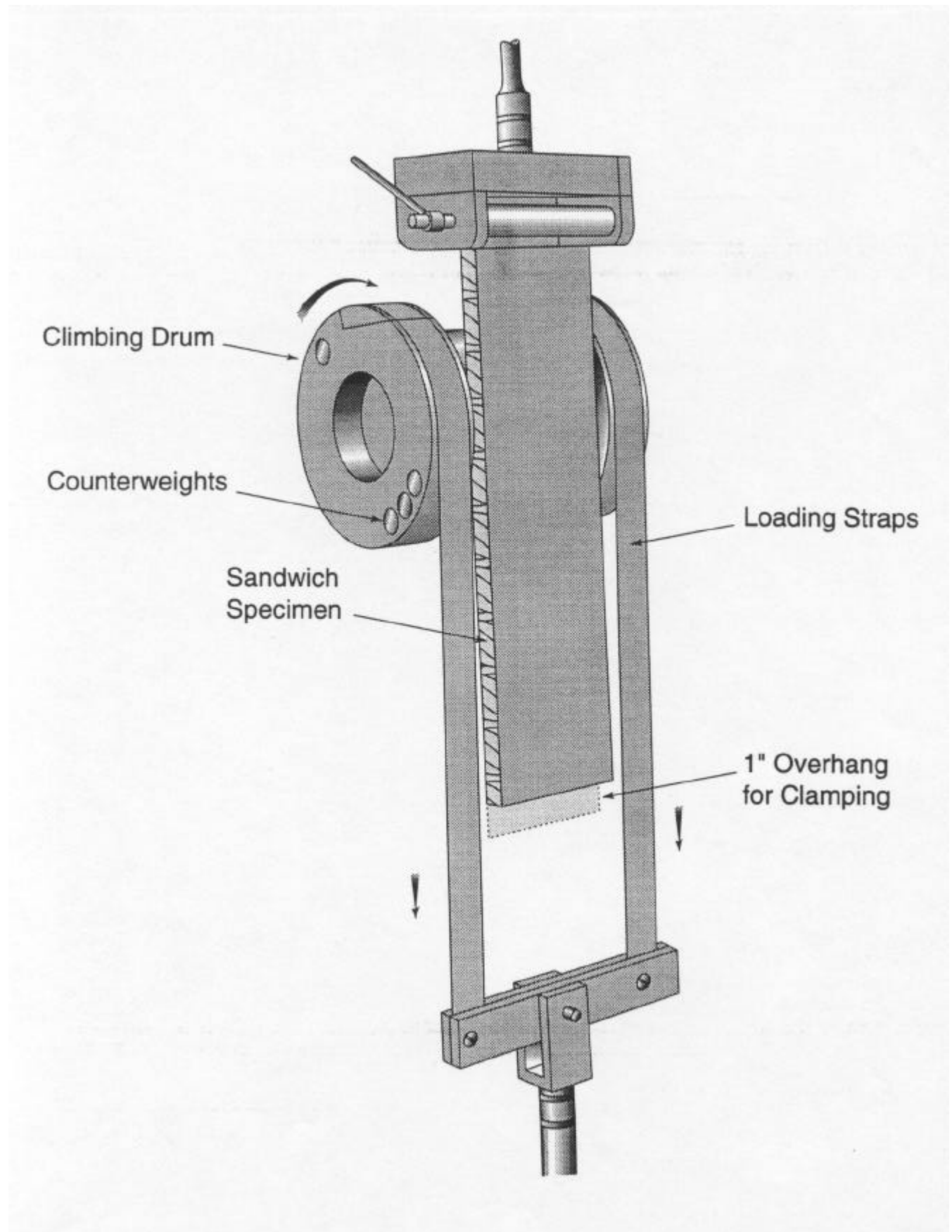


Figure 4 Climbing drum test with sandwich specimen.

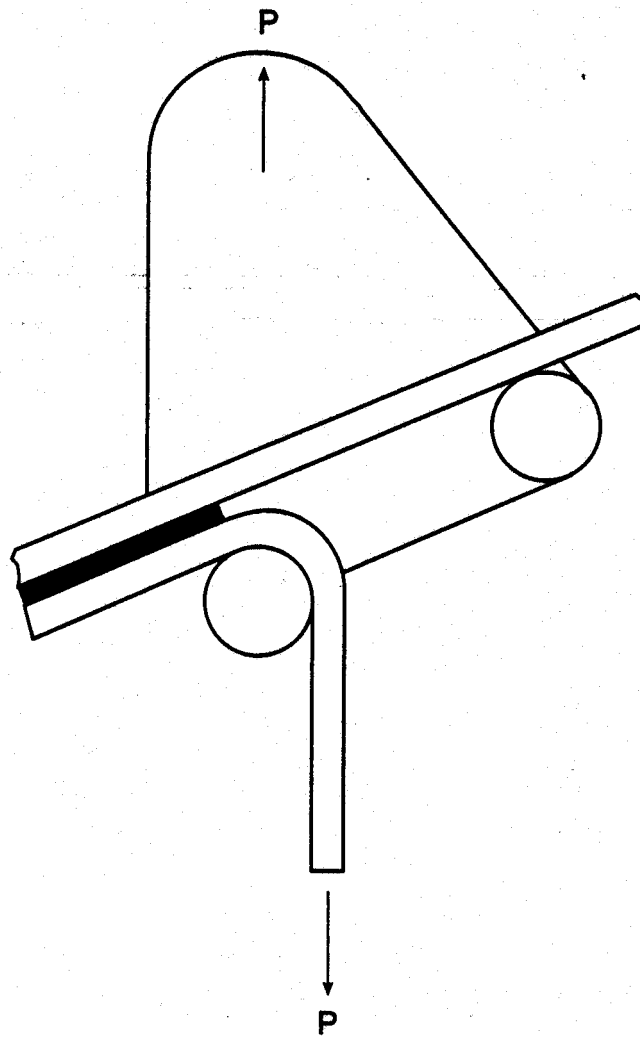


Figure 5 Floating roller method.

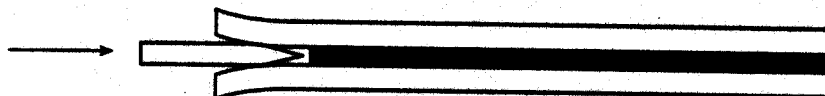


Figure 6 Cleavage strength (compact tension) test.

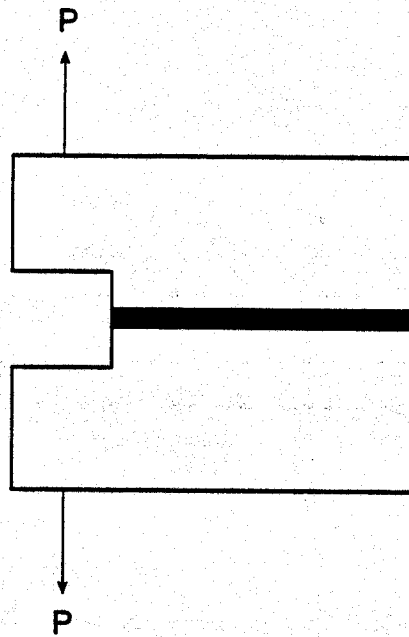


Figure 7 Cleavage strength (compact tension) test.

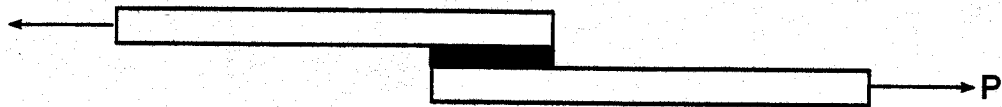


Figure 8 Single-lap joint.

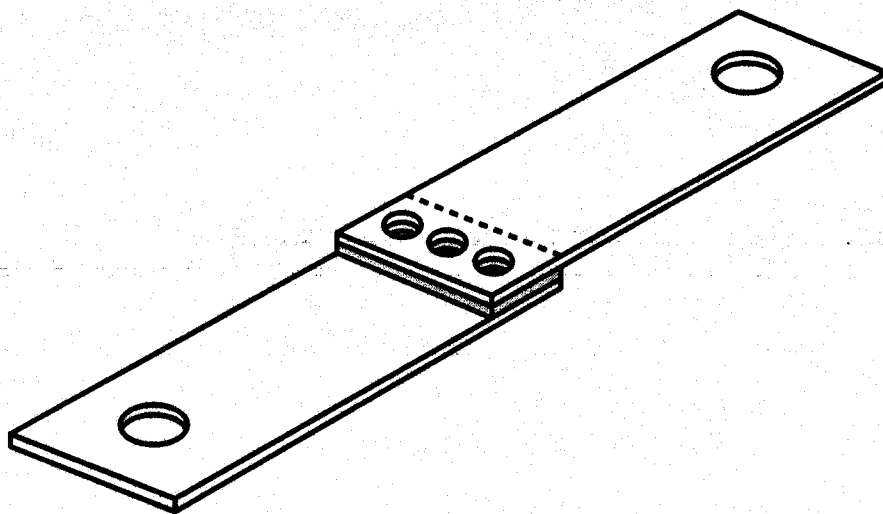


Figure 9 Perforated single-lap joint.

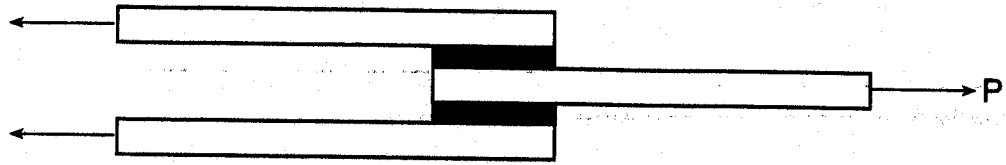


Figure 10 Double-lap joint.

