

Measurement Good Practice Guide No. 28

Durability Performance of Adhesive Joints

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Abstract:

Considerable effort is required in selecting adhesive systems and optimising process variables to maximise long-term strength retention under hostile environments. This document is intended to give guidance on the selection and use of test methods and environmental conditioning procedures (including accelerated testing) for generating design data and for quality assurance purposes. The document is primarily concerned with structural applications. Guidance is provided on specimen preparation, hot/wet conditioning and testing of bulk adhesives and adhesive joints. Static, cyclic fatigue and creep rupture testing are covered. Consideration is given to the effect of material and geometric factors on joint performance under static, cyclic and creep loading, and hostile environments.

The guide also provides a summary of surface analytical techniques, thermal analysis tools and non-destructive testing (NDE) techniques for inspecting bonded joints before, during and after testing.

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Glossary of Terms

(Based on BSI and ASTM definitions)

Accelerated ageing test: Short-term test designed to simulate the effects of longer term service conditions.

Adherend: Body that is, or is intended to be, held to another body by an adhesive.

Adherend failure: Failure of a joint in the body of the adherend.

Adhesion: State in which two surfaces are held together by interfacial bonds.

Adhesive: Non-metallic substance capable of joining materials by surface bonding (adhesion), the bonding possessing adequate internal strength (cohesion).

Adhesive failure: Failure of an adhesive bond such that separation appears to be at the adhesive/adherend interface.

ASTM: American Society for Testing and Materials.

Bond: The union of materials by adhesives.

Bondline: The layer of adhesive which attaches two adherends.

Bond strength: The unit of load applied to tension, compression, flexure, peel, impact, cleavage, or shear, required to break an adhesive assembly with failure occurring in or near the plane of the bond.

BSI: British Standards Institute

Butt joint: Joint in which the plane of the bond is at right angles to a major axis of the adherends.

Bulk adhesive: The adhesive unaltered by the adherend.

Cleavage: Mode of application of a force to a joint between rigid adherends which is not uniform over the whole area, but results in a stress concentrated at one edge.

Cohesion: The ability of the adhesive to resist splitting or rupture.

Cohesive failure: Failure within the body of the adhesive (i.e. not at the interface).

Creep: The time-dependent increase in strain resulting from a sustained load.

Double lap joint: Joint made by placing one or two adherends partly over one or two other adherends and bonding together the overlapped portions.

Dry strength: Strength of an adhesive bond dried under specified conditions.

Durability: The endurance of joint strength relative to the required service conditions.

Environmental test: Test to assess the performance of an assembly under service conditions.

Fatigue life: Number of cycles necessary to bring an adhesive bond to the point of failure when the bond is subjected to repeated cyclic stressing under specified conditions.

Fatigue strength: Force that a joint will withstand when the force is applied repeatedly for an infinite number of cycles.

Fillet: Portion of an adhesive that bridges the adherends outside the bondline.

Glass transition: Reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one.

ISO: International Standards Organisation.

Lap joint: Joint made by placing one adherend partly over another and bonding together the overlapped portions.

Peel: Mode of application of a force to a joint in which one or both of the adherends is flexible and which the stress is concentrated at a boundary.

Peel ply: A layer of resin free material used to protect a laminate for later secondary bonding.

Plasticisation: Increase in softness, flexibility, and extensibility of an adhesive.

Post-cure: Further treatment by time and/or temperature of an adhesive to obtain the required properties by curing.

Porosity: A condition of trapped pockets of air, gas or vacuum within a solid material.

Primer: A coating applied to a surface, prior to the application of an adhesive, to improve the performance of the bond.

Scarf joint: Joint made by cutting identical angular segments at an angle less than 45° to the major axis of two adherends and bonding the adherends with the cut areas fitted together to be coplanar.

Service life (N): Number of stress cycles applied to a specimen until it has reached the chosen end of the test.

Shear: Mode of application of a force to a joint that acts in the plane of the bond.

Shelf life: The period for which the components of the adhesive may be stored, under the conditions specified by the manufacturer, without being degraded.

Strain: Unit change due to force in size of body relative to its original size.

Stress: Force exerted per unit area at a point within a plane.

Stress-cycles (SN) curve: Curve, allowing the resistance of the material to be seen, which indicates the relationship observed experimentally between the service life N and maximum stress.

Stress-strain diagram (or curve): A diagram in which corresponding values of stress and strain are plotted against each other.

Structural bond: Bond which is capable of sustaining in a structure a specified strength level under a combination of stresses for a specified time.

Substrate: A material upon which an adhesive is applied.

Surface preparation (or treatment): Physical and/or chemical treatments applied to adherends to render them suitable or more suitable for adhesive bonding.

Tension: Mode of application of a tensile force normal to the plane of a joint between rigid adherends and uniformly distributed over the whole area of the bondline.

Thermoset: A resin that is substantially infusible and insoluble after being cured.

Traveller: A test specimen used for example to measure moisture content as a result of conditioning.

Wet strength: Strength of an adhesive bond determined immediately after removal from a liquid in which it has immersed under specified conditions.

Yield stress: The stress (either normal or shear) at which a marked increase in deformation occurs without an increase in load.

Yield strain: The strain below which a material acts in an elastic manner, and above which it begins to exhibit permanent deformation.

Executive Summary

This measurement guide aims to provide guidance to technologists, laboratory staff and quality assurance personnel on the selection and use of test methods and accelerated ageing regimes to determine the durability of adhesively bonded joints to combinations of heat, moisture and mechanical load. Guidance is provided on static, cyclic fatigue and creep rupture testing. Consideration is given to the effect of material and geometric factors on joint performance under static, cyclic and creep loading, and hostile environments. The guide is concerned with adhesives used in structural applications involving substrates fabricated from either metals or fibre-reinforced plastic composites. The latter will include only thermosetting resin systems reinforced with either glass or carbon fibres.

The intention of the guide is to provide designers and users with sufficient information which, when coupled with their own expertise, can be used to select the appropriate test methods for producing design data and to enable initial screening of adhesive/adherend/surface treatments. If the intention is to generate design data, then the guide should be used in conjunction with the appropriate structural design codes. The guide assumes some basic knowledge of the materials and techniques involved, and is not intended as a text book. There are a number of published works, which provide a comprehensive coverage of adhesive technology and preliminary design [1-7]. There is also a Guide to “The Structural Use of Adhesives” produced by the Institution of Structural Engineers [8]. The intention of the guide is to complement this published work, which is considered to be an invaluable reference. It is recommended that specialist advice be sought from adhesive manufacturers on adhesive selection, use of associated technologies and health and safety requirements.

Correct surface preparation is essential for ensuring both initial adhesion and long-term joint durability. Although the guide provides advice on the surface preparation of the commonly used structural materials, it only covers the main steps in each process. It is essential that expert advice from the adhesive manufacturer is obtained and that the detail requirements specified by the manufacturer are completely satisfied. The Guide provides a summary of useful surface analytical and thermal analysis techniques that can be used for analysing the morphology, and chemical and physical degradation of both the adhesive and surface layers of the substrate. Non-destructive evaluation (NDE) techniques for inspection of bonded joints are also briefly discussed. The emphasis being on inspection of test specimens rather than in-service inspection of bonded structures.

1. SCOPE

Stress analysis of adhesive joints requires a database of basic engineering properties of the adhesive, adherend and joint geometry. Material properties required are listed below [5]:

- Adhesive shear modulus
- Adhesive elastic tensile or compressive modulus
- Adhesive Poisson's ratio
- 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 (composite adherends only)

Numerous test methods exist for characterising adhesives and bonded joints. A number of these may be used to determine fatigue resistance, environmental durability and creep behaviour. Adhesive tests can be divided into those methods that provide comparative 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 (i.e. ranking of 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 (BSI) and International Standards Organisation (ISO) is presented in NPL Report CMMT(A)61 [9].

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. The second approach for determining engineering properties of adhesives is to use especially designed joint geometries with a thin bondline, 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 adhesive layer, free of stress concentrations.

Ideally, the test method should employ simple and easily prepared specimens with testing, and data collection and analysis being relatively straightforward and economic.

This Guide is mainly concerned with test methods and environmental conditioning procedures (including accelerated testing) suitable for use with structural adhesives. These methods can be used for generating design data and for quality assurance purposes. Guidance is provided on specimen preparation, environmental conditioning and testing of bulk adhesives and adhesive joints. Static, cyclic fatigue and creep rupture testing are covered. Consideration is given to the effect of material and geometric factors on joint performance under combined cyclic or creep loading and hostile environments.

Appendix I shows a list of recommended test methods for determining input data for the design and analysis of bonded joints. In addition, the table includes a number of relevant standards for durability testing.

The guide also includes generalised surface preparation techniques for metal and polymer matrix composite (PMC) substrates and a summary of useful surface analytical and thermal analysis techniques that can be used for analysing the morphology, and chemical and physical degradation of both the adhesive and surface layers of the substrate. Non-destructive evaluation (NDE) techniques for inspection of bonded joints are also briefly covered. A list of some of the organisations which can provide specialist advice are provided at the back of the guide along with relevant standards and publications.

2. Measurement of Bulk Adhesive Properties

Bulk adhesive testing is generally the preferred approach for generating engineering data because of the relative simplicity of specimen fabrication and testing, however, this approach is fraught with problems. Bulk adhesive specimens can be cast and machined to the required shape (e.g. dumbbell tensile specimens). Many liquid and film adhesives can be cast into bulk specimens without the need for machining. This section provides advice on specimen preparation, test methods and standards, and environmental conditioning and testing of bulk adhesives. Detailed coverage of individual methods may be obtained in referenced standards.

2.1 Specimen Preparation

Tests on bulk adhesive specimens can provide reliable elastic and strength property data for design purposes. However, there are a number of key points that should be considered when producing bulk adhesive specimens [10-16]:

- 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. Specimens should be prepared using methods that minimise the inclusion of air in the test specimens.
- The cure state of the bulk adhesive specimens used to obtain the mechanical properties data should be similar to that of the adhesive layer in the adhesive joint [11-12]. Failure to achieve similar thermal histories can result in significant differences in material properties.
- Adhesives should be fully cured prior to conditioning and testing otherwise the adhesive will continue to cure, thus invalidating the test data.
- Adhesives have a low thermal conductivity. This may prevent dissipation of heat generated by exothermic cure reactions [11-12], which can occur when casting bulk specimens. Overheating can result in material degradation. The problem is exacerbated with increasing thickness. Temperatures in the adhesive should be monitored throughout the cure cycle using a thermocouple embedded in the adhesive.
- 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 in the material. This is an undesirable situation, as tensional strain at the surface enhances environmental stress cracking.
- Bulk adhesive properties will be affected by the conditions experienced after cure. For example, adhesive specimens can absorb moisture under standard laboratory conditions (23 °C, 50% RH), which can reduce the glass-transition temperature (T_g) and mechanical properties of the adhesive.
- Handling adhesives can be hazardous to human health, thus COSHH procedures should be followed to minimise operator exposure.

