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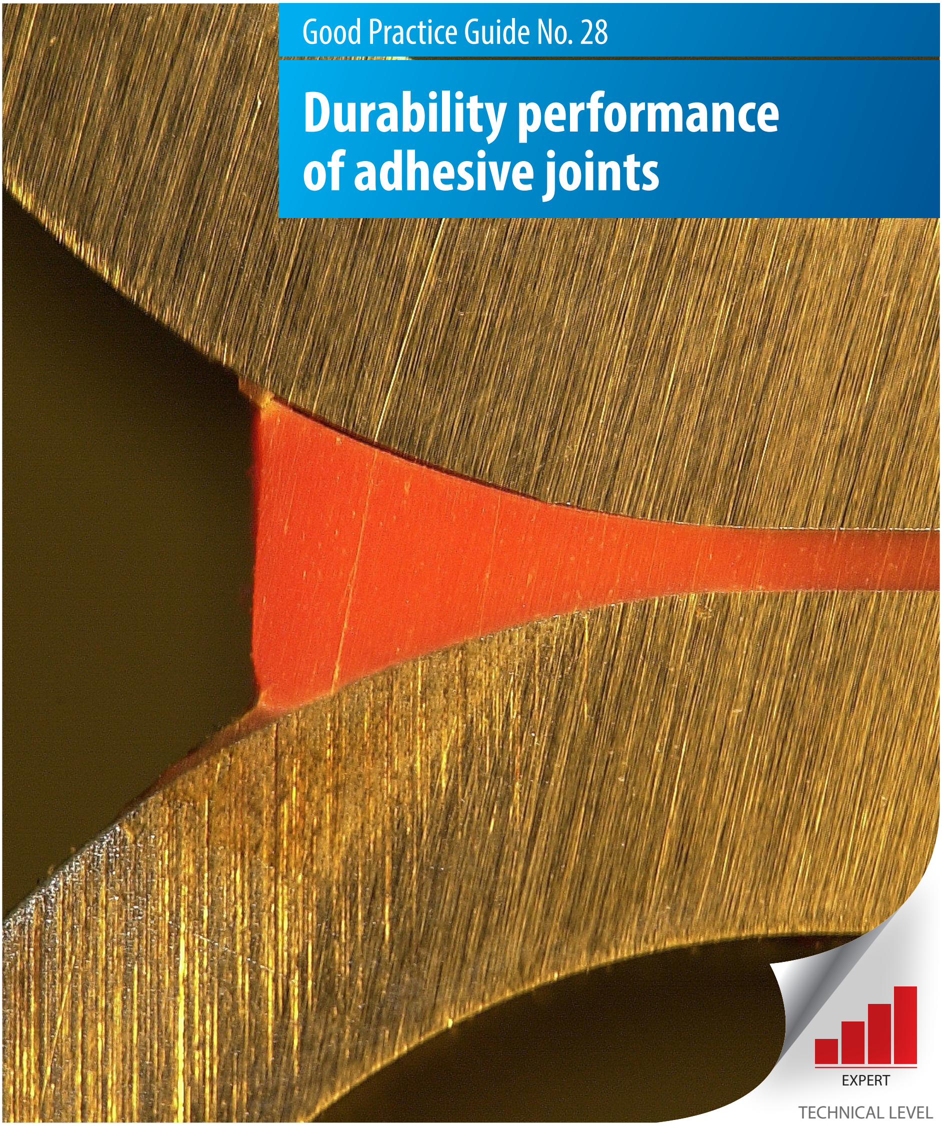

Good Practice Guide No. 28

Durability performance of adhesive joints

EXPERT

TECHNICAL LEVEL

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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.

Acknowledgements

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Contents

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 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.

Bond-line: 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.

Cure: To set or harden by means of a chemical reaction.

Cure time: Time required to affect a cure at a given temperature.

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

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

Elastomer: A rubbery material that returns to approximately its original dimensions in a short time after undergoing a large amount of deformation.

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

Exothermic: A chemical reaction that emits heat.

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 bond-line.

Fracture toughness: The resistance of a material (or interface) to crack propagation. Defined for different modes of fracture. Mode I is direct tension, Mode II is in-plane shear and Mode III is out-of-plane shear.

Gel: A semi-solid system consisting of a network of solid aggregates in which liquid is held.

Gelation: Formation of a gel.

Glass transition: A 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.

Hygroscopic: Material capable of absorbing and retaining environmental moisture.

ISO: International Standards Organisation.

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

Open time: Time interval from when an adhesive is applied to when the material becomes unworkable.