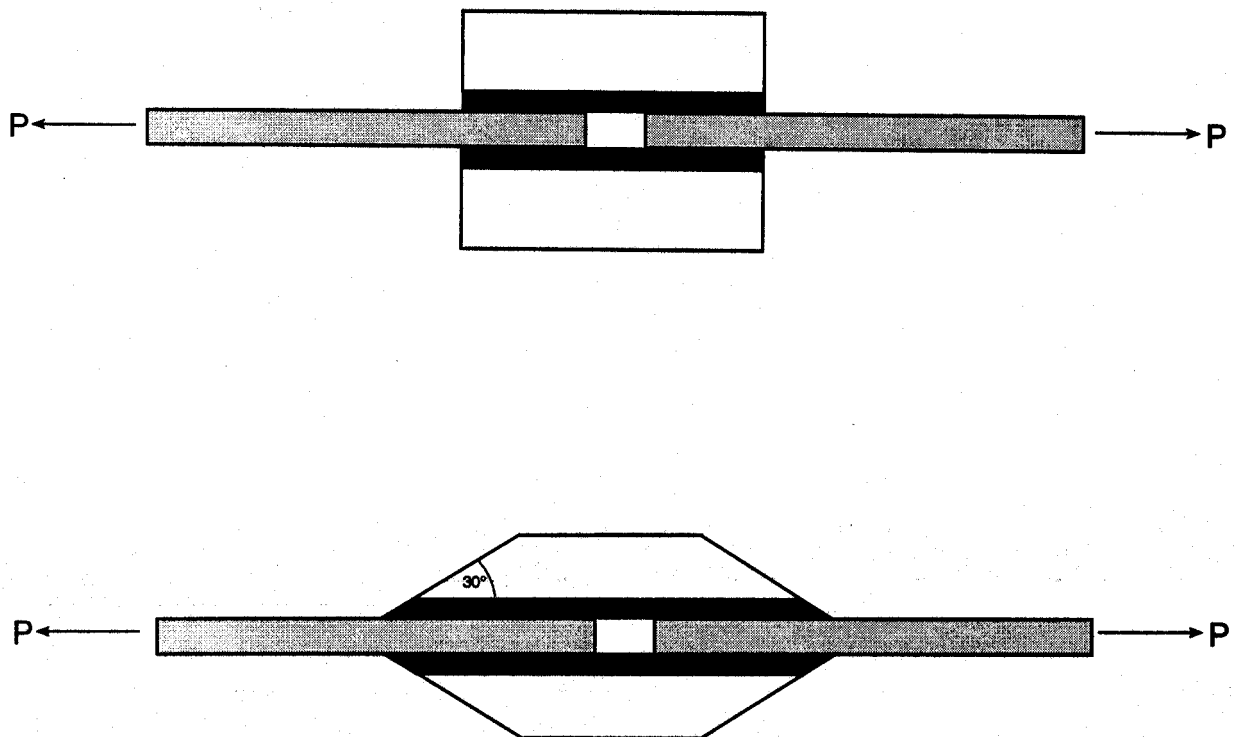


Figure 11 Double-strap and tapered double-strap joints.

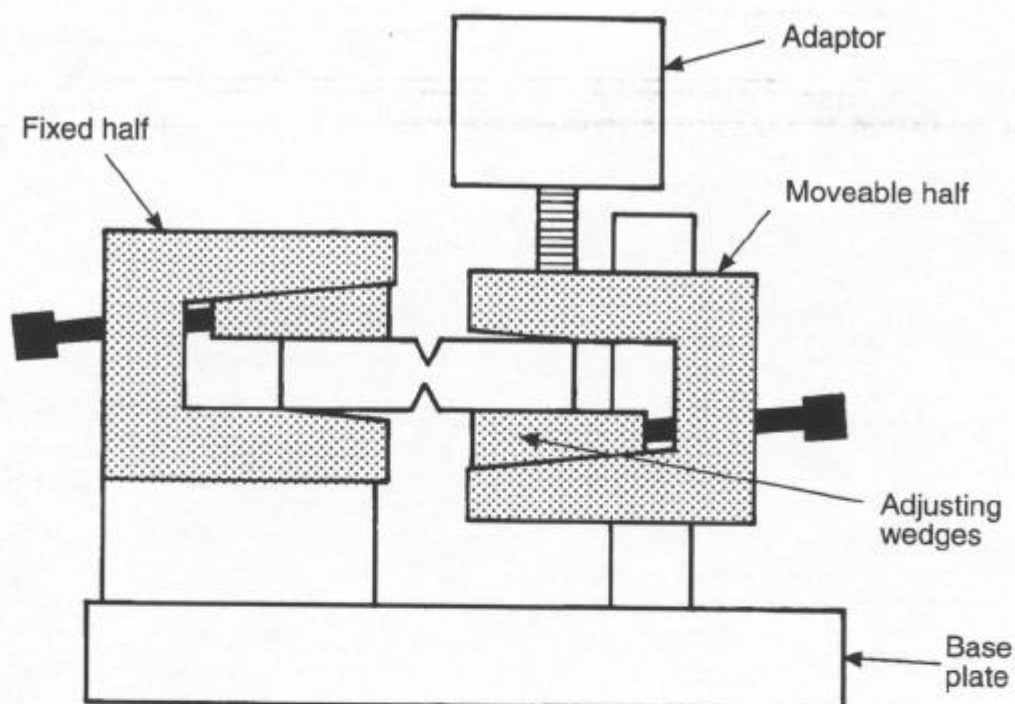
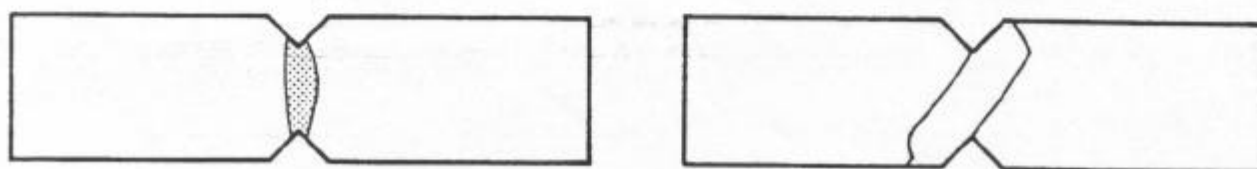


Figure 12 V-notched beam test fixture and specimen.



Thermoplastic - Shear Yielding

Thermoset - Brittle Tensile

Figure 13 Typical failure modes for V-notched beam test.

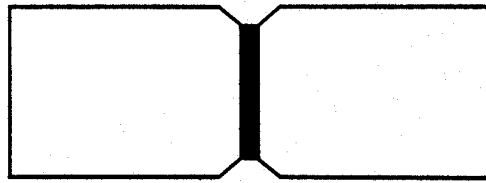


Figure 14 V-notched beam joint specimen.

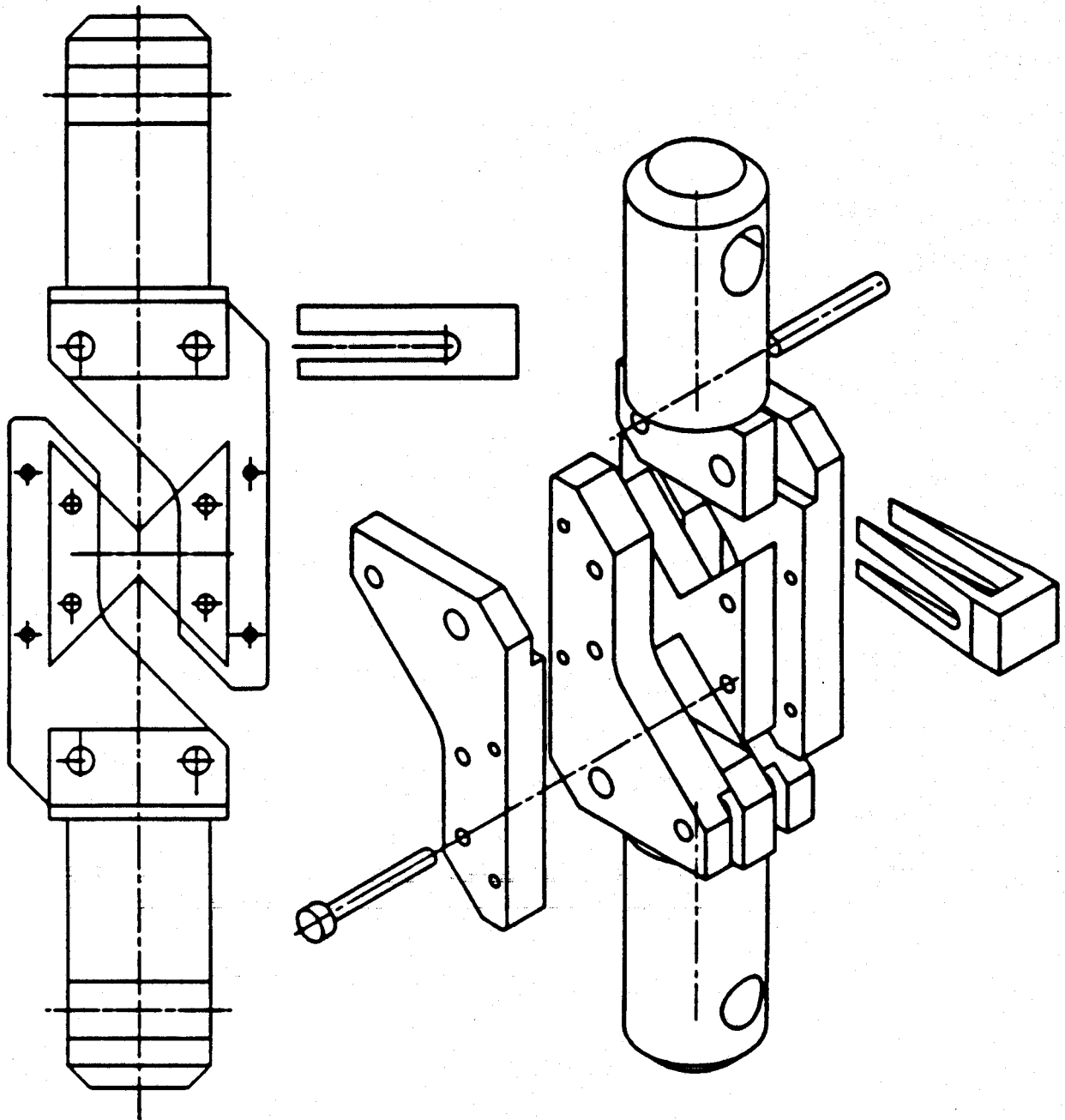


Figure 15 Arcan loading assembly and bulk test specimen [38].