Joint specimens, due to the additional thermal mass, are slower to heat than bulk test specimens and therefore the final temperature of the adhesive joint at the end of the cure period can be significantly lower than in the bulk adhesive. Real-time monitoring of material property development in bulk adhesive specimens can be achieved using either ultrasonic or rheological techniques [16]. Dynamic Mechanical Analysis (DMA) measurements can be used to compare the final state of cure of the materials (see Section 6.2) [17].

Recommended procedures for the preparation of bulk specimens of adhesives are given in the ISO standard ISO 15166 [13-14]. The standard considers two part adhesives cured at ambient or elevated temperatures (Part 1) [13] and single component systems requiring an elevated temperature to cure the adhesive (Part 2) [14] - see also references [10-11]. Test specimens may either be moulded to shape or cut from manufactured plaques. It is important that the specimen is free of surface damage (i.e. scratches and nicks). To minimise the deleterious effect of surface scratches,

which may cause premature failure, the edges and faces of the specimens should be carefully polished to remove any surface defects.

2.2 Test Methods and Standards

2.2.1 Tension

Tensile properties (i.e. modulus of elasticity or Young's modulus E , tensile strength, failure strain and Poisson's ratio ν) can be obtained by monotonic loading of a waisted specimen in tension. Specimens are waisted to ensure that the maximum strain in the specimen occurs in the middle of the gauge-length [10]. No end tabs are required. Standard geometries for testing plastics, as specified in ISO 3167 [18], are also suitable for testing adhesives. ISO 527-2 [19] is the recommended standard for determining the tensile properties of unreinforced polymers (including adhesives) under constant deformation rate. This standard includes several specimen configurations for testing plastics.

Longitudinal and transverse strain can be measured using either strain gauges, contacting extensometers or video extensometers (non-contact technique) [10]. It is important when using contact techniques, such as strain gauges and extensometers, that the transducer(s) are capable of functioning within the test environment and have suitable response time. Strain gauges are not recommended for adhesive characterisation due to: (i) stiffening effects on low stiffness materials; (ii) limited range of operation; and (iii) tendency to act as failure initiation sites on bulk adhesives. Care should be taken to avoid inducing surface damage (e.g. scratches and nicks) when attaching extensometers to the test specimen or producing gauge marks for video extensometry. Non-contact extensometers should be employed to determine failure strains, whilst contact extensometers are the preferred method for accurate measurement of small strains (i.e. determination of Young's modulus and Poisson's ratio).

Tensile tests, although designed for use at ambient conditions are compatible with long-term testing such as creep or cyclic fatigue loading under non-ambient temperatures and hostile environments. All attachments should be environmentally resistant to the test conditions. It may be necessary to regularly coat threaded surfaces with a protective grease.

2.2.2 Shear

The V-notched beam method (ASTM 5379 [20]) can be used for characterising the shear properties of bulk adhesives [21-22]. The ASTM method employs a special fixture for loading a double edge-notched, flat rectangular specimen (Figure 1) with 90° angle notches cut at the edge mid-length with faces orientated at $\pm 45^\circ$ to the longitudinal axis. The specimen is 76 mm long, 20

mm wide and between 3 and 4 mm thick. Specimens with a thickness less than 3 mm require adhesively bonded tabs (~1.5 mm thick) to prevent out-of-plane bending or twisting which could lead to premature failure. Local crushing near the inner loading regions can also be avoided by the use of tabs. The test fixture is monotonically loaded in compression. The test method is suitable for cyclic fatigue, creep and environmental testing. When cyclic loading specimens, consideration needs to be given the possibility of frictional effects between the bearing post and the movable upper grip, and fretting at the loading points.

Shear strain is measured with biaxial strain gauges (1-2 mm gauge-length) bonded at $\pm 45^\circ$ to the longitudinal axis onto both sides of the test specimen. This enables strain averaging to account for non-uniform specimen loading to produce more accurate and consistent modulus values. A keyed bearing post will reduce out-of-plane bending in the specimen by eliminating possible rotation between the bearing post and movable upper grip. The failure process is highly dependent on the microstructure of the material. Tensile failure (Figure 2) is characteristic of brittle polymers (e.g. untoughened epoxy adhesives). For these materials, ultimate failure stress does not correlate with shear strength. Thermoplastic polymers and toughened adhesives tend to undergo shear yielding along the notch axis (Figure 2).

Alternative methods for measuring shear properties include: (i) plate twist (ISO 15310 [23]) - shear modulus only; (ii) Arcan method [10, 21-22]; and torsion of cylindrical rod [9-10].



Figure 1: V-notched beam test fixture and specimen

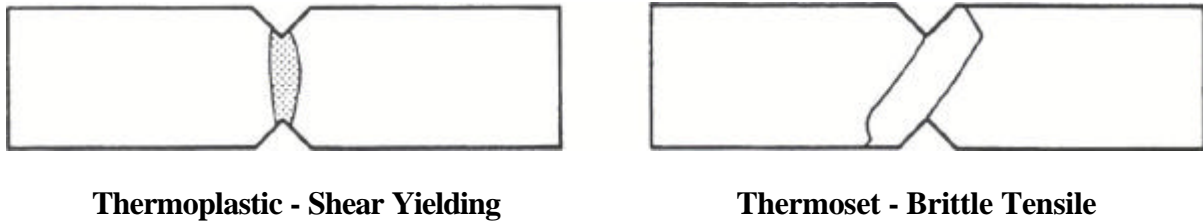


Figure 2: Typical failure modes for isotropic V-notched beam specimens

2.2.3 Compression

Current options for determining compressive properties of adhesives are limited. ISO 604 [24] specifies a short rectangular block 12.7 to 25 mm thick with a 13 mm x 13 mm square cross-section. The method is only suitable for generating elastic property data for thick sections. ASTM D 695 [25] is suitable for measuring elastic and strength properties of rigid plastics and adhesives. The test consists of direct compression loading a small waisted specimen 80 mm long and ~3 mm thick. A support jig is used to prevent buckling induced failure. Strain gauges are recommended for measuring longitudinal and transverse strains. Both methods are probably suitable for creep and environmental testing.

2.3 Moisture Conditioning and Testing

Conditioning is the process of exposure of material to an environment prior to subsequent testing. Most polymeric materials (e.g. adhesives and PMCs) are capable of absorbing small, but potentially damaging amounts of moisture from the surrounding environments with the degree of degradation that occurs being linked directly with the amount of moisture absorbed. The absorbed water may adversely affect an adhesive in a number of ways: (i) dimensional changes (swelling); (ii) reduction in the glass-transition temperature T_g of the resin; and (iii) reduction in mechanical properties. This will effectively lower the maximum service or operating temperature of the adhesive. This section provides advice on moisture conditioning of bulk adhesive and testing of adhesive specimens that have been exposed to either hot/humid environments or immersed in water at elevated temperatures.

2.3.1 Moisture Conditioning

The two main types of basic moisture conditioning are: (i) fixed-time conditioning, where a test specimen is exposed to a conditioning environment for a specified period of time; and (ii) equilibrium conditioning, where a specimen is exposed until the material reaches equilibrium with the conditioning environment. The first technique is routinely employed for screening adhesive systems. This approach results in non-uniform moisture distribution through the thickness of the test specimen. Test data obtained from specimens conditioned in this manner are only considered suitable for comparing different batches of the same material or for quality control tests. It is essential that test specimens used in this manner are identical in dimensions and have similar surface finishes.

Ideally, comparative studies of water absorption properties of materials should be carried out only using the equilibrium moisture content of polymeric materials exposed to identical conditions. Comparisons between adhesive systems with different moisture absorption characteristics are possible if the materials are pre-conditioned to equilibrium. The thicker the material the longer the time required to reach equilibrium, hence the use of relatively thin specimens to determine the “through-the-thickness” moisture diffusion coefficient.

The International standard BS EN ISO 62 [26] describes a procedure for determining the moisture absorption properties and/or diffusion coefficients in the “through-the-thickness” direction of flat and curved solid plastics. BS EN ISO 62 is suitable for use with adhesive and PMC specimens. The method can be applied to vapour exposure and liquid immersion.

Conditioning usually consists of exposing pre-dried specimens to a steady-state environment (i.e. constant temperature and constant moisture exposure level) and measuring the moisture gain (i.e. average moisture content) for a prescribed period of time or until the specimen reaches moisture equilibrium. The amount of water absorbed by the test specimen is determined by measuring its change in mass (i.e. difference between initial mass and the mass after exposure). All surfaces are in contact with the test environment. It is recommended that when determining moisture absorption properties that square shape specimens be used for homogeneous plastics and adhesives. In this case, specimen dimensions and tolerances should comply with ISO 294-3 [27]. For reinforced plastics, it is recommended that square specimens also be used with a width $w \leq 100 \times$ nominal thickness d (typically 2 mm).

It is recommended that specimens be pre-dried in an oven maintained at 50 ± 2 °C until the specimen weight reaches a constant value. The temperature of the drying oven should not exceed the maximum operating temperature of the adhesive system. Specimens are removed at fixed intervals (typically 24 hrs) and allowed to cool to room temperature in a desiccator before being weighed. After weighing, the specimen is returned to the oven and the process is repeated until the mass of the specimen is constant (zero datum level). Specimens should be free of voids in order to ensure accurate moisture absorption measurements. To minimise moisture uptake prior to pre-conditioning, specimens are stored in a desiccator (sealed container with desiccant), at room temperature. It should be noted that under standard laboratory conditions many adhesives absorb significant levels of moisture.

Pre-conditioned specimens need to be wiped with a clean cloth to ensure all surface water is removed prior to weighing. Damage may accumulate during long-term conditioning, and hence handling and monitoring of test specimens should be minimal. This is particular pertinent to those specimens used for generating engineering data. Travellers are required to monitor specimen

moisture content throughout the environmental history (i.e. manufacture, storage, pre-conditioning and testing). Traveller specimens should have identical material properties, geometry and processing history as the test specimen. It is essential that moisture content prior to pre-conditioning be established.