Peel: Mode of application of a force to a joint in which one or both 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.

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

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

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

Substrate: An adherend, 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.

Tack: The property of an adhesive that enables it to form a bond of measurable strength immediately after adhesive and adherend are brought into contact under low pressure.

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 bond-line.

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

Thermoplastic: A material that can be repeatedly softened by heating.

Traveller: A test specimen used for example to measure moisture content because of environmental conditioning.

Viscosity: Resistance of a liquid material to flow.

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

Wetting: A surface is considered completely wet by a liquid if the contact angle is zero, and incompletely wet if the contact angle has a finite value.

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

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

Executive summary

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 polymer matrix 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 basic knowledge of the materials and techniques involved and is not intended as a textbook. There are several 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 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 both initial adhesion preparation 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 manufacturer is obtained, and 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 (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.

There are several published works, which provide a comprehensive coverage of adhesive technology and preliminary design [1-9]. Other NPL Measurement Good Practice Guides “Preparation and Testing of Bulk Specimens of Adhesives”, “Durability Performance of Adhesive Joints” and “Characterisation of Flexible Adhesives for Design” [10-12] provide advice on issues relating to the preparation and testing of bulk adhesive and adhesive joint specimens,

acquisition of design data from bulk specimens, finite element modelling of adhesives, flexible adhesives, and durability testing.

The intention of the guide is to complement these published works. It is recommended that specialist advice be sought from adhesive manufacturers on adhesive selection, use of associated technologies and health and safety requirements.

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Chapter 1

Scope

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. Several of these methods 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 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 bond-line, often referred to as in-*situ* testing. 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 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 (QA) 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 1 shows a list of recommended test methods (see Table A1.1) for determining input data for the design and analysis of bonded joints.

The guide also includes generalised surface preparation techniques for metal and fibre-reinforced polymer matrix composites (PMCs) substrates and a summary of useful surface analytical and thermal analysis techniques. These techniques 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.

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Chapter 2

Measurement of bulk adhesive properties

- Introduction
- Specimen preparation
- Test methods and standards
- Moisture conditioning and testing

Introduction

Bulk adhesive testing is 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 chapter 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.

Specimen preparation

Tests on bulk adhesive specimens can provide reliable elastic and strength property data for design purposes. However, there are several key points that should be considered when producing bulk adhesive specimens [l0-16]:

- 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 ± 2 °C, 50 ± 10 relative humidity (RH)), which can reduce the glass-transition temperature T_g and mechanical propertiese of the adhesive.

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 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 thermal analysis (DMTA) measurements can be used to compare the final state of cure of the materials (see Chapter 6) [17].

Recommended procedures for the preparation of bulk specimens of adhesives are given in ISO 15166 Parts 1 and 2 [13-14]. Part 1 of the standard considers two-part (component) adhesives cured at ambient or elevated temperatures, whilst Part 2 covers single component systems requiring an elevated curing temperature (see also [10-11]). Test specimens may either be moulded or sectioned from manufactured resin plaques. It is important that the specimen is free of surface damage (i.e., scratches and nicks). To minimise the deleterious effects of surface scratches, which may cause premature failure, the edges and faces of the specimens should be carefully polished to remove any surface defects.

Handling adhesives can be hazardous to human health, thus COSHH (Control of Substances Hazardous to Health Regulations) procedures should be followed to minimise operator exposure. A safety data sheet should accompany all adhesive samples (Health and Safety at Work Act 1974). The user prior to handling the material must read this document. The safety data sheet supplied with the adhesive identifies:

- Hazardous ingredients and concentrations - includes regulatory information according to legislative directives.
- Hazard statements, such as H225 (highly flammable liquid and vapour), H315 (causes serious eye irritation) and H319 (causes serious eye irritation), and precautionary statements, such as P260 (do not breathe dust/fume/gas/mist/vapours/spray) and P280 (wear protective gloves/protective clothing/eye protection/face protection). Hazard statements are accompanied by classification (e.g., Flammable liquid. 2 – H225 and Eye irritation. 2 – H315).
- First aid measures (including inhalation, ingestion, skin contact and eye contact), and important symptoms and effects, both acute and delayed (e.g., prolonged skin contact may cause redness and irritation). The severity of the symptoms will vary dependent on the concentration and the length of exposure.
- Fire extinguishing/fighting (suitable and unsuitable extinguishing media, and protective equipment and actions) and accidental release (personal and environmental precautions, and methods for cleaning up spillages or discharges) measures.