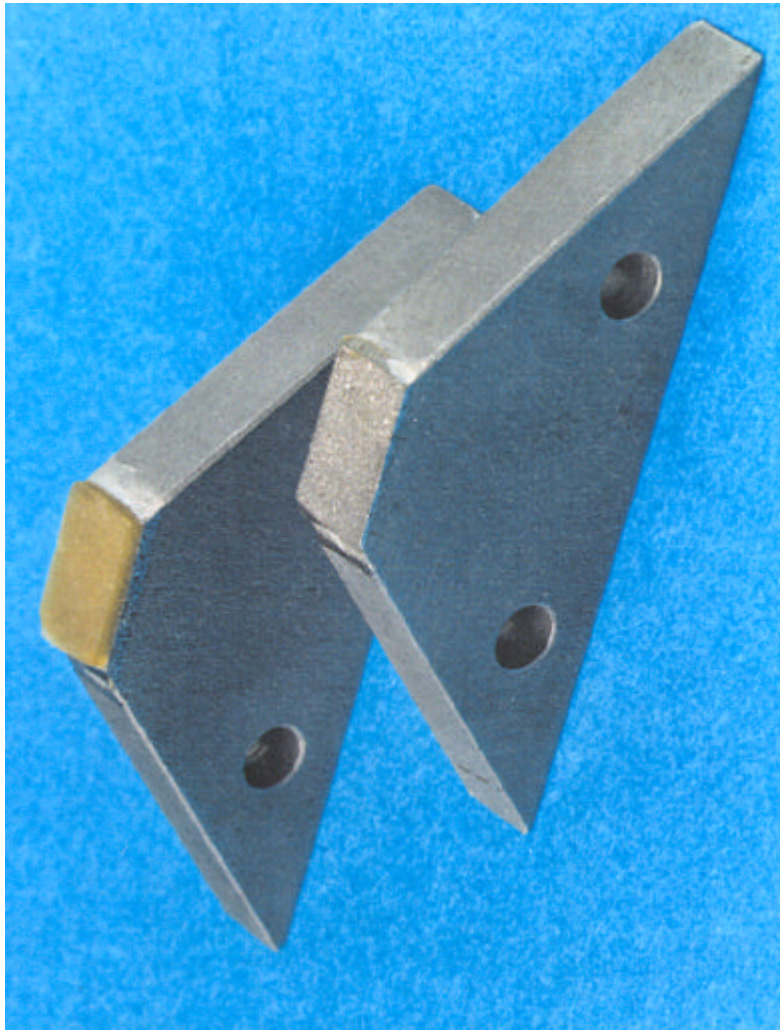


Figure 16 Arcan joint specimen [38].

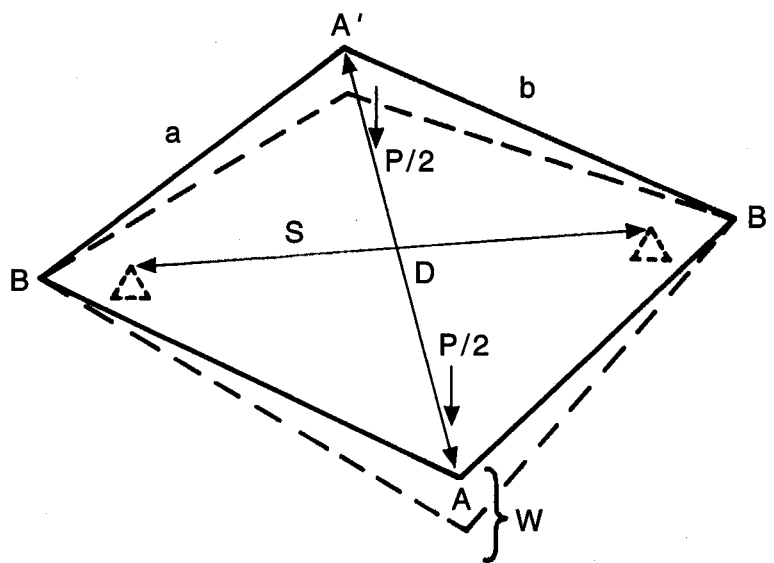


Figure 17 Plate-twist test.

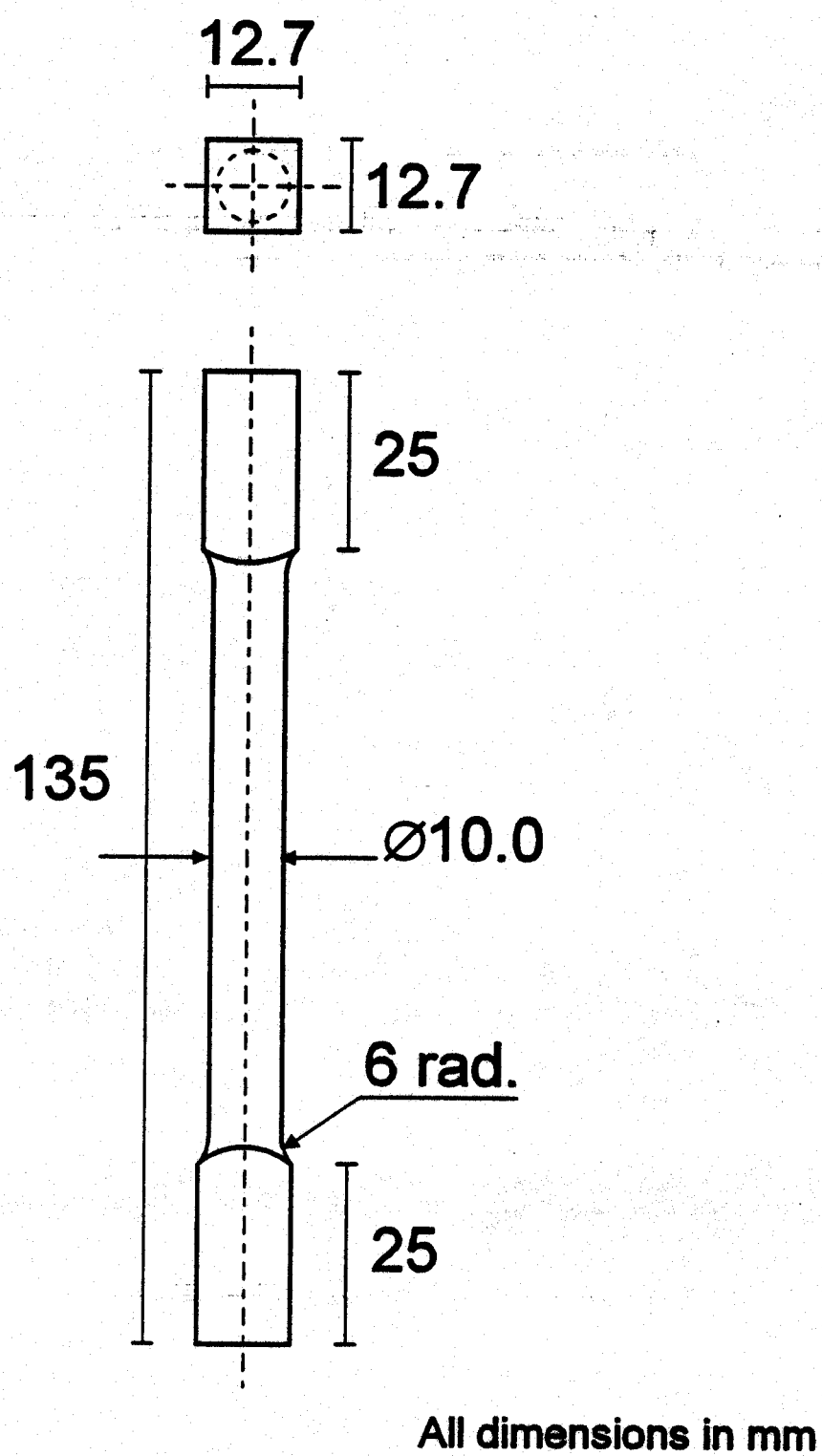


Figure 18 Bulk torsion specimen [55].

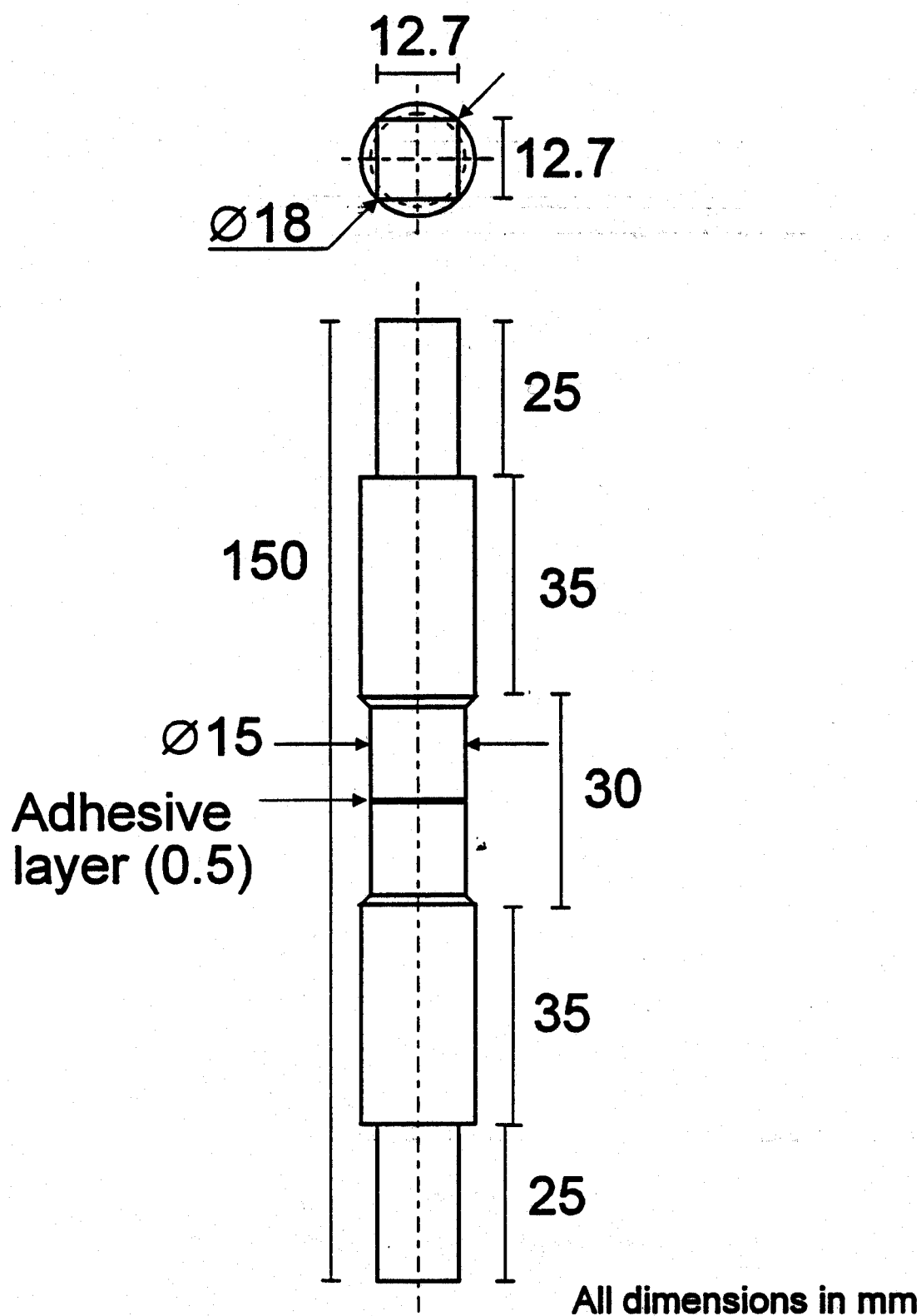


Figure 19 Butt torsion specimen [55].