Conditioning is often carried out at elevated temperatures or humidities to accelerate moisture uptake. Care should be taken to avoid exceeding the T_g of the material. The recommended maximum conditioning temperatures are 45 °C and 70 °C for 120 °C and 180 °C cure systems, respectively [28]. These temperature levels should not be exceeded. It is recommended that information on the moisture and temperature response of the material be obtained prior to environmental conditioning.

The rate of moisture uptake is fairly rapid in the early stages of conditioning with the rate of moisture uptake decreasing with time (Figure 3). It is therefore necessary to make frequent weight measurements in the early stages (3-4 measurements on day one) followed by at least two readings per day for the remainder of the first week.. At least one reading per day is required per day for the second week, followed by a gradual decrease in frequency as the rate of weight gain diminishes. It is recommended that weighing be carried out at approximately equal intervals of $(\text{time})^{1/2}$. The derived moisture content M can be determined as follows:

$$M = \frac{(W_{WET} - W_{DRY})}{W_{DRY}} \times 100\%$$

where the wet and dry weights are denoted by W_{WET} and W_{DRY} . A precision analytical balance capable of reading to within 0.0001 g is required. Accurate records need to be kept on pre-conditioning sequence including relative humidity, temperature and time, measured weights and derived moisture contents.

At temperatures well below the T_g of the wet polymer, water absorption of most adhesives correlates well with Fick's laws (see Annex A of BS EN ISO 62 [26]). The diffusion coefficient, independent of time and concentration, can be calculated from the Fickian diffusion curve. The diffusion coefficient D is determined from the linear region of the Fickian diffusion using the following relationship [28]:

$$D = \frac{p}{16} \left(\frac{d(M_2 - M_1)}{M_\infty(\sqrt{t_2} - \sqrt{t_1})} \right)^2$$

where M_∞ is the equilibrium moisture concentration, M_1 is the moisture uptake after time t_1 , M_2 is the moisture uptake after time t_2 and d is the thickness. The moisture equilibrium concentration or content corresponds to the asymptotic value on the Fickian diffusion curve.

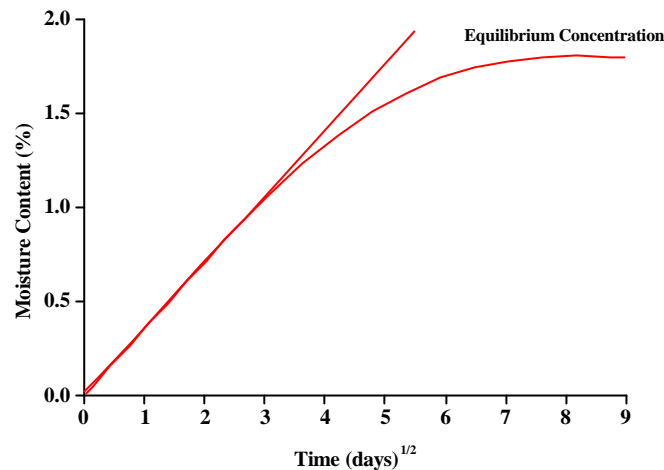


Figure 3: Fickian diffusion curve

Fickian diffusion behaviour [26] is valid for homogeneous materials and for reinforced PMCs tested below their T_g value. However, some two phase adhesive systems may require a multi-phase absorption model. Further information on mathematical modelling of diffusion in solids can be obtained from references [26, 28-29].

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 time required to reach full equilibrium. 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% equilibrium value can be justified in terms of a non-significant change in the material properties.

Although humidity conditions can be controlled using salt solutions, this procedure is not particularly reliable as it is difficult to maintain the required tolerances on humidity and temperature. The recommended procedure is to use an environmental cabinet which can control the temperature to within ± 2 °C and the relative humidity to within $\pm 5\%$. It is essential that boiler units of humidity cabinets be supplied from a deionised/distilled water reservoir in order to prevent salt deposits on test specimens, corrosion of bonded joints and scaling of the plumbing.

2.3.2 Measurement of Coefficient of Moisture Expansion

Determination of the coefficient of moisture expansion involves measuring the dimensional change of the material in the principal directions as a function of moisture concentration (i.e. moisture weight gains). Details of specimen dimensions and preparation (i.e. drying) are given in Section 2.3.1. Specimen dimensions and tolerances should comply with ISO 294-3 [27]. Moisture expansion or swelling can be measured with a micrometer and/or vernier calliper, or a travelling microscope.

Strain gauges are not particularly suited for this purpose due to environmental attack on the strain gauge adhesive. A reduction in accuracy of the strain gauge measurements can be expected with exposure time. In addition, the presence of strain gauges on the surface of the specimen may inhibit moisture absorption within the vicinity of the gauge. Embedding encapsulated strain gauges in the specimen offers the potential of continuous strain monitoring without the need to remove the specimen from the water bath, however, it is important that the strain gauge does not cause any local disturbance, which may affect the process of moisture diffusion.

Note: It is assumed that all absorbed moisture is translated into a change in resin volume. In fact, during the initial stages of conditioning, water may also be filling microvoids and cracks. A plot of swelling strain versus weight gain will show a change in gradient for high porosity materials.

2.3.3 Mechanical Testing of Pre-conditioned Specimens

For design purposes, a material test programme should measure both the moisture absorption properties of a material (i.e. diffusion rate and equilibrium content) and the resultant mechanical properties at equilibrium. Mechanical property measurements at intermediate moisture levels (based on average moisture contents) can be used to determine temporal and spatial distributions of stresses and strains within a bonded structure. Figure 4 shows tensile stress-strain responses under ambient test conditions for an adhesive that has been immersed in water for periods ranging from 30 minutes to 12 days. To evaluate worst case effects of moisture content on material properties, tests are performed on specimens that have been preconditioned to the design service moisture content. For aircraft, the worst-case environment is considered to be represented by a relative humidity of 85% RH and a temperature of 70 °C.

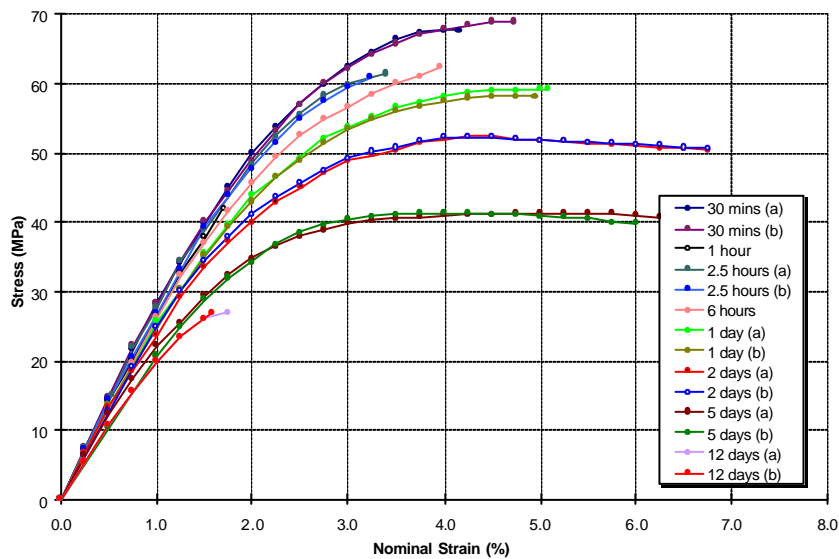


Figure 4: Tensile stress-strain curves for different water immersion periods

It should be noted that the material operational limit is influenced markedly by temperature and moisture content. Figure 4 shows a significant reduction in tensile stiffness and strength with increasing moisture content. The reduction in material properties will be more severe at elevated temperatures.

Most strain gauge adhesives are sensitive to moisture, which can often preclude bonding of strain gauges to the specimen prior to the preconditioning stage. Moisture attack of the strain gauge adhesive and strain gauges will occur from the top, edges and through the test specimen. It is therefore important to ensure that the adhesive selected for bonding the strain gauges remains unaffected for the entire duration of the test and that strain gauges and associated electrical wiring are suitably encapsulated. The strain gauge manufacturer should be contacted to obtain advice on adhesive selection and procedures for strain gauge protection. The preferred method is to use either contact extensometers or video extensometers for monitoring strain.

At elevated temperatures, preconditioned specimens tend to dry out during the test, although for static tests the effects are minimal provided testing is completed within 15 minutes of the specimen being removed from the conditioning environment. Methods of inhibiting moisture loss, such as encapsulating specimens with a sealant or enclosing the specimen in a polythene bag containing a salt solution appropriate to the humidity requirements of the test, are not practical. Travellers are required for monitoring the moisture content loss that occurs during the test. For coupon testing, it is common practice to allow a soak period of 10 minutes at the test temperature prior to testing. The purpose of “heat soaking” is to eliminate distortion due to non-uniform temperature distributions

3. Measurement of Adherend Properties

This section briefly summarises test methods and associated standards that can be used to measure elastic and strength properties of metallic and PMC adherends.

3.1 Tension

BS EN ISO 10002-1 [30] is the recommended method for measuring the tensile properties of metallic materials. ISO 527-2 [19] can also be adopted for measuring in-plane tensile properties (i.e. tensile modulus, tensile strength and Poisson’s ratio). For composites, the recommended in-plane methods are BS EN ISO 527-4 [31] for multidirectional composite laminates and BS EN ISO 527-5 [32] for unidirectional laminates (see also [33]). Tensile tests using straight edge specimens, as specified in ISO 527, are suitable for determining long-term performance of composite laminates exposed to combined stress (cyclic fatigue and creep) and hot/wet environments.

3.2 Compression

Currently, there is no ISO standard for the determination compressive properties of metallic materials. The recommended ASTM standard is ASTM E9 [34]. Compression standard BS EN ISO 14126 [35] is the preferred test specification for the determination of in-plane compressive properties of laminated composites. This standard allows for a range of shear face and end-loaded fixtures to be used (i.e. Celanese, IITRI and end-loading blocks). In all cases, the gauge-length is unsupported. ISO 14126 recommends using specimens 110 mm long, 10 mm wide with a gauge-length of 10 mm. The required thickness is 2 mm for continuous aligned materials and 2-10 mm for multidirectional laminates. Specimens need to be strain gauged on both faces and end-tapped (to prevent failure at the specimen ends). No particular problem is expected for the use of the test configurations for creep and environmental testing of metals or composites.