- Precautions for safe handling and storage including storage precautions (e.g., keep away from heat, sparks, and open flame) and storage class (e.g., flammable liquid storage).
- Control parameters and occupational exposure limits (short-term and long-term exposure limits).
- Exposure controls (includes engineering measures (e.g., adequate ventilation), respiratory protection, eye, skin and body protection, hygiene measures, respiratory protection, and environmental exposure controls).
- Physical and chemical properties (i.e., colour, odour, melting and boiling points, vapour pressure, flammability, etc.), and stability and reactivity of ingredients, and storage and transportation conditions (e.g., temperature).
- Toxicological information (refer to COSHH) – includes potential health effects and hazardous toxicity data.
- Ecological information, disposal considerations and transport information.

Test methods and standards

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.

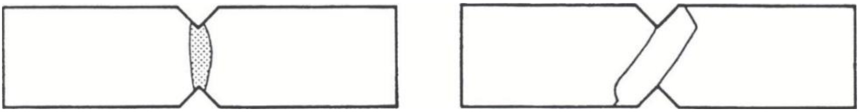
Longitudinal and transverse strain can be measured using either strain gauges, contacting extensometers or video extensometers (non-contact technique) [10]. It is important when using contact techniques, such as strain gauges and extensometers, that the transducer(s) are capable of functioning within the test environment and have suitable response time. Strain gauges are not recommended for adhesive characterisation due to: (i) stiffening effects on lowstiffness materials; (ii) limited range of operation; and (iii) tendency to act as failure initiation sites in 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 is advisable to regularly coat threaded surfaces with a protective grease.

Shear

The V-notched beam method (ASTM D5379 [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 ±45° 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 using 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.

Figure 1. V-notched beam shear fixture and specimen



Thermoplastic – Shear Yielding **Thermoset – Brittle Tensile**

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

Shear strain is measured with biaxial strain gauges (1-2 mm gauge-length) bonded at ±45° 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 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].

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 D695 [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.

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 FRPs) absorb 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 several 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 conditioningof bulk adhesive and testing of adhesive specimens that have been exposed to either hot/humid environments or immersed in water at elevated temperatures.

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; 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 thin specimens to determine the "through-the-thickness" moisture diffusion coefficient.

The International standard 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. 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 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, 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 (sealed container with desiccant) 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 to ensure accurate moisture absorption measurements. To minimise moisture uptake prior to conditioning, specimens are stored in a desiccator at room temperature. It should be noted that under standard laboratory conditions many adhesives absorb significant levels of moisture.

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 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 humidity's 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 rapid in the initial 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 initial 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 conducted at approximately equal intervals of $(\text{time})^{1/2}$. The derived moisture content M can be determined as follows:

$$M = \frac{\left(W_{wet} - W_{dry}\right)}{W_{dry}} \times 100\% \tag{1}$$

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 throughout the conditioning process (includes temperature, relative humidity, 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 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]:

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

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.

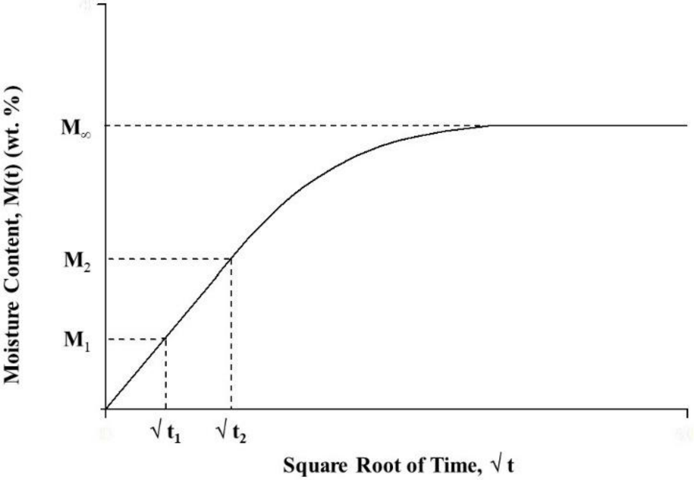


Figure 3. Fickian diffusion curve

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 shorter time to reach than time required to reach full equilibrium. The time required to obtain the last 5% can be longer than the time taken to reach the 95% level. Clearly a 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 ±5%. It is essential that boiler units of humidity cabinets be supplied from a deionised/distilled water reservoir to prevent salt deposits on test specimens, corrosion of bonded joints and scaling of the plumbing.

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 the previous section. 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 1: It is assumed that all absorbed moisture is translated into a change in resin volume. fu 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.