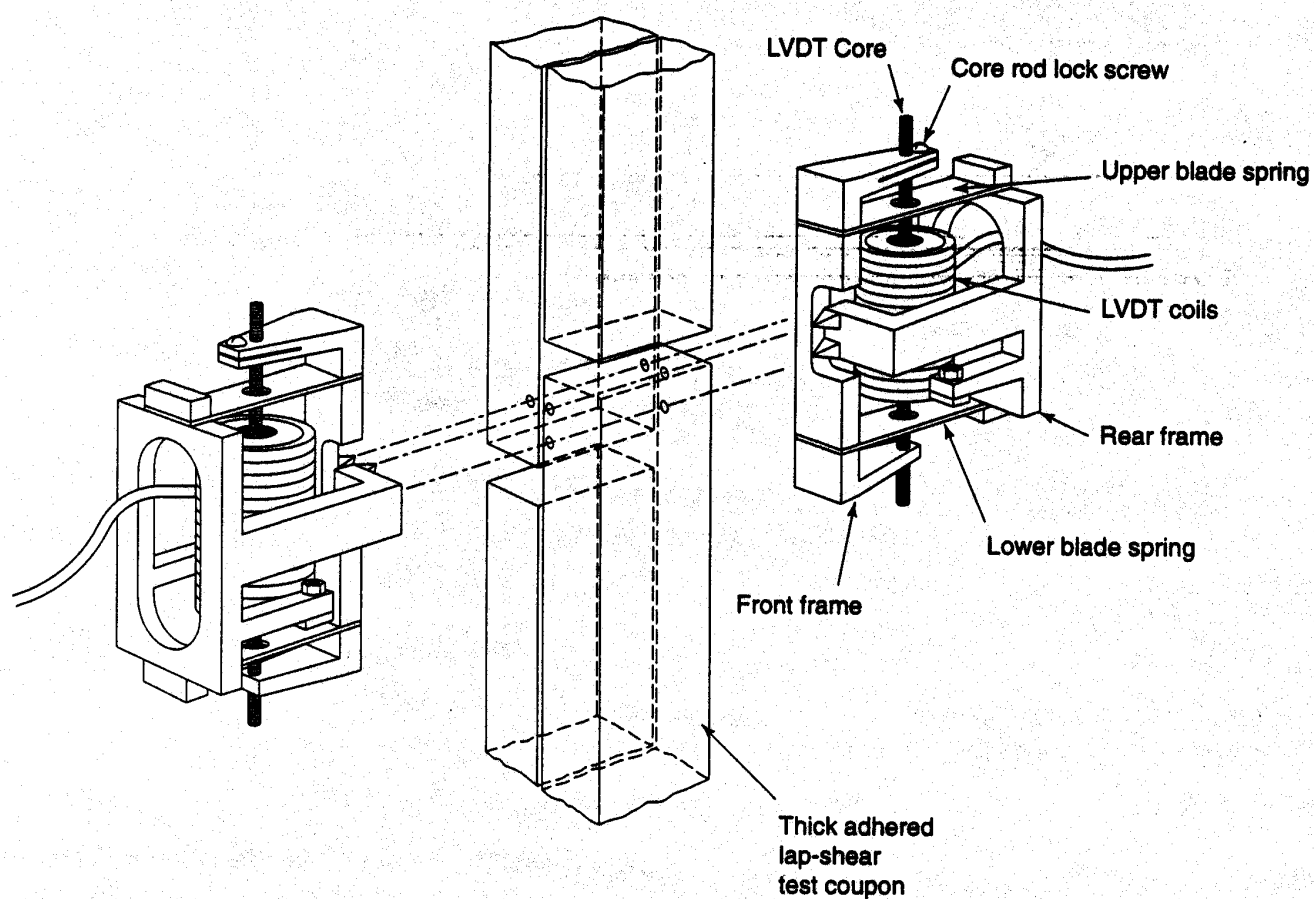


Figure 20 Thick-adherend test specimen.

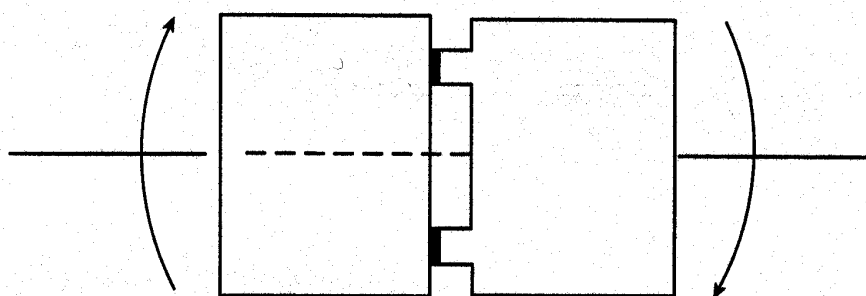


Figure 21 Napkin ring specimen.

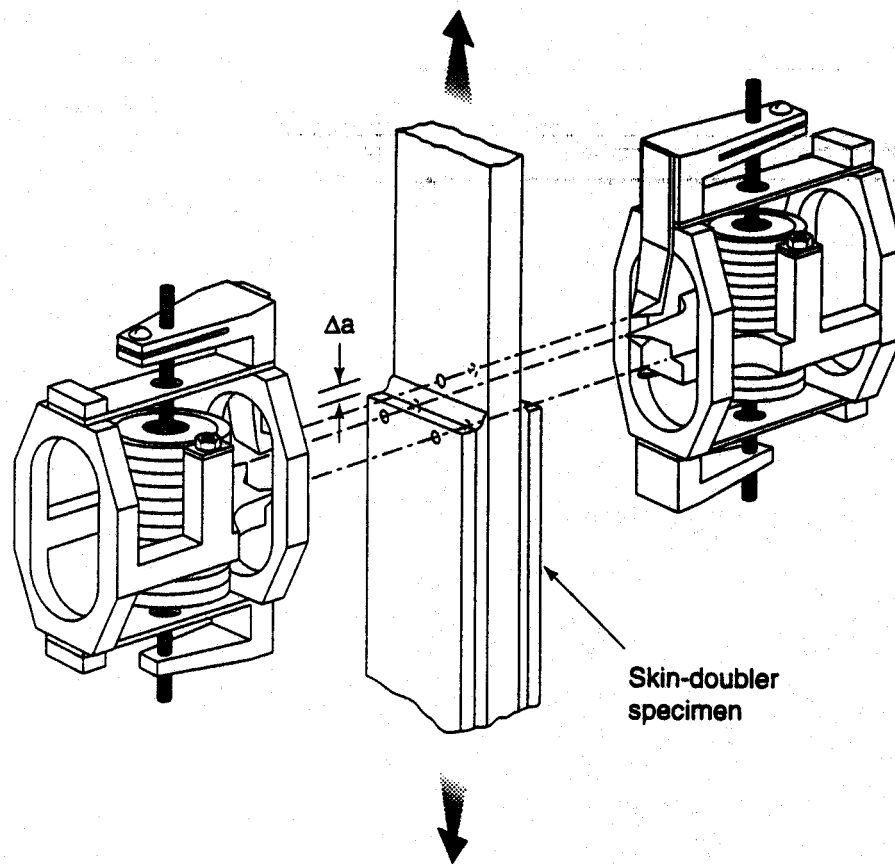


Figure 22 Skin-doubler test.

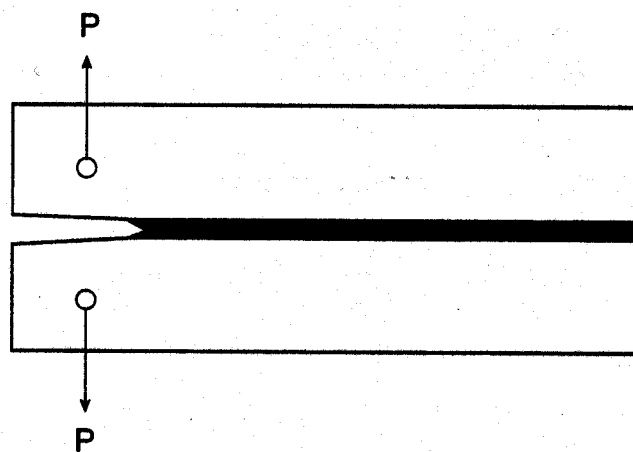


Figure 23 Double cantilever beam test.

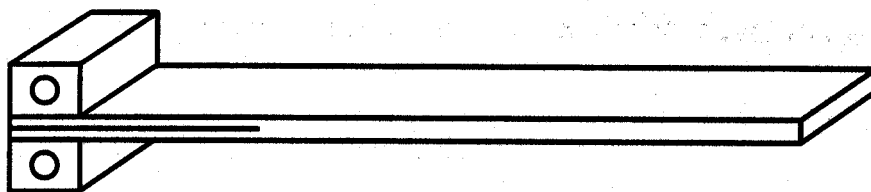


Figure 24 Double cantilever beam test configuration for testing thin adherends.

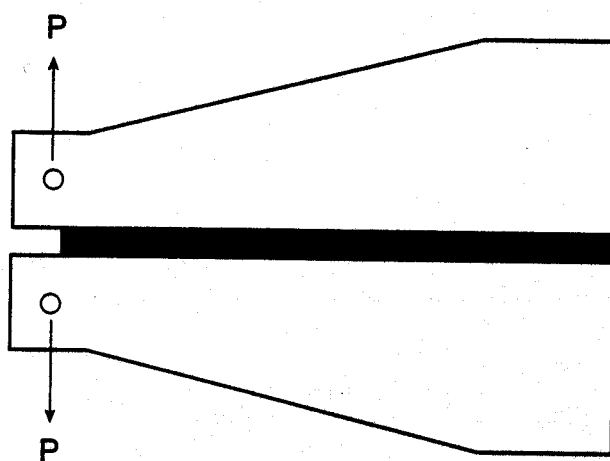


Figure 25 Tapered double cantilever beam test.

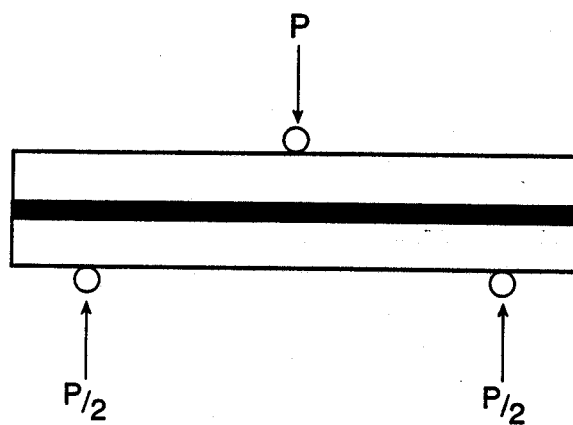


Figure 26 End-notched flexure test.