3.3 Shear

There is no standard at present for determining shear strength of metals. Shear modulus for these materials can be determined from tensile tests. BS EN ISO 14129 [36] is the recommended test method for determining in-plane shear modulus and shear strength of continuously aligned laminates. The test involves the application of uniaxial tension to a balanced $\pm 45^\circ$ laminate, 250 mm long, 25 mm wide and 2 mm thick. Specimen preparation and testing are similar to those specified in ISO 527-4 [27]. The test is terminated at 5% shear strain, thus shortening the test duration, which can be excessive for tough matrix systems. The 5% limit also minimises fibre rotation and heating effects. The peak load at or before 5% strain is taken as the shear strength. This test configuration is suitable for generating shear fatigue and environmental data. Internal generation of heat during cyclic fatigue testing could be a problem considering the number of interfaces present in 16 ply laminates.

3.4 Through-Thickness (T-T) Properties

Through thickness (T-T) properties are increasingly important for the detailed analysis of complex composite structures. Currently, there are very few standards at national or international level that can be recommended for determining T-T properties. A comprehensive experimental evaluation has led to the drafting of standards for T-T tension, compression and shear [37]. The tension and compression procedures enable the measurement of both T-T elastic and strength properties. ASTM D 5379 [20] can be used to determine T-T shear properties (see Section 2.2). Insufficient data exists to pass judgement on the suitability of the T-T tensile and compression methods for use under hostile environments or cyclic loading conditions.

4. Adhesive Joint Testing

An alternative approach for determining engineering properties of adhesives is to use specially designed joint geometries with a thin bondline. There are a number of problems associated with adhesive joint configurations. These are listed below:

- 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.
- 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 for accelerated testing.
- 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 [5] (see also Figure 5):

- 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 ends of the joints.

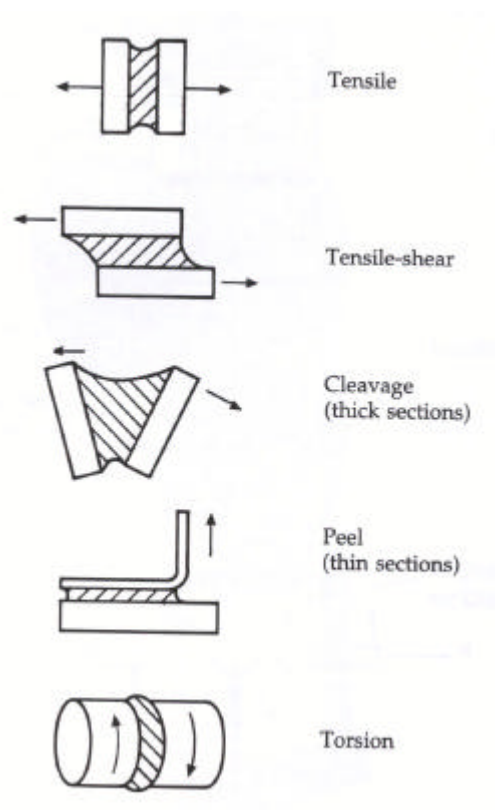


Figure 5: Basic loading modes experienced by adhesive joints

4.1 Tensile Properties

The cylindrical butt joint (Figure 6) can be used to test thin bondline specimens in tension, torsion or compression. The test provides (with difficulty) 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. The test is difficult to perform. Significant bending can be induced due to misalignment of the adherends or misalignment in the loading assembly. Care needs to be taken to ensure good alignment during specimen preparation (i.e. bonding of adherends) and testing. Small misalignment can severely reduce strength data. The loading assembly should be rigid and accurately aligned. Special equipment is required to ensure the latter. At least three extensometers placed at equi-spaced around the specimen circumference are required for monitoring deformation and to ensure bending loads are minimal. Variations of the test configuration have been included in ASTM 897 [38] and ASTM D 2095 [39] and BS EN 26 922 (ISO 6922) [40]. At present, there is no definitive standard test method.

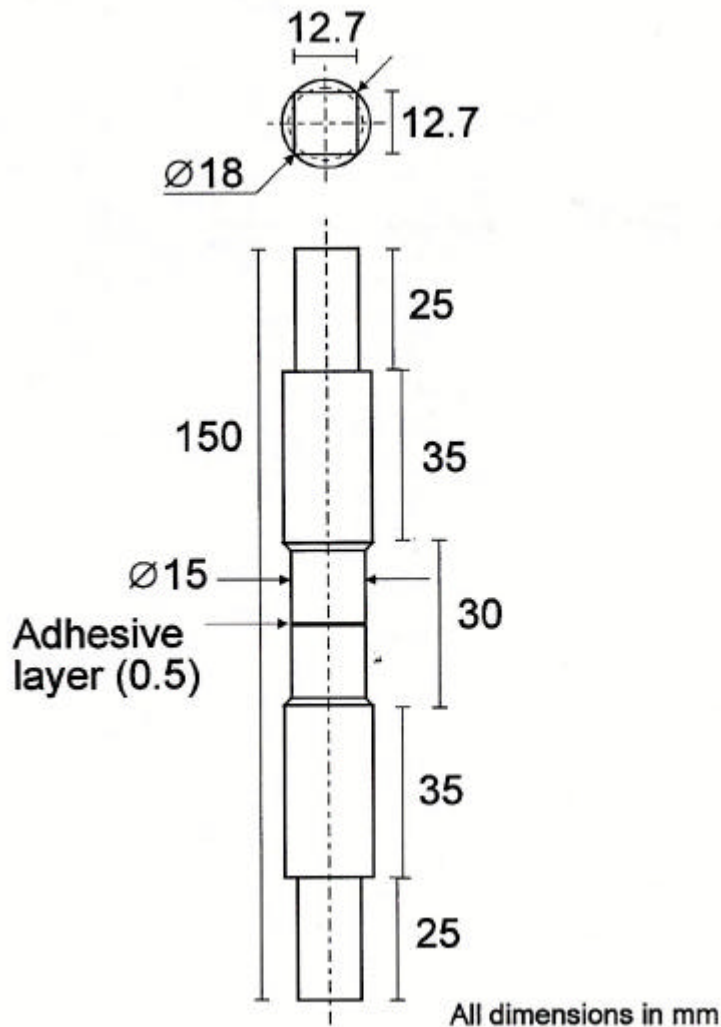


Figure 6: Tensile butt joint

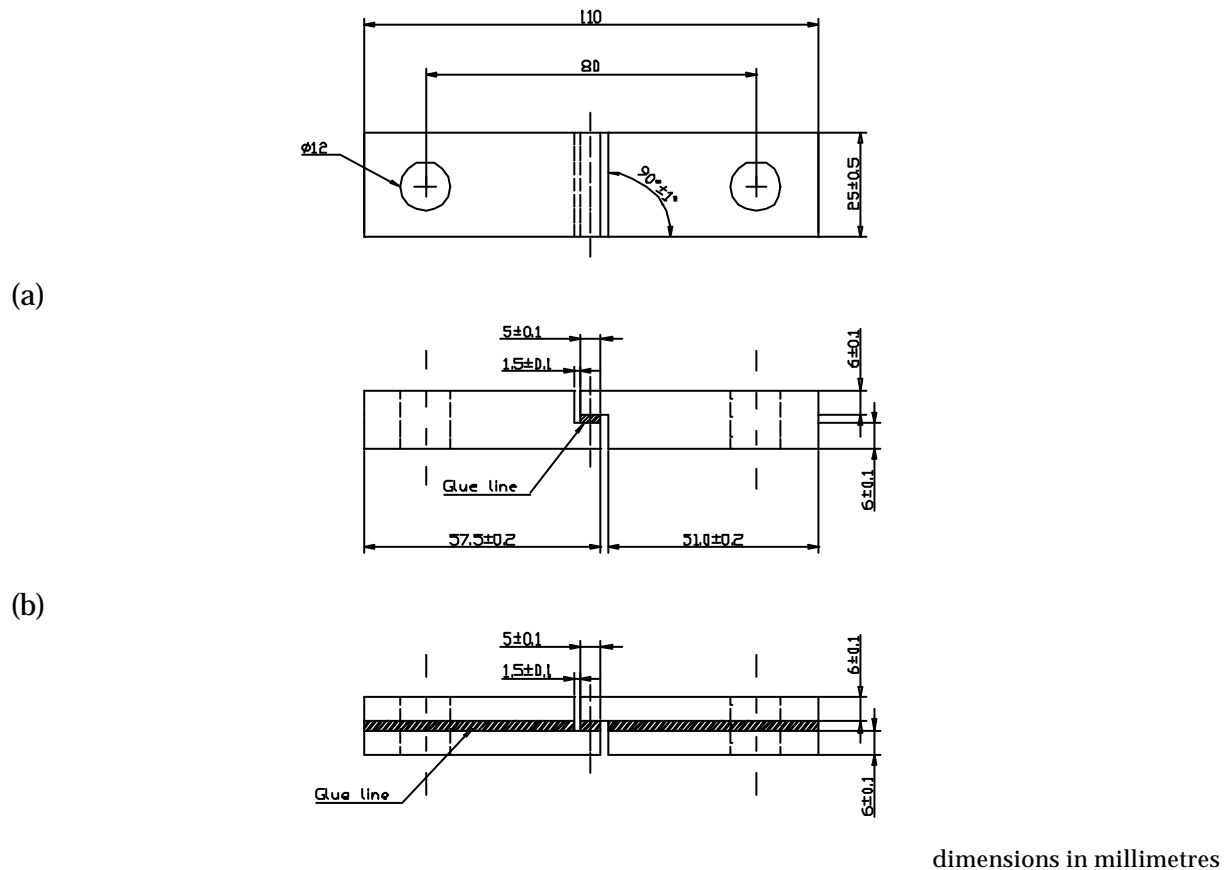
It is difficult to envisage using the butt joint for assessing long-term performance of adhesive joints under combined cyclic loading and hostile environments, considering the difficulties associated with this configuration (including monitoring of strain, which is relatively small). **Bulk adhesive tensile tests are the preferred approach for generating tensile data for design and analysis purposes.**

4.2 Shear Properties

Shear properties of an adhesive can be determined by applying uniaxial tension load to a specimen consisting of thick, rigid adherends, with a short overlap length. The specimen can be produced by either:

- (i) Bonding two pre-shaped bars together (Figure 7a); or
- (ii) Bonding two sheets together and then milling two parallel slots (Figure 7b).