Mechanical testing of 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 represented by a relative humidity of 85% RH and a temperature of 70 °C.

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.

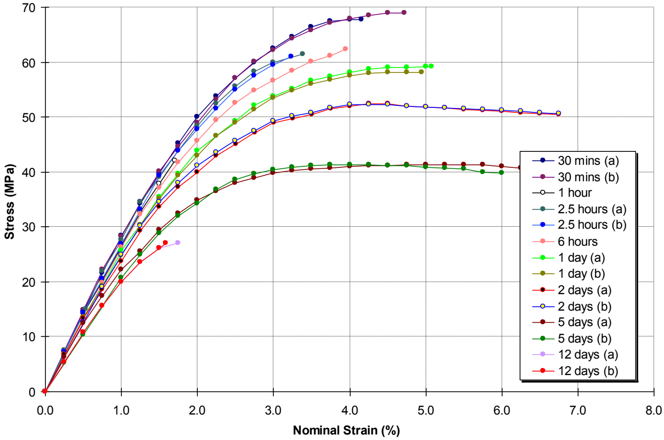


Figure 4. Tensile stress-strain curves for different water immersion periods

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 wi1ing 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, conditioned 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 polythenebag containing a salt solution appropriate to the humidity requirements of the test, are not practical. Traveler specimens 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.

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Chapter 3

Measurement of adherend properties

- Tension
- Compression
- Shear
- Through-thickness (T-T) properties
- Scope

Introduction

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

Tension

ISO 6892-1 [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 ISO 527-4 [31] for multidirectional composite laminates and 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.

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-load 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-tabbed (to prevent failure at the specimen ends). No problem is expected for the use of the test configurations for creep and environmental testing of metals or composites.

Shear

There is no standard at present for determining shear strength of metals. Shear modulus for these materials can be determined from tensile tests. 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 ±45° laminate, 250 mm long, 25 mm wide and 2 mm thick. Specimen preparation and testing are similar to those specified in ISO 527-4 [31]. 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 minimizes 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.

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 Chapter 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.

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Chapter 4

Adhesive Joint Testing

- Introduction
- Tensile properties
- Shear properties
- Quality assurance testing
- General issues relating to environmental testing
- Creep
- Cyclic fatigue
- Basic design considerations

Introduction

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

- The stress distribution within the bond-line tends to be non-uniform in a majority of test joint configurations with stress concentrations existing at the bond-line 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 bond-line 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.

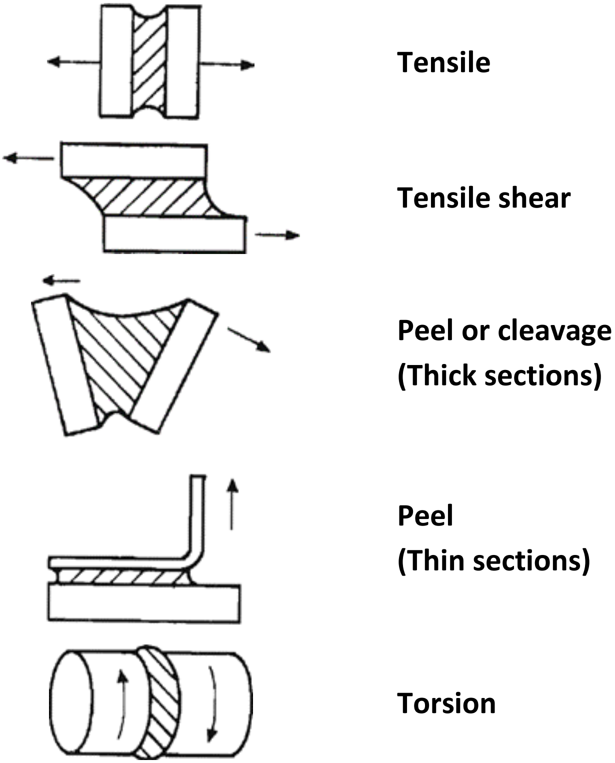

Figure 5. Basic loading modes experienced by adhesive joints

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.

Tensile properties

The cylindrical butt joint (Figure 6) can be used to test thin bond-line 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 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 D897 [38], ASTM D2095 [39], and BS EN 15870 [40].