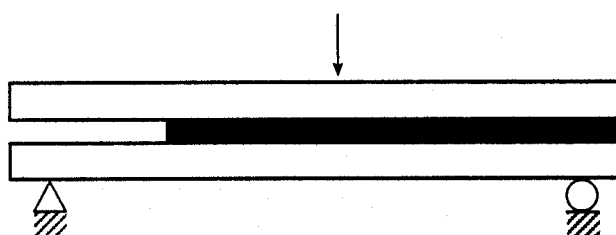


Figure 27 Mixed mode flexure configuration.

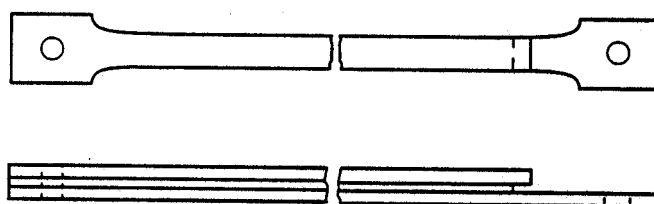


Figure 28 Crack lap shear configuration.

**APPENDIX I - MECHANICAL TEST METHOD STANDARDS****Table 1 ISO Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>                              | <b>TITLE</b>                                                                                                                              | <b>PURPOSE</b>    |
|--------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------|-------------------|
| ISO 11343: 1993                            | Adhesives - Determination of dynamic resistance to cleavage of high-strength adhesive bonds under impact conditions - wedge impact method | Cleavage strength |
| ISO 9653: 1991<br>(= BS EN 29653)          | Adhesives - Test method for shear impact strength of adhesive bonds                                                                       | Impact strength   |
| ISO 4578: 1990<br>(= BS EN 1464)           | Adhesives - Determination of peel resistance of high strength adhesive bonds - floating roller method                                     | Peel strength     |
| ISO 8510-1: 1990<br>(= BS EN 28510 Part 1) | Adhesives - Peel test for a flexible-bonded-to-rigid test specimen assembly - 90° peel                                                    | Peel strength     |
| ISO 8510-2: 1990<br>(= BS EN 28510 Part 2) | Adhesives - Peel test for a flexible-bonded-to-rigid test specimen assembly - 180° peel                                                   | Peel strength     |
| ISO 11339: 1993<br>(= BS 5350: Pt C12)     | Adhesives - 180° peel test for flexible-to-flexible bonded assemblies (T-peel test)                                                       | T-peel strength   |
| ISO 4587: 1995<br>(= BS EN 1465)           | Adhesives - Determination of tensile lap-strength of rigid-to-rigid assemblies [single lap]                                               | Shear strength    |
| ISO 6237: 1987<br>(= BS EN 205)            | Wood to wood adhesive bonds - determination of shear strength by tensile loading                                                          | Shear strength    |
| ISO 6238: 1987<br>(= NFB 51-032)           | Wood to wood adhesive bonds - Determination of shear strength by compressive loading                                                      | Shear strength    |
| ISO 10123: 1990                            | Adhesives - Determination of shear strength of anaerobic adhesives using pin-and-collar specimens                                         | Shear strength    |
| ISO 13445: 1995                            | Adhesives - Determination of shear strength of adhesive bonds between rigid substrates by the block-shear method                          | Shear strength    |
| ISO 11003/1: 1993                          | Adhesives - Determination of shear behaviour of structural bonds - Torsion test method using butt bonded hollow cylinders                 | Shear strength    |
| ISO 11003/2: 1993                          | Adhesives - Determination of shear behaviour of structural bonds - Thick-adherend tensile-test method                                     | Shear strength    |
| ISO 6922: 1987<br>(= BS EN 26922)          | Adhesives - Determination of bond strength in direct tension                                                                              | Tensile strength  |
| ISO / DIS 14679 (1996)                     | Adhesives - Measurement of adhesion characteristics by a three-point bending method                                                       |                   |

**Table 2 BS Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>                         | <b>TITLE</b>                                                                                                               | <b>PURPOSE</b>    |
|---------------------------------------|----------------------------------------------------------------------------------------------------------------------------|-------------------|
| BS 5350: Part C1: 1986                | Methods of test for adhesives - Determination of cleavage strength of adhesive bonds                                       | Cleavage strength |
| BS EN 29653 (= ISO 9653)              | Adhesives - Test method for shear impact strength of adhesive bonds                                                        | Impact strength   |
| BS 5350: Part C12: 1994 (= ISO 11339) | Methods of test for adhesives - 180° peel test for flexible-to-flexible bonded assemblies (T-peel test)                    | T-peel strength   |
| BS 5350: Part C13: 1990               | Methods of test for adhesives - Climbing drum peel test                                                                    | Peel strength     |
| BS 5350: Part C14: 1979               | Methods of test for adhesives - Adhesively bonded mechanical joints - 90° peel test for a rigid-to-rigid assembly          | Peel strength     |
| BS EN 1464: 1995 (= ISO 4578)         | Adhesives - Determination of peel resistance of high strength adhesive bonds - floating roller method                      | Peel strength     |
| BS EN 2243: Part 2: 1991              | Structural adhesives - test methods - peel metal-metal                                                                     | Peel strength     |
| BS EN 2243: Part 3: 1992              | Structural adhesives - Test methods -Peeling test metal-honeycomb core                                                     | Peel strength     |
| BS EN 28510: Pt 1: 1993 (=ISO 8510-1) | Methods of test for adhesives - 90° peel test for a flexible-to-rigid assembly                                             | Peel strength     |
| BS EN 28510: Pt 2: 1993 (=ISO 8510-2) | Methods of test for adhesives - 180° peel test for a flexible-to-rigid assembly                                            | Peel strength     |
| BS 5350: Part C15: 1990               | Methods of test for adhesives - Determination of bond strength in compressive shear                                        | Shear strength    |
| BS EN 205: 1991 (= ISO 6237)          | Test methods for wood adhesives for non-structural applications - Determination of tensile shear strength of lap joints    | Shear strength    |
| BS EN 302-1: 1992                     | Adhesives for load-bearing timber structures - test methods - Determination of bond strength in longitudinal tensile shear | Shear strength    |
| BS EN 1465: 1995 (= ISO 4587)         | Adhesives - Determination of tensile lap-shear strength of rigid-to-rigid bonded assemblies                                | Shear strength    |
| BS 5350: Part C5: 1990                | Methods of test for adhesives - Determination of bond strength in longitudinal shear                                       | Shear strength    |
| BS 5350: Part C6: 1990                | Methods of test for adhesives - Determination of bond strength in direct tension in sandwich panels                        | Tensile strength  |
| BS EN 2243: Part 4: 1991              | Structural adhesives - Test methods - Metal-honeycomb core flatwise tensile test                                           | Tensile strength  |
| BS EN 26922: 1993 (= ISO 6922)        | Methods of test for adhesives - Determination of bond strength in direct tension                                           | Tensile strength  |

**Table 3 ASTM Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>   | <b>TITLE</b>                                                                                                          | <b>PURPOSE</b>                   |
|-----------------|-----------------------------------------------------------------------------------------------------------------------|----------------------------------|
| ASTM D 1062-92  | Cleavage strength of metal-to-metal adhesive bonds                                                                    | Cleavage strength                |
| ASTM D 3433-93  | Fracture strength in cleavage of adhesive in bonded joints [fracture toughness $G_{IC}$ ]                             | Cleavage strength                |
| ASTM D 3807-79  | Strength properties of adhesives in cleavage peel by tension loading (engineering plastics-to-engineering plastics)   | Cleavage strength                |
| ASTM D 5041-93b | Fracture strength in cleavage of adhesives in bonded joints                                                           | Cleavage strength                |
| ASTM D 5573-94  | Classifying failure modes in fiber-reinforced-plastic (FRP) joints                                                    | Failure analysis                 |
| ASTM D 1184-93  | Flexural strength of adhesive bonded laminated assemblies                                                             | Flexural strength                |
| ASTM D 950-94   | Impact strength of adhesive bonds                                                                                     | Impact strength                  |
| ASTM D 903-93   | Peel or stripping strength of adhesive bonds                                                                          | Peel strength                    |
| ASTM D 1781-93  | Climbing drum peel test for adhesives                                                                                 | Peel strength                    |
| ASTM D 1876-93  | Peel resistance of adhesives (T-peel test)                                                                            | T-peel strength                  |
| ASTM D 3167-93  | Floating roller peel resistance of adhesives                                                                          | Peel strength                    |
| ASTM D 905-94   | Strength properties of adhesive bonds in shear by compression loading [plywood laminate constructions]                | Shear strength                   |
| ASTM D 906-94a  | Strength properties of adhesives in plywood type construction in shear by tension loading                             | Shear strength                   |
| ASTM D 1002-94  | Apparent shear strength of single-lap-joint adhesively bonded metal specimens by tension loading (metal-to-metal)     | Shear strength                   |
| ASTM D 2339-94a | Strength properties of adhesives in two-ply wood construction in shear by tension loading                             | Shear strength                   |
| ASTM D 3163-92  | Determining the strength of adhesively bonded rigid plastic lap-shear joints in shear by tension loading [single lap] | Shear strength                   |
| ASTM D 3164-92a | Strength properties of adhesively bonded plastic lap-shear sandwich joints in shear by tension loading [single lap]   | Shear strength                   |
| ASTM D 3165-91  | Strength properties of adhesives in shear by tension loading of single-lap-joint laminated assemblies                 | Shear strength                   |
| ASTM D 3528-92  | Strength properties of double lap shear adhesive joints by tension loading                                            | Shear strength                   |
| ASTM D 3983-93  | Measuring strength and shear modulus of nonrigid adhesives by the thick adherend tensile lap specimen                 | Shear strength and shear modulus |
| ASTM D 4027-93  | Measuring shear properties of structural adhesives by the modified-rail test                                          | Shear strength and shear modulus |
| ASTM D 4501-91  | Shear strength of adhesive bonds between rigid substrates by the block-shear method                                   | Shear strength                   |

**Table 3 ASTM Standard Test Methods for Adhesively Bonded Joints (Cont.)**

| <b>NUMBER</b>  | <b>TITLE</b>                                                                                                                   | <b>PURPOSE</b>                             |
|----------------|--------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------|
| ASTM D 4896-89 | Use of adhesive-bonded single lap-joint specimen test results                                                                  | Shear strength                             |
| ASTM D 5656-95 | Thick adherend metal lap-shear joints for determination of the stress-strain behavior of adhesives in shear by tension loading | Shear strength and shear modulus           |
| ASTM D 897-85  | Tensile properties of adhesive bonds                                                                                           | Tensile strength and tensile modulus       |
| ASTM D 2095-92 | Tensile strength of adhesives by means of bar and rod specimens                                                                | Tensile strength                           |
| ASTM D 3658-90 | Determining the torque strength of ultraviolet (UV) light-cured glass/metal adhesive joints                                    | Torsional shear strength                   |
| ASTM E 229-92  | Shear strength and shear modulus of structural adhesives                                                                       | Torsional shear strength and shear modulus |