The adherends shown in Figure 7b have a lower bending stiffness than the continuous geometry in Figure 7a. Consequently, the peel stresses at the ends of the adhesive in the specimen in Figure 7b will be higher than those in the specimen in Figure 7a. Since failure is generally initiated by these peel stresses, the specimen design shown in Figure 7b will probably fail at a lower stress and strain than the design shown in Figure 7a.



dimensions in millimetres

Figure 7: TAST specimen (tension): (a) pre-shaped adherends; (b) bonded adherends

ISO 11003-2 [41] specifies a specimen with an overall length of 110 mm, a width of 25 mm and overlap length of 5 mm. The ISO standard recommends an adherend thickness of 6 mm and a bondline thickness of 0.5 mm. Slots should be 1.5 mm wide. Load is introduced to the specimen preferably 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. The test method is suitable for measuring shear modulus and shear strength of adhesive joints under ambient and hostile environments.

The relative displacement of the adherends is measured using a purpose-built transducer located in the central region of the specimen or by non-contact strain measurement techniques (i.e. video extensometry and electronic speckle pattern interferometry (ESPI)). Force and displacement are measured from the start of application of the load until fracture of the specimen. The shear stresses and strains are then calculated from bond dimensions.

4.3 Quality Assurance Testing

This section provides guidance on the use of lap shear (single and double) and T-peel tests, which are used on a routine basis for quality assurance purposes and for ranking adherend/adhesive/surface treatment combinations. Details on other test methods (including fracture toughness) are available in reference [9].

4.3.1 Single-Lap Shear Test

The single-lap test (Figure 8) essentially consists of two rectangular sections, typically 25 mm wide, 100 mm long and 1.5 to 2.0 mm thick, bonded together, with an overlap length ranging from 12.5 to 25 mm [8]. Variations of this test method are included in both national and international standards [43-45]. End tabs, cut from the same material as the adherend sections, are adhesively bonded to the specimen. The end tabs are typically 37.5 mm in length. The end tabs have been introduced to reduce (not eliminate) the eccentricity of the load path that causes out-of-plane bending moments, resulting in high peel stresses and non-uniform shear stresses in the adhesive layer. BS EN 1465 [43] does not specify the use of end tabs, but specifies that the long axis of the specimen coincides with the direction of the applied force through the centre line of the grip assembly.

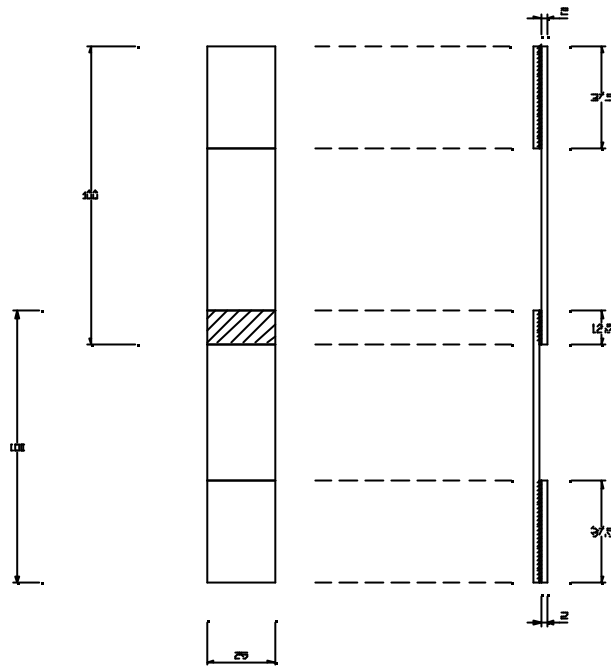


Figure 8: Schematic of single-lap joint (dimensions in mm)

It is undesirable to exceed the yield point of the adherend in tension, hence the overlap length L should be sufficient to ensure adhesive failure occurs before the adherend yields. The maximum permissible length, which is a function of thickness and stiffness of the adherend, can be determined from the following equation [44]:

$$L = \frac{S_Y t}{1.5t}$$

where S_Y = yield stress of the adherend, t = average shear strength of the adhesive and t = adherend thickness, in metres.

The single-lap specimen is easy to prepare and test. A bonding fixture is recommended to ensure correct overlap and accurate alignment of the adherend. Special care is needed to ensure that bond line thickness is uniform (e.g. ballontini glass spheres, wires and clamping arrangements). Alternatively, large test panels (typically 180 mm wide) capable of providing 6 specimens can be made and then cut into specimens. Adherend surfaces, unless specified otherwise, should be prepared according to ISO 4588 [46] for metals and ISO 2818 [15] for polymer matrix composites (PMCs). Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e. adherend bending). Testing is conducted using standard tension/compression mechanical test equipment with a suitable pair of self-aligning (manual or servo-hydraulic wedge-action) grips to hold the specimen. The recommended standard for tension-tension fatigue is BS EN ISO 9664 [47].

4.3.2 T-Peel Test

The T (or 180°) peel test is used for determining the relative peel resistance of adhesive joints manufactured from flexible metallic adherends (e.g. thin steel or aluminium alloy sheet). The term flexible refers to the ability of the adherend to bend through 90° without breaking or cracking. The T-peel test is suited for use with metal adherends, but other flexible adherends (e.g. PMCs) may also be used. This test method has been adopted by most standards bodies and is widely used to evaluate environmental durability of adhesively bonded systems.

Specimens (Figure 9) are typically 25 mm wide, have a minimum bonded length of 150 mm, and 50 mm long arms [48-50]. The recommended thickness is 0.5 mm for steel and 0.7 mm for aluminium. It may be necessary to use thicker adherends (e.g. 1.5-2.0 mm) to minimise bending of the specimen arms. Adhesive layer thickness is not specified. The force is applied to the unbonded ends of the specimen. The angle between the bond line and the direction of the applied force is not fixed. It is recommended that the test specimen has an external radius **R_o** of 6.5 mm and a 50% adhesive fillet. Definition of fillet size is shown in Figure 10 [51]. The fillet size is controlled using a special tool shaped to fit within the bonded joint. The fillet size is the most important parameter controlling T-peel static strength. As the fillet size increases, the strength of the joint also increases.

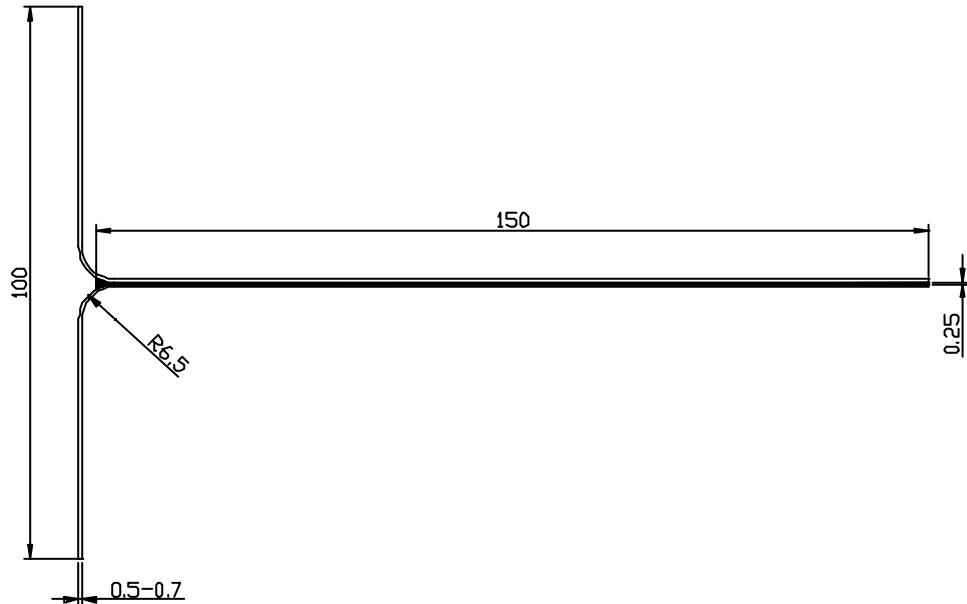


Figure 9: Schematic of T-peel joint (dimensions in mm)

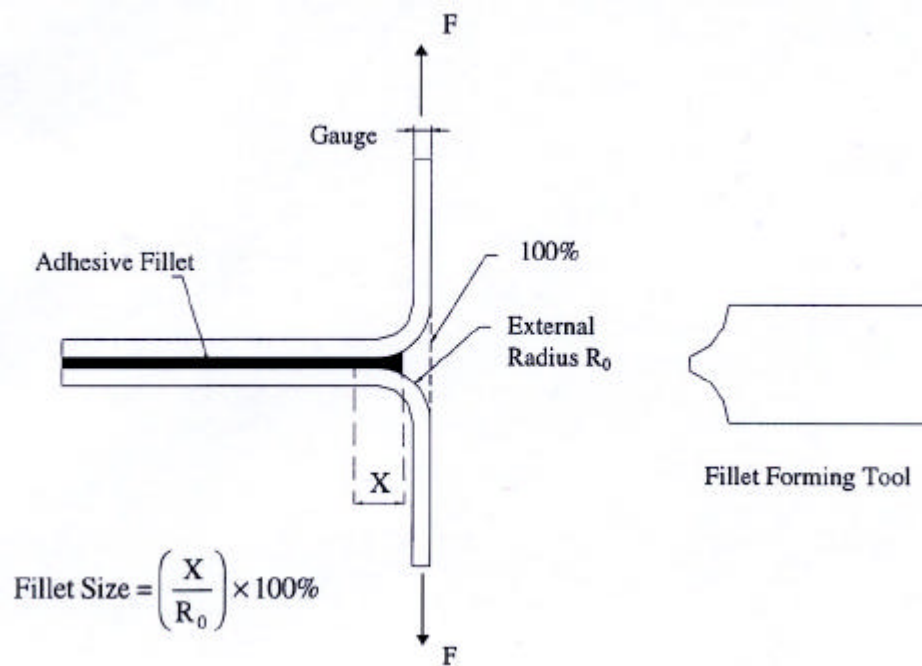


Figure 10: Definition of fillet size in T-peel joint [51] - including fillet forming tool

4.4 General Issues Relating to Environmental Testing

This section briefly covers general issues relating to environmental conditioning testing of bonded joints. Surface preparation or analysis techniques are not included in this section. Common surface preparations of metal and PMC adherends are described in Section 5, whilst Section 7 provides a brief summary of surface and chemical characterisation techniques. The standard 'BS EN ISO 10365 [52] provides guidance on failure mode interpretation. This standard applies to all mechanical tests performed on bonded assemblies; independent of adhesive and adherend materials used in the structures.

For the purpose of the measurement of the adhesive properties, steel adherends are recommended because of the materials high stiffness. For ambient tests, a suitable steel is XC18 or E24 grade 1 or 2. However, corrosion-resisting steel (e.g. A167, Type 302) or titanium alloy (e.g. Ti-6Al-4V) are preferable for environmental testing. A bonding fixture is recommended to ensure correct overlap and accurate alignment of the adherends. Special care is needed to ensure that bond line thickness is uniform and free of voids. Assume the degradation process is irreversible and commences on completion of the cure cycle. Joints should be stored in a desiccator or bagged with a desiccant.