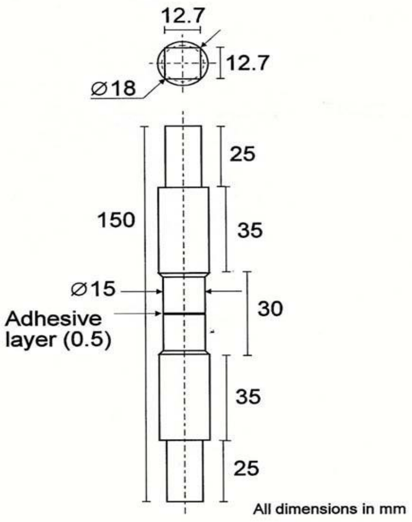

Figure 6. Tensile butt joint

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.**

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 (thick adherend shear test (TAST)). The TAST specimen can be produced by either:

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

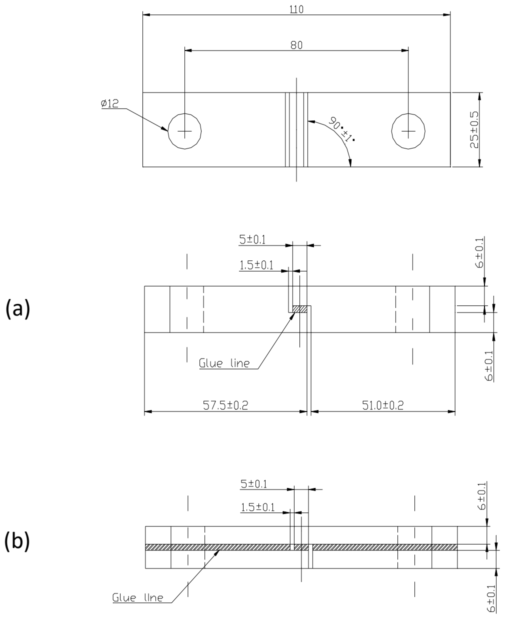

Figure 7. TAST specimen: (a) pre-shaped adherends; (b) bonded sheets (dimensions in mm)

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.

ISO 11003-2 [41] specifies a specimen with an overall length of 110 mm, a width of25 mm and overlap length of 5 mm. The ISO standard recommends an adherend thickness of 6 mm and a bond-line 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.

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].

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 [42-45]. End tabs, cut from the same material as the adherend sections, are adhesively bonded to the specimen. The end tabs, which are typically 37.5 mm in length, 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.

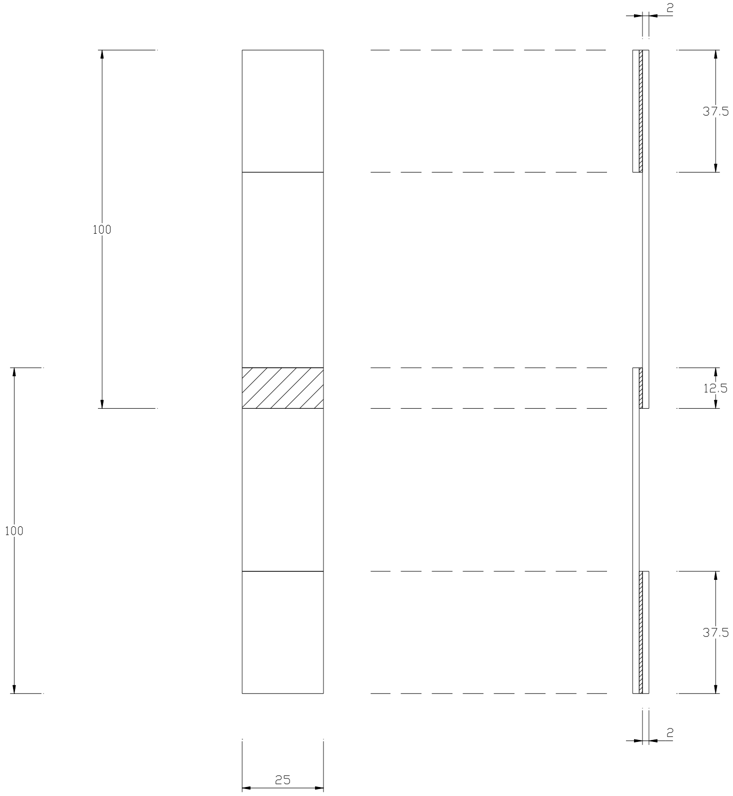

Figure 8. Schematic of single-lap joint (dimensions in mm)

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]:

$$L = \frac{\sigma_Y t}{1.5\,\tau} \tag{3}$$

where σ is the yield stress of the adherend, τ = average shear strength of the adhesive and t is the 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 fibre-reinforced plastics (FRPs). 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].