**Table 4 DIN Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>               | <b>TITLE</b>                                                                                                                                                                     | <b>PURPOSE</b>        |
|-----------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| DIN 65 448<br>(1988)        | Structural adhesives; wedge test                                                                                                                                                 | Cleavage strength     |
| DIN 53 284<br>(1979)        | Testing of adhesives for metals and adhesively bonded metal joints; creep test on single lap joints                                                                              | Creep                 |
| DIN 54 456<br>(1989)        | Testing of structural adhesives: test of resistance to climatic conditions                                                                                                       | Environmental testing |
| DIN 53 286<br>(1979)        | Testing of adhesives for metals and adhesively bonded metal joints; conditions for testing at different temperatures                                                             | Environmental testing |
| DIN 53 289<br>(1974)        | Testing of adhesives for metals; floating roller peel test                                                                                                                       | Peel strength         |
| DIN 53 295<br>(1974)        | Testing of adhesives for metallic honeycomb sandwiches; climbing drum peel test                                                                                                  | Peel strength         |
| DIN 50 021<br>(1988)        | Salt spray testing                                                                                                                                                               | Salt spray            |
| DIN 53 283<br>(1974)        | Testing of adhesives for metals; determination of the shear strength of single lap joints by tensile test                                                                        | Shear strength        |
| DIN 53 769 Part 1<br>(1988) | Testing of glass fibre reinforced plastics pipelines; determination of the adhesive shear strength of Type B pipeline components                                                 | Shear strength        |
| DIN 54 451<br>(1978)        | Testing of adhesives for metals and adhesively bonded metal joints; tensile shear test for the determination of the shear stress-strain diagram of an adhesive in a bonded joint | Shear strength        |
| DIN 54 452<br>(1981)        | Testing of adhesives for metals and adhesively bonded metal joints; compression shear test                                                                                       | Shear strength        |
| DIN 53 292<br>(1974)        | Testing of adhesives for metallic honeycomb sandwiches; flatwise tensile test                                                                                                    | Tensile strength      |
| DIN 53 766 Part 1<br>(1991) | Testing of glass fibre reinforced plastics apparatus and containers; determination of adhesive strength                                                                          |                       |
| DIN 54 455<br>(1984)        | Testing of adhesives for metals and adhesively bonded metal joints; torsional shear test                                                                                         | Torsional shear       |

**Table 5 JIS Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>                               | <b>TITLE</b>                                                                              | <b>PURPOSE</b>        |
|---------------------------------------------|-------------------------------------------------------------------------------------------|-----------------------|
| JIS K 6858 (1974)                           | Testing methods for resistance of adhesive bonds to chemical substances                   | Chemical resistance   |
| JIS K 6853 (1994)                           | Testing methods for cleavage strength of adhesives                                        | Cleavage strength     |
| JIS K 6859 (1994)                           | Testing methods for creep rupture of adhesive bonds                                       | Creep                 |
| JIS K 6857 (1973)                           | Testing methods for resistance of adhesive bonds to water or moisture                     | Environmental testing |
| JIS K 6856 (1994)                           | Testing methods for flexural strength of adhesives                                        | Flexural strength     |
| JIS K 6855 (1994)                           | Testing methods for impact strength of adhesives                                          | Impact strength       |
| JIS K 6854 (1994)                           | Testing methods for peel strength of adhesives                                            | Peel strength         |
| JIS C 0023 (1989)<br>(=BS 2011: Pt 2<br>Ka) | Basic environmental testing procedures Part 2<br>- Test Ka: salt mist                     | Salt spray            |
| JIS C 0024 (1989)<br>(=BS 2011: Pt 2<br>Kb) | Basic environmental testing procedures Part 2<br>- Test Kb: salt mist, cyclic             | Salt spray            |
| JIS K 6850 (1976)                           | Testing methods for strength properties of adhesives in shear                             | Shear strength        |
| JIS K 6851 (1976)                           | Testing methods for strength properties of adhesives for wood in shear by tension loading | Shear strength        |
| JIS K 6852 (1976)                           | Testing methods for strength properties of adhesives in shear by compression loading      | Shear strength        |
| JIS K 6849 (1976)                           | Testing methods for tensile strength of adhesives                                         | Tensile strength      |

**Table 6 Other Standard Test Methods for Adhesively Bonded Joints**

| <b>NUMBER</b>        | <b>TITLE</b>                                                                                                                   | <b>PURPOSE</b>    |
|----------------------|--------------------------------------------------------------------------------------------------------------------------------|-------------------|
| NFT 76-114<br>(1983) | Adhesives - structural adhesives - cleavage test using a wedge                                                                 | Cleavage strength |
| SAE J 1863<br>(1993) | Coach joint fracture test                                                                                                      |                   |
| SAE J 1553<br>(1995) | Cross peel test for automotive type adhesives for fiber reinforced plastic bonding                                             | Peel strength     |
| SAE J 1907<br>(1988) | Peel adhesion test for glass to elastomeric material for automotive glass encapsulation                                        | Peel strength     |
| AS 1937.9 (1991)     | Methods of test for sealers and adhesives for Automotive purposes - Determination of shear strength of metal-to-metal adhesive | Shear strength    |
| CRAG 102             | Method of test for lap shear strength of fibre reinforced plastics [single and double lap]                                     | Shear strength    |
| NFB 51-032<br>(1981) | Laminated bonded wood - Compressive shear test                                                                                 | Shear strength    |
| NFT 76-121<br>(1984) | Structural adhesives - Determination of the shear strength of an axle ring type assembly                                       | Shear strength    |
| prEN 2243-6          | Structural adhesives - Test methods - Determination of shear stress and shear displacement                                     | Shear stress      |
| SAE J 1523<br>(1993) | Metal to metal overlap shear strength test for automotive type adhesives                                                       | Shear strength    |
| SAE J 1525<br>(1985) | Lap shear test for automotive type adhesives for fiber reinforced plastic bonding                                              | Shear strength    |
| SAE J 1529<br>(1986) | Overlap shear test for automotive type sealant for stationary glass bonding                                                    | Shear strength    |
| SAE J 1836<br>(1988) | Overlap shear test for sealant adhesive bonding of automotive glass encapsulating material to body opening                     | Shear strength    |

**Table 7 ISO Standard Test Methods for Adhesively Bonded Joints - Tension**

| NUMBER         | TITLE                                            | ALTERNATIVES                   |
|----------------|--------------------------------------------------|--------------------------------|
| ISO 6922: 1987 | Determination of tensile strength of butt joints | BS EN 26922,<br>ASTM D 2095-92 |

**Table 8 BS Standard Test Methods for Adhesively Bonded Joints - Tension**

| NUMBER                   | TITLE                                                                            | ALTERNATIVES                |
|--------------------------|----------------------------------------------------------------------------------|-----------------------------|
| BS EN 2243: Part 4: 1991 | Structural adhesives - Test methods - Metal-honeycomb core flatwise tensile test |                             |
| BS EN 26922: 1993        | Determination of tensile strength of butt joints                                 | ISO 6922,<br>ASTM D 2095-92 |

**Table 9 ASTM Standard Test Methods for Adhesively Bonded Joints - Tension**

| NUMBER         | TITLE                                                           | ALTERNATIVES          |
|----------------|-----------------------------------------------------------------|-----------------------|
| ASTM D 897-85  | Tensile properties of adhesive bonds                            |                       |
| ASTM D 2095-92 | Tensile strength of adhesives by means of bar and rod specimens | ISO 6922, BS EN 26922 |

**Table 10 ISO Standard Test Methods for Adhesively Bonded Joints - Shear**

| NUMBER              | TITLE                                                                                                         | ALTERNATIVES                               |
|---------------------|---------------------------------------------------------------------------------------------------------------|--------------------------------------------|
| ISO 4587: 1995      | Determination of tensile lap-strength of high strength adhesive bonds [single lap]                            | BS EN 1465, ASTM D 3163-92, ASTM D 1002-94 |
| ISO 6237: 1987      | Wood to wood adhesive bonds - Determination of shear strength by tensile loading                              | BS EN 205, DIN 53 254,<br>ASTM D 906-94a   |
| ISO 6238: 1987      | Wood to wood adhesive bonds - Determination of shear strength by compressive loading                          | ASTM D 905-94,<br>NFB 51-032               |
| ISO 10123: 1990     | Determination of shear strength of anaerobic adhesives using pin-and-collar specimens                         |                                            |
| ISO 11003 / 1: 1993 | Determination of shear behaviour of structural bonds - Torsion test method using butt bonded hollow cylinders |                                            |
| ISO 11003 / 2: 1993 | Determination of shear behaviour of structural bonds - Thick adherend tensile-test method                     |                                            |
| ISO 13445: 1995     | Determination of shear strength of adhesive bonds between rigid substrates by the block-shear method          | ASTM D 4501-91                             |

**Table 11 BS Standard Test Methods for Adhesively Bonded Joints - Shear**

| <b>NUMBER</b>              | <b>TITLE</b>                                                                                                               | <b>ALTERNATIVES</b>                      |
|----------------------------|----------------------------------------------------------------------------------------------------------------------------|------------------------------------------|
| BS 5350: Part C5:<br>1990  | Determination of bond strength in longitudinal shear                                                                       | ASTM D 3528-92                           |
| BS 5350: Part C15:<br>1990 | Determination of bond strength in compressive shear [double lap joint]                                                     |                                          |
| BS EN 205: 1991            | Test methods for wood adhesives for non-structural applications - Determination of tensile shear strength of lap joints    | ISO 6237                                 |
| BS EN 302-1:<br>1992       | Adhesives for load-bearing timber structures - Test methods - Determination of bond strength in longitudinal tensile shear | ASTM D 2339-94a                          |
| BS EN 1465:<br>1995        | Determination of tensile lap-shear strength of rigid-to-rigid bonded assemblies                                            | ISO 4587, ASTM D 3163-92, ASTM D 1002-94 |