The basic procedures for environmental conditioning, monitoring moisture content and testing of bulk adhesive specimens also apply to adhesive joints (Section 2.3). Monitoring moisture in adhesive joints is relatively difficult in comparison with bulk adhesives. Corrosion products and moisture absorption of adherends (i.e. PMCs) can contribute substantially to changes in mass, thus resulting in large inaccuracies in moisture content measurements. For finite element modelling purposes, it is recommended that moisture diffusion coefficient values be determined using bulk adhesive data (see

BS EN ISO 62 [26]). For non-equilibrium moisture levels, the moisture distribution is non-uniformly distributed; as shown in Figure 11.

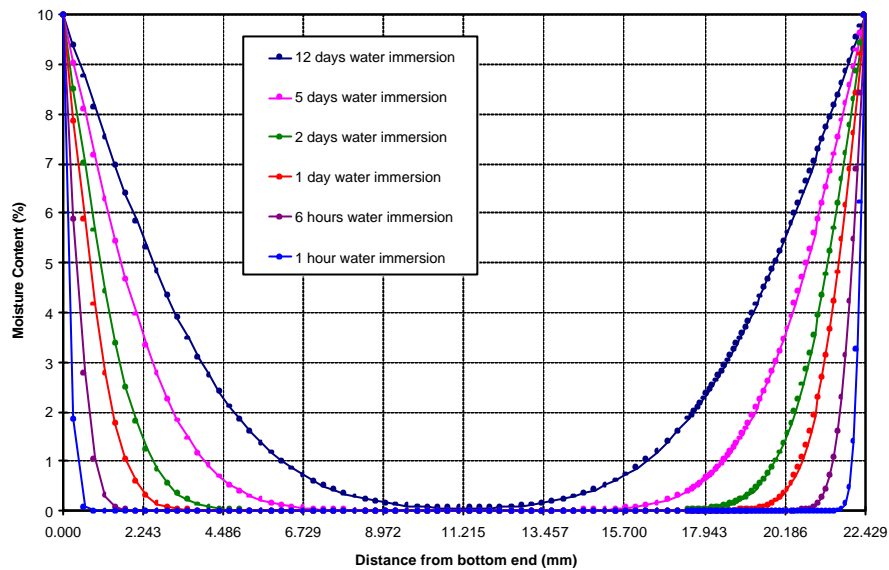


Figure 11: Typical moisture concentration distributions along an adhesive layer

4.5 Creep Rupture

The extent of creep damage and its importance is dependent primarily on the stress level at which irreversible damage occurs relative to the stress for complete failure (i.e. ultimate static strength). The degradation process is exacerbated under hot/wet conditions with the rate of degradation increasing with increasing temperature, humidity and mechanical stress. Mechanical acceleration methods tend to use stress levels that are significantly higher than stress level limits used in design, thus the limiting design strains are reached in shorter times than in actual service.

Two approaches have been adopted for assessing the degree of degradation under combined static load and environment:

- **Rate of strength loss with time (i.e. residual strength):** This approach determines the time taken for the strength of the joint to decline to a design stress limit, below which the joint is no longer considered safe.
- **Time-to-failure:** This approach attempts to determine the probable average life expectancy of a bonded joint at a prescribed stress level or to determine the percentage of failures that can be expected to occur within a given exposure period.

Creep tests can be carried out using:

- (i) Servo-hydraulic test machines;

- (ii) Dead-weight and lever creep testing machines;
- (iii) A screw jack in series with a load cell (Figure 12); and
- (iv) Self-stressing fixture where specimens are placed in either a tube equipped with a pre-calibrated spring system (Figure 13) for loading specimens or a circular ring.



Figure 12: Screw-jack test machines



Figure 13: Specimen loading tube

For small or medium size enterprises or a large series of tests, the use of a servo-hydraulic test machine is often not an economic option. A bank of small creep machines can be assembled at a considerably lower cost compared with the capital outlay involved with purchasing and operating servo-hydraulic units. Self-stressing fixtures (Figure 13), which are light and economic to produce and maintain, are particularly suited for field trials and for large batch testing. Care should be taken to ensure that the thermal mass of the tubes does not exceed the capacity of the conditioning cabinet, thus preventing correct maintenance of humidity and temperature.

Small single-lap and T-peel joints have been successfully tested using self-stressing tubes. Testing consists of placing specimens in a tube equipped with a pre-calibrated spring system for loading the specimens (BS ISO 14615 draft [53]). The spring system can be compressed and locked in place to apply the required load with the spring stiffness determining the load range. The amount of load is determined by measurement of the spring compression. The fixture specified in BS ISO 14615 is capable of loading a series of 3-6 specimens at a time. The specimens are bolted together with either stainless steel or polyamide bolts. The tubes should be suspended vertically within the environmental cabinet to ensure uniform exposure of the test specimens.

The stress tubes are inspected at frequent intervals to check on the condition of the test specimens (i.e. failed or intact). Failed joints are replaced with spacers and the remaining specimens re-stressed. The failure times are measured at which the first three specimens fail. When the third specimen fails, the remaining specimens are removed from the loading tube and tested to failure to determine residual strength. The average life-time of the failed specimens and the residual strength of the remaining specimens should be recorded.

The large uncertainty associated with time-to-failure measurements, especially at the high stress levels will require either electromechanical or optical devices to monitor load or deformation in order to accurately determine time-to-failure. Specimens loaded by springs can often be in an unstressed state for a considerable period of time (overnight or weekends) before the failed joint is replaced by a solid “dummy” and the string re-tensioned. There is also a tendency for surviving specimens to be damaged in the re-stressing process with the probability of occurrence increasing at high stresses. Creep/relaxation histories of specimens will be different due to the replacement of failed specimens and subsequent re-loading. This contributes further to the uncertainty of creep rupture data. For long term tests over months or years, this effect will probably be minimal.

Load levels need to be established for any particular system tested. Typically these are between 10 and 50% of the short-term strength of the joint. It is generally recommended that that sustained stress in an adhesive boned joint under service conditions should be kept below 25% of the short-term strength of the joint [8]. For joint characterisation purposes it is recommended that specimens

are mechanically loaded at each of five stress levels (i.e. 80%, 70%, 55%, 40% and 25% of the short-term strength of the joint). The large uncertainty associated with creep test results, especially those obtained under hot/wet conditions, implies that the current approach of conducting three tests per stress level is inadequate and that considerably more data points are required for generating reliable creep rupture curves for engineering design purposes. Five (preferably 10) per stress level with five stress levels (see above) per condition should provide a reasonable number of data points.

4.6 Cyclic Fatigue

Fatigue data are normally obtained at the highest frequency possible in order to minimise the duration of testing. Restrictions can arise from test equipment limitations (response time), time-dependent processes and hysteretic heating. Hysteretic heating, which increases with increasing load and frequency, can result in thermal softening of the adhesive, adversely affecting the fatigue performance of composite joint. Reliable data can be obtained at high frequencies provided the stress levels are low. Test frequencies of the order of 10 to 30 Hz can result in substantial heating, particularly in the grip regions.

It is recommended that the maximum acceptable temperature rise of the material surface should be limited to 10 °C above the test temperature. It may be necessary to stop testing to allow the specimen to cool. Alternatively, the test could be carried out in an environmental cabinet with a thermocouple attached to the specimen surface for monitoring and controlling the temperature of the test specimen, although the cooling rate may be too slow to be practical. Thermal imaging equipment can be used to monitor surface temperature.

The fatigue properties of a bonded joint are a function of the joint geometry and thus do not correspond to intrinsic properties of the adhesive. For joint characterisation purposes it is recommended that specimens are mechanically loaded at each of five stress levels (i.e. 80%, 70%, 55%, 40% and 25% of the short-term strength of the joint). Five specimens should be tested per stress level in order to generate sufficient data to establish the S-N (Stress-No of Cycles at Failure) curve. S-N curves permit the estimation of the confidence zone concerning the fatigue resistance of the joint. Tests tend to be terminated after 10^6 cycles if the specimen has not failed in order to reduce costs and time. It should be noted, that the fatigue performance of adhesive joints tends to decrease as the frequency decreases, and thus accelerated test data will be non-conservative. Deformation should be measured using contact extensometers. Video extensometers are not suited for measuring small strains, particularly at high strain rates.

4.7 Basic Design Considerations

Generally, the basic rules of good design of adhesive joints apply to most loading and environmental conditions (see [2-8]). For example, thickening the adhesive at the ends of an overlap through the

use of large adhesive fillets or by internal tapering can reduce peel and shear stresses at the ends of an overlap (Figure 14), thus improving creep and fatigue performance. It also provides added protection from environmental attack. Increasing the bondline spreads the strain over a larger volume, resulting in lower strain in the adhesive and therefore, a lower stress concentration. The taper ends of lap joints should have a thickness of 0.76 mm and a slope of 1/10.



Figure 14: Bevelled strap joint

The total overlap length must be sufficiently long to ensure that the shear stress in the middle of the overlap is essentially zero or at least low enough to prevent creep. Short overlaps can result in failure through creep-rupture. For single-lap specimens, the optimum overlap length is approximately 30 times the adherend thickness. Increasing the overlap lengths beyond this value does not result in substantial increases in static and fatigue performance. The low stress region in the middle of a long overlap contributes to joint strength by providing an elastic restoring force or reserve.

At ambient conditions, the normalised **S-N** curve (Figure 15) for adhesive joints can be approximated (rule of thumb) by a straight line fit as follows:

$$P_{MAX} / P_O = 1 - k \log Nf$$

where k is the fractional loss in strength per decade of cycles, Nf is the number of cycles to failure, P_{MAX} is the maximum load applied to the specimen, P_O is the ultimate strength of identically conditioned specimens measured at the fatigue test loading rate.

The lower the k value the better the fatigue performance. Table 1 shows typical k values for a number of metal and PMC joints bonded with epoxy adhesives. A scarf joint with a 30° taper has a far better fatigue performance than tests where peel stresses are the major cause of failure.