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 Ro 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.

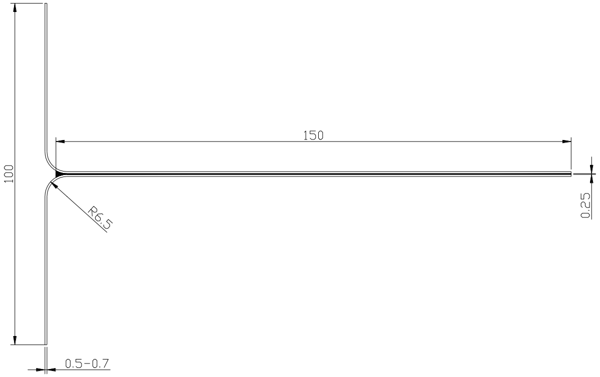

Figure 9. Schematic T-peel joint (dimensions in mm)

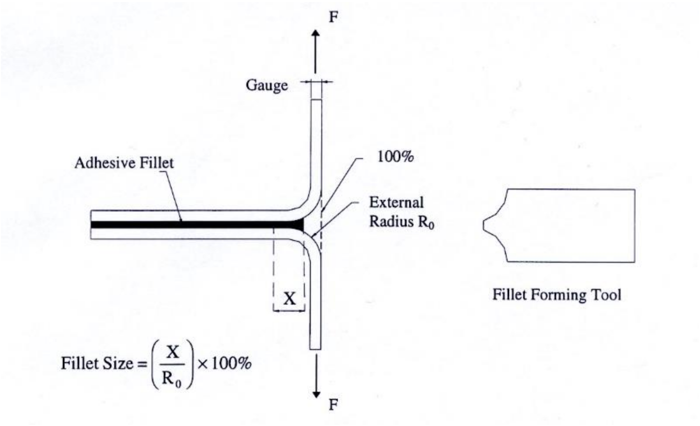

Figure 10. Definition of fillet size in T-peel joint [51] – including fillet forming tool

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 Chapter 5, whilst Chapter 7 provides a brief summary of surface and chemical characterisation techniques. The standard 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 (Chapter 2). 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 ISO 62 [26]). For non-equilibrium moisture levels, the moisture distribution is non-uniformly distributed; as shown in Figure 11.

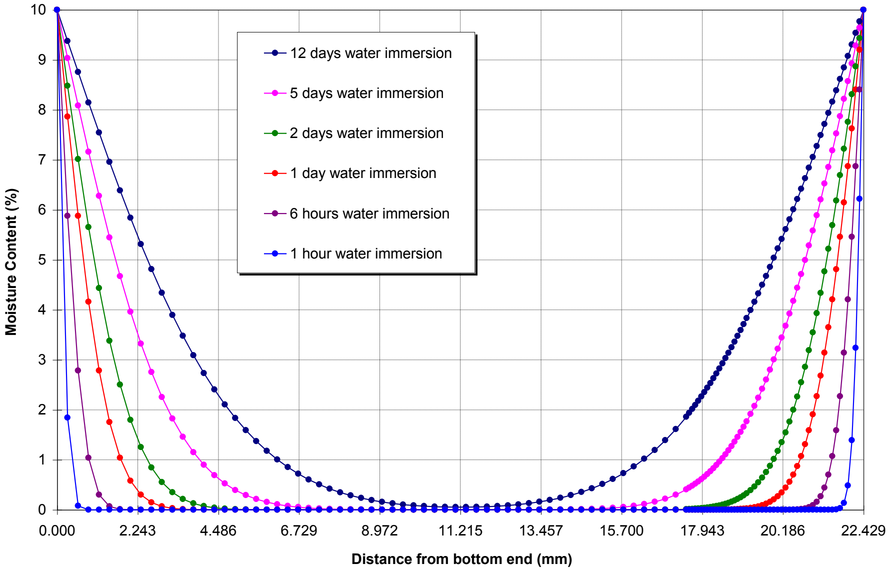

Figure 11. Typical moisture concentration distributions along an adhesive layer

Creep

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. Specimens are removed at regular intervals to assess strength reduction.
- **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:

1. Servo-hydraulic test machines.
2. Dead-weight and lever creep testing machines.
3. A screw jack in series with a load cell (see Figure 12); and
4. Self-stressing fixture where specimens are placed in either a tube equipped with a calibrated spring system (see Figure 12) for loading specimens or a circular ring.