**Table 12 ASTM Standard Test Methods for Adhesively Bonded Joints - Shear**

| <b>NUMBER</b>   | <b>TITLE</b>                                                                                                                    | <b>ALTERNATIVES</b>              |
|-----------------|---------------------------------------------------------------------------------------------------------------------------------|----------------------------------|
| ASTM D 905-94   | Strength properties of adhesive bonds in shear by compression loading [plywood laminate constructions]                          | ISO 6238                         |
| ASTM D 906-94a  | Strength properties of adhesives in plywood type construction in shear by tension loading                                       | ISO 6237                         |
| ASTM D 1002-94  | Apparent shear strength of single-lap-joint adhesively bonded metal specimens by tension loading (metal-to-metal)               | ISO 4587, BS EN 1465, SAE J 1525 |
| ASTM D 2339-94a | Strength properties of adhesives in two-ply-wood construction in shear by tension loading                                       | BS EN 302-1                      |
| ASTM D 3163-92  | Determining strength of adhesively bonded rigid plastic lap-shear joints in shear by tension loading [single lap]               |                                  |
| ASTM D 3164-92a | Strength properties of adhesively bonded plastic lap-shear sandwich joints in shear by tension loading [single lap]             |                                  |
| ASTM D 3165-91  | Strength properties of adhesives in shear by tension loading of single-lap-joint laminated assemblies                           |                                  |
| ASTM D 3528-92  | Strength properties of double lap shear adhesive joints by tension loading                                                      | BS 5350: Part C5                 |
| ASTM D 3983-93  | Measuring strength and shear modulus of nonrigid adhesives by the thick adherend tensile-lap specimen                           |                                  |
| ASTM D 4027-93  | Measuring shear properties of structural adhesives by the modified-rail test                                                    |                                  |
| ASTM D 4501-91  | Shear strength of adhesive bonds between rigid substrates by the block-shear method                                             | ISO 13445                        |
| ASTM D 4896-89  | Use of adhesive-bonded single lap-joint specimen test results                                                                   |                                  |
| ASTM D 5656-95  | Thick-adherend metal lap-shear joints for determination of the stress-strain behaviour of adhesives in shear by tension loading |                                  |
| ASTM D 3658-90  | Determining the torque strength of ultraviolet (UV) light-cured glass/metal joints                                              |                                  |
| ASTM E 229-92   | Shear strength and shear modulus of structural adhesives                                                                        |                                  |

**APPENDIX II - ACCELERATED TEST METHOD STANDARDS****Table 1 ISO Standard Test Methods for Accelerated Ageing  
(Ageing, Environmental Resistance, Fatigue and Creep)**

| <b>NUMBER</b>   | <b>TITLE</b>                                                                                                                                                 | <b>ALTERNATIVES</b>                       |
|-----------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------|
| ISO 62          | Plastics - Determination of water absorption                                                                                                                 | BS 2782/4/430A-D,<br>DIN 4618, DIN 53 495 |
| ISO 75 (Part 1) | Plastics - Determination of temperature of deflection under load - General test method                                                                       | BS 2782/1/121A                            |
| ISO 75 (Part 3) | Plastics - Determination of temperature of deflection under load - High-strength thermosetting laminates and long-fibre-reinforced plastics                  | BS 2782/1/121D                            |
| ISO 175         | Plastics - determination of the effects of liquid chemicals including water                                                                                  | BS 2782/8/830A, BS 4618,<br>DIN 53 476    |
| ISO 291         | Plastics - Standard atmospheres for conditioning and testing                                                                                                 | BS 2782/10, BS 1004,<br>DIN 50 014        |
| ISO 483         | Plastics - Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value                             | BS 3718                                   |
| ISO 846         | Determination of behaviour under the action of fungi and bacteria - Evaluation by visual examination or measurement of change in mass or physical properties |                                           |
| ISO 877         | Plastics - Determination of resistance to change upon exposure under glass to daylight                                                                       |                                           |
| prEN 2378       | Fibre reinforced plastics - Determination of water absorption by immersion in demineralised water                                                            |                                           |
| prEN 2489       | Fibre reinforced plastics - Determination of the action of liquid chemicals                                                                                  |                                           |
| prEN 2823       | Fibre reinforced plastics - Determination of the effect of exposure to humid atmosphere on physical and mechanical properties                                |                                           |
| ISO 4582        | Plastics - Determination of changes in colour and variations in properties and exposure to daylight under glass, natural weathering or artificial light.     |                                           |
| ISO 4599        | Plastics - Determination of resistance to environmental stress cracking (ESC) - Bent strip method                                                            |                                           |
| ISO 4600        | Determination of environmental stress cracking - Ball and pin                                                                                                |                                           |
| ISO 4607        | Plastics - Methods of exposure to natural weathering                                                                                                         | BS 2782/5/550A                            |
| ISO 4611        | Plastics - Determination of the effects of exposure to damp heat, water spray and salt mist (on optical and colour properties)                               | BS 2782/5/551A                            |
| ISO 4892        | Plastics - Method of exposure to laboratory light sources                                                                                                    | BS 2782/5/540B,<br>DIN 53 387             |
| ISO 6252        | Plastics - Determination of environmental stress cracking (ESC) - Constant tensile stress method                                                             | DIN 53 449                                |

**Table 1 ISO Standard Test Methods for Accelerated Ageing (Cont.)  
(Ageing, Environmental Resistance, Fatigue and Creep)**

| <b>NUMBER</b>   | <b>TITLE</b>                                                                                           | <b>ALTERNATIVES</b>          |
|-----------------|--------------------------------------------------------------------------------------------------------|------------------------------|
| ISO 9142: 1990  | Adhesives - Guide to the selection of standard laboratory ageing conditions for testing bonded joints  | BS EN ISO 29142              |
| ISO 9664        | Test methods for fatigue properties of structural adhesives in tensile shear                           | BS EN ISO 9664<br>DIN 53 285 |
| ISO 10354: 1992 | Adhesives - Characterisation of durability of structural adhesive assemblies - wedge rupture test      | NFT 76-144                   |
| ISO 10363: 1992 | Hot melt adhesives - Determination of thermal stability                                                | BS EN ISO 10363              |
| ISO 9142        | Adhesives - Guide to the selection of standard laboratory ageing conditions for testing bonded joints  | BS EN ISO 29142              |
| ISO / DIS 14615 | Adhesives - Durability of structural adhesive joints - Exposure to humidity and temperature under load | BS ISO / DIS 14615           |
| ISO TR 9673     | Solar radiation and its measurement for determining outdoor weather exposure levels                    |                              |

**Table 2 BS Standard Test Methods for Accelerated Ageing  
(Ageing, Environmental Resistance, Fatigue and Creep)**

| <b>NUMBER</b>                         | <b>TITLE</b>                                                                                                                                            | <b>PURPOSE</b>                      |
|---------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------|
| BS 2011: Part 2.1Ca: 1977             | Environmental testing - Test Ca. Damp heat, steady state                                                                                                | IEC 68-2-3                          |
| BS 2011: Part 2.1Cb: 1990             | Environmental testing - Test Cb. Damp heat, steady state (primarily for equipment)                                                                      | IEC 68-2-56                         |
| BS 2011: Part 2.1Db: 1981             | Environmental testing - Test Db. Damp heat, cyclic                                                                                                      | IEC 68-2-30                         |
| BS 2011: Part 2Ka                     | Environmental testing - Test Ka. Salt mist                                                                                                              | IEC 68-2-11, JIS C 0023, NFC 20-711 |
| BS 2011: Part 2Kb<br>BS EN 60068-2-52 | Environmental testing - Test Ka. Salt mist, cyclic                                                                                                      | IEC 68-2-52, JIS C 0024             |
| BS 2011: Part 2.1Rc                   | Environmental testing - Test Rc. Water immersion                                                                                                        | IEC 68-2-18: 1989                   |
| BS 2782: Part 5: Method 551A          | Plastics - Determination of the effects of exposure to damp heat, water spray and salt mist                                                             | ISO 4611: 1987                      |
| BS 5350: Part BXX                     | Methods of test for adhesives - Freeze-thaw stability                                                                                                   | prEN 1239                           |
| BS 5350: Part C7: 1990                | Determination of creep and resistance to sustained application of force                                                                                 |                                     |
| BS 5350: Part H3                      | Determination of heat resistance of hot-melt adhesives                                                                                                  |                                     |
| BS 5480: Part 2                       | Specification for glass reinforced plastics (GRP) pipes and fittings for use for water supply and sewerage. Part 2: Design and performance requirements |                                     |
| BS EN 2243: Part 5                    | Structural adhesives - Test methods - Ageing tests                                                                                                      |                                     |
| BS EN 60068-2-1                       | Environmental testing - Tests A. Cold                                                                                                                   |                                     |
| BS EN 60068-2-2                       | Environmental testing - Tests B. Dry heat                                                                                                               | IEC 68-2-2: 1974                    |
| BS EN ISO 9142: 1990                  | Adhesives - Guide to the selection of standard laboratory ageing conditions for testing bonded joints                                                   | ISO 29142                           |
| BS EN ISO 9664: 1995                  | Test methods for fatigue properties of structural adhesives in tensile shear                                                                            | ISO 9664, DIN 53 285                |
| BS EN ISO 10363: 1992                 | Hot melt adhesives - Determination of thermal stability                                                                                                 | ISO 10363                           |
| BS ISO / DIS 14615 (1996)             | Durability of structural adhesive joints - Exposure to humidity and temperature under load                                                              | ISO / DIS 14615                     |

**Table 3 ASTM Standard Test Methods for Accelerated Ageing  
(Ageing, Environmental Resistance, Fatigue and Creep)**

| <b>NUMBER</b>  | <b>TITLE</b>                                                                                           | <b>PURPOSE</b>           |
|----------------|--------------------------------------------------------------------------------------------------------|--------------------------|
| ASTM D 1183-92 | Resistance of adhesives to cyclic laboratory ageing conditions                                         | Ageing                   |
| ASTM D 3434-90 | Multiple-cycle accelerated aging test (automatic boil test) for exterior wet use wood adhesives        | Ageing (accelerated)     |
| ASTM D 3632-77 | Accelerated aging of adhesive joints by the oxygen- pressure method                                    | Ageing (accelerated)     |
| ASTM D 896-92  | Resistance of adhesive bonds to chemical reagents                                                      | Chemical reagents        |
| ASTM D 3310-74 | Determining corrosivity to adhesive materials                                                          | Corrosivity              |
| ASTM D 1151-90 | Effect of moisture and temperature on adhesive bonds                                                   | Environmental resistance |
| ASTM D 1828-91 | Atmospheric exposure of adhesive-bonded joints and structures                                          | Environmental resistance |
| ASTM D 2918-93 | Durability assessment of adhesive joints stressed in peel                                              | Environmental resistance |
| ASTM D 2919-90 | Determining durability of adhesive joints stressed in shear by tension loading (single lap)            | Environmental resistance |
| ASTM D 3762-79 | Adhesive-bonded surface durability of aluminium (wedge test)                                           | Environmental resistance |
| ASTM D 4502-92 | Heat and moisture resistance of wood-adhesive joints                                                   | Environmental resistance |
| ASTM D 2295-92 | Strength properties of adhesives in shear by tension loading at elevated temperatures (metal-to-metal) | High temperature         |
| ASTM D 4498-85 | Heat-fail temperature in shear of hot melt adhesives                                                   | High temperature         |
| ASTM B 117-73  | Salt spray (fog) testing                                                                               | Salt spray               |
| ASTM G 85-84   | Modified salt spray (fog) testing                                                                      | Salt spray               |
| ASTM D 3166-93 | Fatigue properties of adhesives in shear by tension loading (metal-to-metal) [single lap]              | Fatigue                  |
| ASTM D 1780-94 | Conducting creep tests of metal-to-metal adhesives (tensile shear loading)                             | Creep                    |
| ASTM D 2293-92 | Creep properties of adhesives in shear by compression loading (metal-to-metal)                         | Creep                    |
| ASTM D 2294-69 | Creep properties of adhesives in shear by tension loading (metal-to-metal)                             | Creep                    |
| ASTM D 4680-92 | Creep and time to failure of adhesives in static shear by compression loading (wood-to-wood)           | Creep                    |