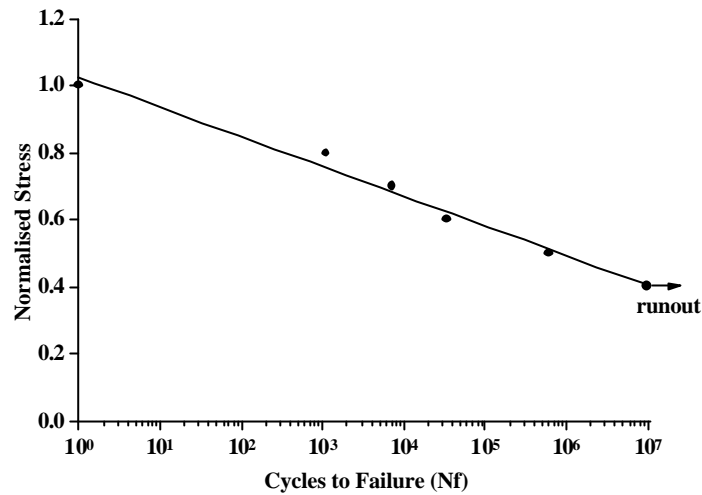


Figure 15: Normalised S-N curve for a tapered-strap joint

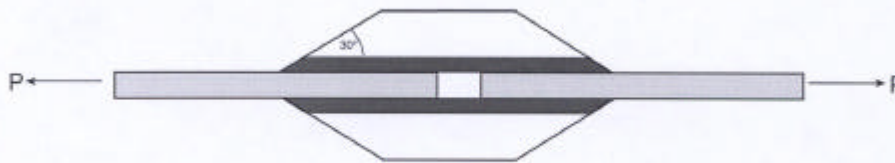


Figure 16: Tapered-strap joint

Table 1: Typical k Values for Bonded Joints Tested at Room Temperature

Joint Configuration	k
Scarf joint (aluminium with 30° taper)	0.055
Double-lap (titanium alloy)	0.075
Double strap joint (aluminium with tapered straps)	0.088
Single-lap (mild steel)	0.093
Double-lap (PMC)	0.097
T-Peel (mild steel)	0.130

It is important to accurately define the environmental conditions which the bond must withstand and select an adhesive capable of operating under those conditions. Increasing ambient temperature or moisture content results in a reduction in fatigue performance. Surface preparation is critical and no matter how well a joint is designed, if the surface preparation is inadequate the joint will not survive. The presence of flaws or porosity in the critical zone of a joint (i.e. overlap ends) will severely compromise the static and fatigue performance of a bonded joint. A large central debond may result in no significant change in stresses at the ends of the overlap, whereas a small defect at the edges could prove catastrophic.

5. Surface Preparation of Adherends

Correct surface preparation is essential for maintaining long-term structural integrity of bonded joints. The role of surface preparation is to remove surface contaminants (grease and dust), increase surface area for bonding, promote micro-mechanical interlocking, and/or chemically modify a surface. It is important that the process of surface preparation only effects the chemistry and morphology of thin surface layer of the adherend(s) and does not alter the mechanical and physical properties of the underlying substrate. This section provides a brief description of general procedures required for preparing different substrates for adhesive bonding. Most of the procedures have been extracted from “Guide to The Structural Use of Adhesives” produced by The Institution of Structural Engineers [8]. Specific treatments can be found in BS 7079, BS EN 12 768, ASTM D 2651, ASTM D 2093 and BS EN 1840 [54-58]. Advice should be sought on surface preparation from the adhesive manufacturer.

Note: After completion of the surface preparation process, the adherends must not be exposed to physical handling or atmospheric environments in order to prevent surface contamination prior to bonding. Bonding should be performed immediately following surface treatment to maximise performance. It is advisable to use solvent cleaners before and after mechanical abrasion. Clean grit, clean solvent and clean cloths must be used to avoid spreading contamination.

Surface preparation procedures often require potentially hazardous or environmentally damaging chemicals. All preparation activities should be carried out to COSHH specifications.

5.1 Steel

The recommended method for preparing steel substrates for bonding is as follows:

- Degrease with a suitable solvent (e.g. acetone or methyl ethyl ketone (MEK)).
- Abrade the surface to remove mill-scale and metal oxides. Abrasion of the substrate surface can be achieved using either a wire brush, an abrasive disc or by grit-blasting. A very high pressure water jet system can also be used.
- Remove dust/debris by brushing, by oil-free air blast or by vacuum cleaner.
- Dry the surface.
- Apply a suitable primer, if required by the adhesive manufacturer.

5.2 Zinc Coated Steel

Galvanised steel surfaces are less prone to rusting in most environments than untreated steels and when treated with a suitable surface preparation the adhesive-zinc interface is stronger than the steel-

zinc interface. This section provides details on the method to be used for preparing galvanised steel substrates for bonding:

- Degrease to remove oils and lubricants present on the substrate surface.
- Lightly abrade the surface ensuring the protective zinc layer is not penetrated/compromised.
- Remove dust/debris by oil-free air blast or by vacuum cleaner.
- Chemically etch.
- Dry the surface.
- Apply a suitable primer if required by the adhesive manufacturer.

5.3 Stainless Steels

The recommended steps to be taken in preparation of stainless steel surfaces is as follows:

- Degrease to remove oils and lubricants present on the substrate surface.
- Grit blast the surface ensuring the protective zinc layer is not penetrated/compromised.
- Acid etch the surface.
- Remove the products of the etching process.
- Apply a suitable primer (e.g. silane).

5.4 Aluminium Alloys

The recommended steps to be taken in preparation of aluminium alloy surfaces are:

- Degrease surface with a suitable solvent (e.g. acetone or MEK).
- Clean with a suitable alkaline solution.
- Acid etch, followed by neutralisation.
- Apply a suitable primer (e.g. silane) if required by the adhesive manufacturer.

It may be necessary to remove the surface layer of sealed anodised sheet in order to bond to the substrate. Alternatively, the surface may be grit blasted and then treated with a silane primer.

5.5 Titanium Alloys

The recommended steps to be taken in preparation of titanium alloy surfaces is as follows:

- Degrease surface with a suitable solvent (e.g. acetone or MEK).
- Grit blast.
- Remove dust/debris by brushing, by oil-free air blast or by vacuum cleaner.
- Acid etch the surface.

- Rinse and dry.
- Chemically stabilise oxide layer.

5.6 Fibre-Reinforced Polymer Composites

All fibre-reinforced plastic composites absorb small amounts of moisture from the atmosphere when exposed to ambient levels of humidity (i.e. standard laboratory conditions). For ambient temperature curing adhesive systems, the presence of moisture in the composite adherend may pose no particular problems, however it is advisable, to dry the adherends prior to bonding. It is essential, however, that the adherends are fully dried before bonding with high temperature curing adhesives. The heating process will draw moisture out of the adherend(s) and into the bondline, resulting in a weakened and porous adhesive layer. The glass-transition temperature (T_g) and fracture toughness can be significantly lowered as a result of moisture present in the adherend(s).

The main methods of surface preparation of fibre-reinforced plastic polymers are solvent degreasing, mechanical abrasion and peel-ply, which are often used in combination. The suggested procedure for the preparation of PMCs is as follows:

- Remove grease, dust and other surface contaminants.
- Remove release agents, resin rich surface layers, tissues and random fibre materials using an abrasive cloth or lightly grit blast ensuring only the top layer of resin is removed without causing damage to the fibres beneath.
- Remove any traces of solvents or dust.

Aluminium oxide (alumina) grit of grade 120/150 at a pressure of approximately 55-58 psi should produce a suitable surface. The grit blast and degrease process should be performed immediately prior to bonding to ensure good, clean surfaces. Specimens should be first masked off so that only those areas to be bonded are exposed.

Peel-ply is a sacrificial layer of fabric (e.g. woven glass, nylon or polyester) which is incorporated on the outermost surfaces of fibre-reinforced polymer composites and co-cured with the laminate. This layer is removed prior to bonding. When the dry peel-ply is removed, the top layer of resin on the laminated component is fractured and removed leaving a clean, rough, surface for bonding. The fibre reinforcement should remain unexposed. Care should be taken to ensure the peel-ply material is stored in dry conditions and is not used at temperatures that could result in degradation during process, thus leaving contaminants on the bonding surface. Moisture released from peel-ply materials during the cure process can adversely effect the physical and mechanical properties of the outer layer of the cured laminate.

5.7 General Comments

Silicon carbide grit-coated paper or other abrasive pads can be used dry or in the presence of a solvent can be used to mechanically abrade the adherend surfaces. It is essential that sufficient time is allowed to ensure the solvent evaporates. Surfaces should be cleaned following abrasion to remove any abrasive material left on the surface.

6. Thermal Analysis Techniques

Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), can provide useful information relating to the composition and degradation of adhesives. This section briefly examines the suitability of using thermal analysis techniques for characterising environmental degradation of adhesives.

6.1 Differential Scanning Calorimetry (DSC)

DSC is used to determine the heat flow associated with material transitions as a function of time and temperature or changes in heat capacity using minimal amounts of material. The technique provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions caused by phase changes, melting, oxidation and environmental degradation. The technique involves slowly heating a small sample of material and measuring the heat absorbed or emitted by the sample as a function of temperature compared to a reference material. DSC can be used to measure the lowering of T_g with increasing moisture content for neat resins (including adhesives) and PMCs.

The technique has many advantages, including:

- Fast analysis time (30 minutes)
- Easy sample preparation
- Small test specimens
- Wide range of temperature applicability

ISO 11357 specifies methods for the thermal analysis of polymeric materials, including composite materials using DSC.

6.2 Dynamic Mechanical Analysis (DMA)

This technique enables the determination of transition temperatures, storage modulus and loss modulus of the sample over a wide range of temperatures (-150 °C to 800 °C), frequencies (0.01 to 200 Hz) or time, by free vibration, and resonant or non-resonant forced vibration techniques (ISO 6721). Flexural samples are popular, whereby the sample is mounted on a clamp and then subjected to sinusoidal changes in strain (or stress) while undergoing a change in temperature. DMA is suitable for adhesives and laminated composites with stiffnesses ranging from 1 kPa to 1,000 GPa. Tension, compression and flexure loading configurations can be employed. Three-point flexure specimens can be up to 50 mm in length, 15 mm wide and 7 mm thick.

The technique has many advantages, including:

- Fast analysis time (typically 30 minutes)
- Easy sample preparation
- Wide range of temperature applicability

6.3 Thermal Mechanical Analysis (TMA)

This technique is used in conjunction with DSC to study thermal transition behaviour (e.g. T_g) of polymeric materials (e.g. adhesives and PMCs). TMA can be used to measure heat distortion temperature, and thermal expansion and contraction (i.e. coefficient of thermal expansion) under dynamic or isothermal heating conditions. Thermal transition behaviour can be used as a quality assurance technique for determining the extent of resin cure.