Figure 12. Screw jack machines (left) and a self-stress tube (right)

The use of a servo-hydraulic test machine is not an economic option in most cases. 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 12), 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 (ISO 14615 [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 shown in Figure 12 can load 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 lifetime 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 to accurately determine time-to-failure. Specimens loaded by springs can often be in an unstressed state for a considerable period (overnight or weekends) before the failed joint is replaced (by a “dummy” specimen) and the loading train is 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 system evaluated. Typically, these are between 10 and 50% of the short-term strength of the joint. It is recommended that the sustained stress in an adhesive bonded joint under service conditions should be kept below 25% of the short-term strength of the joint. The onset of failure tends to be catastrophic. In general, damage and stiffness loss prior to crack initiation is minimal. The time involved in crack formation tends to be far greater than the time associated with crack propagation. Methods for accelerating the testing process that use mechanical loading tend to use stress levels that are significantly higher than stress levels used in design, thus the limiting design strains are reached in shorter times than in actual service.

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) specimens per stress level with five stress levels per condition should provide a reasonable number of data points. 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).

Cyclic fatigue

The fatigue properties of a bonded joint are a function of the joint geometry and adhesives, and therefore cannot be determined from the 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). Fatigue data are normally obtained at the highest frequency possible to minimise the duration of tests. The uncertainty in life expectancy at any stress level is typically an order of magnitude.

Restrictions on test frequency can arise from test equipment limitations (response time), time dependent processes and hysteretic (self-generated) 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. The upper frequency limit will be dependent upon the thermal conductivity of the adherend/adhesive system, mode of loading and specimen size. Trials may be necessary to determine the upper frequency limit.

It is recommended that the temperature rise of the material surface be kept to a minimum. 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, although the latter is beyond the budget of most industrial facilities. The temperature resolution is ~1 °C for the two methods.

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 13), thus improving creep and fatigue performance. It also provides added protection from environmental attack. Increasing the bond-line 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.

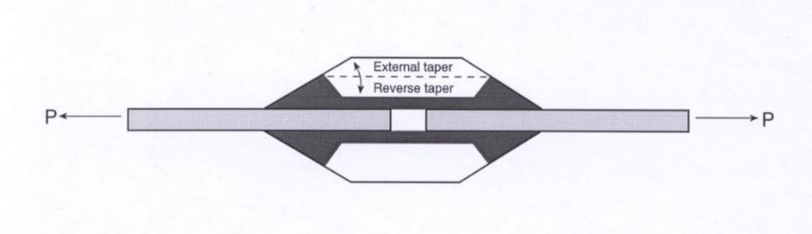

Figure 13. Bevelled strap joint

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 14) for adhesive joints can be approximated (rule of thumb) by a straight line fit as follows:

$$\frac{P_{MAX}}{P_0} = 1 - klogNf \tag{4}$$

where k is the slope, N_f is the number of cycles to failure, P_{MAX} is the maximum load applied to the specimen, P_0 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 (Figure 15) has a far better fatigue performance than tests where peel stresses are the major cause of failure.

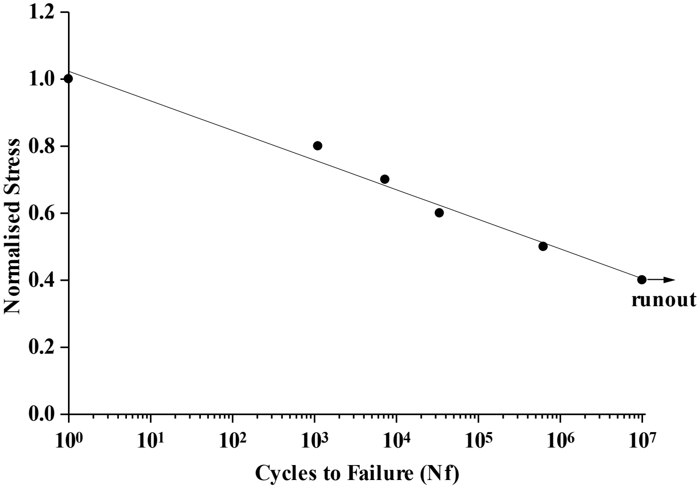

Figure 14. Normalised S-N curve for a tapered-strap joint

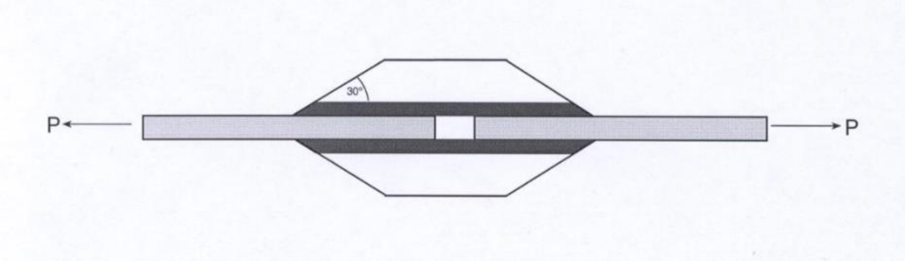

Figure 15. Tapered-strap joint

Joint Configuration	*k value*
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)	0.013

Table 1. Typical k values for bonded joints tested at room temperature

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.