Table 4 ISO Standards Relating to Environmental Conditioning

| Standard                                              | Subject/Comments                                                                                                                       | Materials                                                                                                                                                                          | Conditions                                                                                                                                                                                             | Specimen                                                                                                                                                                                                                       |
|-------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| ISO 4611: 1988<br>(= BS 2782: Part 5:<br>Method 551A) | Specifies exposure conditions of plastics to salt mist, and determines change of mass dimensions, appearance and physical properties   | Plastics                                                                                                                                                                           | Salt solution 50±5 g/l<br>Test duration of 24 hrs, 48 hrs, 96 hrs, 144 hrs, 168 hrs, 1 wk, 2 wks, 4 wks, 8 wks, 16 wks, 26 wks, 52 wks or 78 wks<br>Pre-condition for min 86 hrs at ambient conditions | Min 3 specimens                                                                                                                                                                                                                |
| ISO 9664<br>(= BS EN ISO 9664)                        | Estimates fatigue strength of adhesives in shear by tension loading                                                                    | Steel (single lap) or aluminium (double lap)                                                                                                                                       | General test frequency 30 Hz, max 60 Hz<br>Test at 3 different stress amplitudes for failure to occur between 10 <sup>4</sup> and 10 <sup>6</sup> cycles                                               | Steel<br>Length of each adherend = 100 mm<br>Width = 25 mm<br>Thickness = 1.6 mm<br>Overlap = 12.5 mm<br>Aluminium<br>Length of each adherend = min 91 mm<br>Width = 25 mm<br>Thickness = 3.2 mm<br>Overlap = 12.5 mm          |
| ISO 10354: 1992                                       | Simulates forces and effects on a bonded joint                                                                                         | Aluminium and titanium; may be used for other metals and plastics if rigid<br><br>Wedge (stainless steel)<br>Length = 30 mm<br>Width = 25 mm<br>Thickness = 3 mm<br>60° angle nose | Drive wedge fully home at 30±5 mm/min<br>Measure initial crack length, age according to ISO 9142, then measure final crack length                                                                      | Each adherend<br>Length = 150 mm<br>Width = 25 mm<br>Thickness = 3 mm<br>Bonded length = 115 mm, 60° notch at unbonded end between adherends                                                                                   |
| ISO/DIS 14615 (1996)<br>(= BS ISO/DIS 14615)          | Describes equipment and procedure used to evaluate the durability of adhesive joints subjected to simultaneous heat, humidity and load | Metals, composites, plastics<br>Lap joints and T-peel joints                                                                                                                       | Temperature cycled between 42°C and 48°C (30 + 30 mins)<br>Humidity: 95 - 100% RH<br>Load between 1MPa and 7MPa                                                                                        | Lap joint<br>Length of each adherend = 54 mm<br>Width = 20 mm<br>Thickness = 0.6-3 mm<br>Overlap = 10 mm<br>T-peel joint<br>Length of each adherend = 58 mm<br>Width = 25 mm<br>Thickness = 0.6 - 3 mm<br>Bonded length = 25mm |

Table 5 BS Standards Relating to Environmental Conditioning

| Standard                                                           | Subject/Comments                                                                                                                                                                  | Materials                 | Conditions                                                                                                                                                                                                                                              | Specimen      |
|--------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|
| BS 2011: Part 2.1Ca:<br>1977<br>(= IEC 68-2-3)                     | Specifies test conditions under steady state damp heat environment                                                                                                                | Not specified             | Test at 40±2°C, 93±2%/-3% RH for 4, 10, 21 or 56 days                                                                                                                                                                                                   | Not specified |
| BS 2011: Part 2.1Cb:<br>1990<br>(= IEC 68-2-56)                    | Specifies test conditions under steady state damp heat environment (primarily for equipment)                                                                                      | Electrotechnical products | Increase temperature by 1°C/min to the test temperature then increase humidity to required level in 2 hrs max, to one of the following test conditions:<br>A - 30±2°C, 93±3% RH<br>B - 30±2°C, 85±3% RH<br>C - 40±2°C, 93±3% RH<br>D - 40±2°C, 85±3% RH | Not specified |
| BS 2011: Part 2.1Db:<br>1981<br>(= IEC 68-2-30)                    | Specifies test conditions under cyclic damp heat environment                                                                                                                      | Not specified             | Cycle between 25±3°C and 40°C (12 hr + 12 hr cycle) for 2, 6, 12, 21 or 56 cycles, or between 25±3°C and 55°C for 1, 2 or 6 cycles                                                                                                                      | Not specified |
| BS 2011: Part 2Ka<br>(= IEC 68-2-11,<br>JIS C 0023,<br>NFC 20-711) | Comparison between specimens of the resistance to deterioration from salt mist<br>NOT suitable as general salt corrosion test or for evaluation of individual specimens - QA only | Not specified             | 5% by weight salt solution, containing <0.1% sodium iodide, <0.3% impurities<br>Test conditions: 35±2°C, min 85% RH<br>Duration: 16 hrs, 24 hrs, 2 days, 4 days, 1 wk<br>2 wks or 4 wks                                                                 | Not specified |

Table 5 BS Standards Relating to Environmental Conditioning (Cont.)

| Standard                                                                | Subject/Comments                                                                                                                            | Materials                                                                | Conditions                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | Specimen                         |
|-------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------|
| BS 2011: Part 2Kb<br>BS EN 60068-2-52<br>(= IEC 68-2-52,<br>JIS C 0024) | Method of evaluating<br>resistance of components to salt<br>atmosphere                                                                      | Not specified                                                            | 5% by weight salt solution, containing <0.1%<br>sodium iodide, <0.3% impurities<br>Test conditions: 35±2°C, min 85% RH<br>Spray for 2 hrs between 15°C and 35°C for<br>one of the following tests:<br>A - 4 spray periods (2 hrs each), humidity<br>storage period of 7 days after each<br>B - 3 spray periods (2 hrs each), humidity<br>storage period of 20-22 hrs after each<br>C - 1 test cycle (4 spray periods, humidity<br>storage period of 20-22 hrs after each),<br>followed by storage period of 3 days at<br>23±2°C, 50±5%<br>D - as C, but with 2 test cycles<br>E - as C, but with 4 test cycles<br>F - as C, but with 8 test cycles | Not specified                    |
| BS 2011: Part 2Rc: 1990<br>(= IEC 68-2-18)                              | Specifies test conditions under<br>water immersion:<br>A - Water tank<br>B - Pressurised water chamber                                      | Watertight electrotechnical<br>products                                  | A - fresh tap water or sea water at 35°C max<br>Water head: 0.15m, 0.4m, 1m, 2m or 5m<br>Duration: 0.5, 2 or 24 hrs<br>B - 35°C max<br>Pressure: 20, 50, 100, 200, 500, 1000, 2000,<br>5000 or 10000 kPa<br>Duration: 2, 24 or 168 hrs                                                                                                                                                                                                                                                                                                                                                                                                             | Not specified                    |
| BS 2782: Pt 5:<br>Method 551A                                           | See ISO 4611                                                                                                                                |                                                                          |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                  |
| BS 5350: Part BXX<br>(= prEN 1239)                                      | Describes method for<br>evaluating the freeze-thaw<br>stability of adhesives                                                                | Adhesives                                                                | Measure viscosity, freeze for 16 hrs in sealed<br>container at -5°C, -10°C, -15°C or -20°C then<br>thaw for 8hrs. Repeat cycle twice more, then<br>measure viscosity again.                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | N/A                              |
| BS 5350: Part C7: 1990                                                  | Describes application of test<br>methods in which force is<br>applied to adhesive bond to<br>determine displacement under<br>constant force | As appropriate for tests in BS<br>5350: Parts C1, C3, C5, C14 and<br>C15 | As appropriate to test method                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | As appropriate to test<br>method |

Table 5 BS Standards Relating to Environmental Conditioning (Cont.)

| Standard               | Subject/Comments                                                                                                                                                                                                                                                                   | Materials                                                              | Conditions                                                                                                                                                                                                                                                                                                                                                                                          | Specimen                                                                                                                                                                                    |
|------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| BS 5350: Part H3: 1984 | Determines heat resistance of hot-melt adhesives                                                                                                                                                                                                                                   | Carton board or other suitable adherends<br>Adhesive film 25x25x0.2 mm | Suspend in oven for 15 mins at 30°C with 100g weight at lower end, then raise temperature by 5°C and maintain for 15 mins. Repeat until joint fails - heat resistance = $(T_{fail} - 5)^\circ\text{C}$                                                                                                                                                                                              | Lap joint:<br>Length of each adherend = 50 mm<br>Width = 25 mm<br>Overlap = 25 mm                                                                                                           |
| BS EN 2243: Part 5     | Determines resistance of adhesively bonded joints against environmental influences                                                                                                                                                                                                 | As BS EN 2243: Parts 1 and 2                                           | Single lap shear (BS EN 2243:Part 1) and peel - metal-metal (BS EN 2243: Part 2)<br>50±3°C for 30, 60 or 90 days, 95-100% RH, or 70±3°C for 10, 20 or 30 days, 95-100% RH<br>Single lap shear (EN 2379)<br>Test fuel & anti-icing fluid: 40±2°C for 30, 60 or 90 days<br>Hydraulic fluid: 70±2°C for 15, 30 or 60 days<br>Peel - metal-metal (ISO 3768)<br>Salt spray: 35±2°C for 30, 60 or 90 days | As appropriate                                                                                                                                                                              |
| BS EN 60068-2-1: 1993  | Specifies test conditions for cold environments:<br>Test Aa. Non-heat dissipating specimen with sudden change of temperature<br>Test Ab. Non-heat dissipating specimen with gradual change of temperature<br>Test Ad. Heat dissipating specimen with gradual change of temperature | Not specified                                                          | Temperature: -65°C, -55°C, -40°C, -25°C, -10°C, -5°C or +5°C<br>Duration: 2 hrs, 16 hrs, 72 hrs or 96 hrs<br>Test Aa - specimen at ambient temperature introduced into test chamber at test temperature<br>Tests Ab and Ad - specimen and chamber both at ambient temperature to start, temperature decreased at 1K/min to test temperature, then increased at 1K/min after test duration           | Not specified, but:<br>Tests Aa and Ab - climate chamber with forced air circulation<br>Test Ad - distance from specimen to wall = min 15 cm, volume of chamber = min 5X volume of specimen |