6.4 Thermal Gravimetric Analysis (TGA)

TGA can be used to monitor weight changes in a sample as a function of temperature. The technique is primarily used for studying degradation processes, providing information on thermal oxidative degradation rates and thermal degradation temperatures of polymeric materials (e.g. adhesives and PMCs). This information is useful for determining the life expectancy of polymers at elevated temperatures and for comparing degradation rates of different adhesive systems.

7. Surface Analytical Techniques

Surface and chemical characterisation techniques can provide basic and quantitative information relating to the process of degradation and failure of bonded joints. These techniques can provide important information on:

- Failure modes and mechanisms;
- Chemical composition and morphology (e.g. surface roughness) of surface layers;
- Effects of surface preparation on surface chemistry;
- Stability of surfaces and interfaces;
- Surface contaminants; and
- Chemical and physical degradation of both the adhesive and oxide layers.

Chemical characterisation can be achieved using either spectroscopic or chromatographic techniques. Spectroscopic analysis provides detailed information about molecular structure, conformation, and physical-chemical characteristics of polymers, and chromatographic techniques enable quantitative compositional characterisation. This section provides a brief summary of surface and chemical analysis techniques that can be used to analyse and evaluate chemical, physical and mechanical changes due to the combined effect of mechanical loading and environmental exposure.

7.1 Scanning Electron Microscopy (SEM)

High resolution SEM has proved an invaluable tool for studying surface topography, oxide growth and failure analysis. The technique enables three dimensional imaging of surface features, however, the magnification is insufficient to allow surface roughness characterisation. Atomic force microscopy (AFM), complemented by SEM data, can provide quantitative information on surface roughness. Although, data interpretation can be difficult.

7.2 Electron Dispersive X-Ray Analysis (EDX)

Detection and analysis of characteristic X-ray lines of various elements can be obtained using an EDX system attached to an SEM. The maximum operational depth of EDX is typically 3 to 10 μm . The actual penetration depth depends on the type of material being analysed and the acceleration potential used in the SEM. The technique can be used generate elemental distribution maps of the area of interest, enabling both qualitative (boron to uranium) and quantitative (sodium to uranium) elemental analysis. The technique cannot provide information on chemical bonds.

7.3 Infrared Spectroscopy (IRS)

IRS provides both qualitative and quantitative chemical analysis data and can be used to observe environmental effects on polymer chemistry. The technique provides a fingerprint of the adhesive composition and can be used to analyse gases, liquids and solids. Computerised databases of spectra for common polymeric materials are available to enable characterisation of molecular structure by observing spectral differences between known materials and the test sample. Fourier transform infrared spectroscopy (FTIR) can be used to assess the state of cure of thermoset resins.

7.4 X-ray Photoelectron Spectroscopy (XPS)

XPS is frequently used for quantitative elemental analysis of fracture surfaces and for monitoring chemical changes in adhesive samples. The technique, which is capable of detecting all elements with the exception of hydrogen, can provide information of chemical groups present on the surface of polymeric materials. The technique can be used to determine the effect of elevated temperature and surface preparation on surface chemistry and is used to examine the cause of adhesion problems. The maximum operational depth of XPS is 3 nanometres.

7.5 Auger Electron Spectroscopy (AES)

AES is a non-destructive technique for identifying the elements in the first few atomic layers (~2 nanometres) on a specimen surface and is able to provide quantitative data on the detected elements. Combined with inert gas ion sputtering, AES can be used to obtain depth composition profiles. The technique can be used to map the distribution of elements present on a specimen surface. The technique is capable of detecting all elements with the exception of hydrogen. Spectrometers can be fitted with a facility for in-situ testing of bonded joints and other specimen configurations, which are rapidly analysed under high vacuum conditions. Ultra-high vacuum conditions (1×10^{-10} Pa) are required to prevent contamination and oxidation of the fracture surfaces. The technique is not particularly suited to examination of polymers (i.e. insulating materials) due to the possibility of beam damage and electrical charging of the sample, which can complicate data interpretation. The technique provides limited information on oxidation states.

7.6 Chromatographic Analysis

Gas chromatography (GC) is useful for identifying volatile reaction products during cure. High performance liquid chromatography (HPLC) is particularly useful in determining the average molecular weights and molecular weight distributions of polymeric materials; particularly suited to

analysing thermoplastic resins. This equipment is generally supplied with automated instrumentation which is relatively simple to operate and maintain.

Liquid chromatography technique separates molecules according to their size in solution and employs various detectors to monitor concentrations and identify chemical components. The technique requires calibration with standard polymers.

8. Non-Destructive Evaluation Techniques

A number of techniques are available for the non-destructive inspection of adhesive joints:

- Ultrasonic (C-scan)
- X-ray Radiography
- Thermography
- Acoustic Emission

There is no NDE technique which can provide a quantitative assessment of joint strength.

8.1 Ultrasonic (C-Scan) Inspection

Ultrasonic C-scan technique is particularly suited to the detection of planar type defects (e.g. debonds and delaminations) normal to the incident beam. Voids and porosity in the adhesive and adherends are also detectable. The minimum size of voids and delaminations detectable with C-scan is approximately 2 mm. The technique is not suitable for detecting surface contaminants (e.g. oils and grease).

8.2 X-ray Radiography

This technique is suitable for detecting the presence of voids and solid inclusions (e.g. backing film) in the bondline. Thin debonds and delaminations are difficult to detect because the presence of these defects has minimal effect on the absorption characteristics of polymeric materials. The use of penetrant fluids can enhance the imaging process, however, these fluids can adversely effect the short-term properties and fatigue performance of polymeric materials. Penetrants should not be used in those tests where the test data is to be used for design or quality assurance purposes. Small tensile loads or the use of a vacuum pump can be used to promote fluid penetration.

8.3 Thermography

This non-contact technique can be used for rapid inspection of large bonded structures capable of detection and discrimination of gross defects and discontinuities close to the surface. The technique requires the inspected component to be heated to produce a surface temperature distribution which can be correlated with structural integrity or defect distribution. Heating of the bonded structure can be achieved either by:

- thermally soaking the entire structure (known as soak) to a constant temperature and then measuring the gradual dissipation of heat; or by
- a thermal spike where the uptake and spread of thermal energy is measured.

Spatial and temporal temperature distribution is measured using infrared imaging CCTV cameras.

8.4 Acoustic Emission (AE)

Although, acoustic emission (AE) is not strictly an NDE technique, it can be used to monitor bonded joints for delaminations and debonds during mechanical testing. The technique relies on the operator having sufficient experience to be able to identify particular defect types from the AE data. The technique is essentially a laboratory tool, however, AE is used for proof and qualification tests of pressure vessels prior to installation and routine in-service inspection.

9. Useful Contacts

NPL

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SATRA

SATRA Footwear Technology Centre
SATRA House
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Kettering, Northants, UK, NN16 9JH
Tel: 01536 410000

ASTM

American Society for Testing and Materials
100 Barr Harbor Drive
West Conshohocken
Pennsylvania 19428
USA
Tel: 001 610 832 9500

ISO

International Standards Organisation
1 Rue de Varembe
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Tel: +41 22 74901 11

DERA

Defence Evaluation and Research Agency
Structural Materials Centre
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IoM

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TWI

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PIRA

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BSI

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MERL

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ISE

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Appendix I: Test Methods for Determining Input Design/Analysis Data

Material Property	Standard/Test Method
Elastic Properties - Adherends <u>Metals</u> E, G, ν <u>Composites</u> In-plane (E_{XX} , E_{YY} , ν_{XY}) Through-thickness (E_{ZZ} , ν_{XZ} , ν_{YZ}) In-plane shear (G_{XY}) Through-thickness shear (G_{XZ} , G_{YZ})	Tensile test of plastics - BS EN ISO 527-2 m.d = multidirectional, u.d = unidirectional Tension - BS EN ISO 527-4 (m.d)/BS EN ISO 527-5 (u.d) T-T tension and compression-NPL draft procedures $\pm 45^\circ$ tension method - BS EN ISO 14129 (u.d)* V-notched beam test - ASTM D 5379
Strength Properties - Adherends <u>Metals</u> Tension Compression Shear <u>Composites</u> In-plane tension (S_{XX} , S_{YY}) Through-thickness tension (S_{ZZ}) In-plane compression (S_{XX} , S_{YY}) Through-thickness compression (S_{ZZ}) In-plane shear (S_{XY}) Through-thickness (S_{XZ})	Tensile testing of metallic materials - BS EN 10002-1 Compression testing of metallic materials - ASTM E9 Shear modulus - BS EN 10002-1* m.d = multidirectional, u.d = unidirectional Tensile - BS EN ISO 527-4 (m.d)/BS EN ISO 527-5 (u.d) Through-thickness tension - NPL draft procedure Compression - BS EN ISO 14126 Through-thickness compression - NPL draft $\pm 45^\circ$ tension method - BS EN ISO 14129 (u.d) V-notched beam method - ASTM D 5379 (u.d)
Elastic Properties - Adhesives E, G, ν	Tensile test of plastics - ISO 527-2
Strength Properties - Adhesives Tension Compression Shear Maximum principal strain	Tensile test of plastics - BS EN ISO 527-2 Compressive testing of rigid plastics - ISO 604/ ASTM D695 V-notched beam method - ASTM D 5379* Tensile test of plastics - BS EN ISO 527-2
Fracture Toughness Mode I Mode II	Double cantilever beam (DCB) test - ISO 15024/prEN 6033 End notched flexure (ENF) test - prEN 6034
Joint Coupon Tests Tension shear strength and modulus Shear strength and modulus Compression strength and modulus	Butt joint Thick adherend shear test (tension) - ISO 11003-2 Butt joint
Additional Tests Tensile strength of lap joint Tension-tension fatigue Moisture absorption/conditioning Effect of water/moisture Effect of chemicals Effect of heat ageing Test and conditioning atmospheres Tensile creep behaviour of plastics Failure patterns Dynamic Mechanical Analysis Differential Scanning Calorimetry	BS EN 1465 BS EN ISO 9664 BS EN ISO 62 ISO 62/ISO 175 ISO 175 ISO 216 ISO 291 ISO 899-1 EN 923 ISO 6721-4 ISO 11357

Symbols: E = modulus of elasticity, G - shear modulus, ν = Poisson's ratio, S = strength

Subscripts: XX, YY and XY denote in-plane properties, XZ, YZ and ZZ denote through-thickness properties

* Plate twist method - ISO 15310 (simple test for measuring shear modulus only)