Chapter 5

Surface preparation of adherends

- Introduction
- Steel
- Zinc coated steel
- Stainless steels
- Aluminium alloys
- Titanium alloys
- Fibre-reinforced polymer composites
- General comments

Introduction

Surface preparation is recognised as the most critical step in the adhesive bonding process and considerable adhesive joint testing is performed to optimise surface treatment. The selection of surface treatment is largely dependent on the required strength and durability of the joint, although economic considerations, such as costs and time involved in preparation, also play a role in the selection process. Correct surface preparation is essential for good joint strength and maintaining long-term structural integrity of bonded joints. Unsatisfactory surface preparation will result in the bond failing adhesively and unpredictably at the adhesive/adherend interface.

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 affects 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 chapter 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 ISO 17212, ASTM D2093, ASTM D2651, BS 7079 and BS EN 1840 [54-58]. Advice should be sought on surface preparation from the adhesive manufacturer.

Note 1: After completion of the surface preparation process, the adherends must not be exposed to physical handling or uncontrolled atmospheric environments to prevent surface contamination prior to bonding. It is advisable that bonding be performed immediately following surface treatment to maximise performance. 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 should be carried out to COSHH specifications.

Steel

The recommended method for preparing steel substrates for bonding is as follows [1]:

- 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.

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 [1]:

- 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 and then dry the surface.
- Apply a suitable primer, if required by the adhesive manufacturer.

Stainless steels

The recommended steps to be taken in preparation of stainless-steel surfaces is as follows [1]:

- 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).

Aluminium alloys

The recommended steps to be taken in preparation of aluminium alloy surfaces are [1]:

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

Titanium alloys

The recommended steps to be taken in preparation of aluminium alloy surfaces are [1]:

- Degrease with a suitable solvent (e.g., acetone or methyl ethyl ketone (MEK)).
- Grit blast - remove dust/debris by oil-free air blast or by vacuum cleaner.
- Acid etchthe surface.
- Rinse and dry.
- Chemically stabilise the oxide layer.

Fibre-reinforced polymer matrix composites

All fibre-reinforced polymer 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 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 bond-line, resulting in a weakened and porous adhesive layer. T_g and fracture toughness can be significantly lowered because 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 [1]:

- 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 affect the physical and mechanical properties of the outer layer of the cured laminate.

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.

Chapter 6

Thermal Analysis Techniques

- Introduction
- Differential scanning calorimetry (DSC)
- Dynamic Mechanical Analysis (DMA)
- Thermal Mechanical Analysis (TMA)
- Thermal gravimetric analysis (TGA)

Introduction

Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), can provide useful information relating to the composition and degradation (e.g., effects of moisture ingress on T_g) of adhesives. The analysis of material by any thermal analysis technique is complicated by changes in the material during testing. For adhesive materials, there will most likely be further curing or loss of moisture, when subjected to heating during thermal analysis. In some cases, these changes will prevent the accurate determination of the T_g. Repeat measurements need to be taken to check completeness of cure. It is important that the material assessed is representative of the adhesive within the bonded structure. This section briefly examines the suitability of using thermal analysis techniques for characterising environmental degradation of adhesives.

Differential scanning calorimetry (DSC)

DSC measures temperatures and heat flows associated with thermal transitions in a material (see ISO 11357-1 [59]). Operating temperature range is typically -180 °C to 700 °C, or higher. 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. DSC can be used to measure T_g and degree of cure, however it is not particularly suitable for determining the effect of moisture on T_g as the small samples tend to dry out during testing.

Modulated DSC offers improved resolution and sensitivity not possible using other techniques. The heat flow signal is composed of several parts (i.e., reversing, and non-reversing events). Conventional DSC can only measure and illustrate the sum of those parts in a single signal, whereas with MDSC multiple signals are generated in a single experiment so that individual components of the total heat flow signal can be displayed and analyzed independently. Typical reversing events are glass transitions and examples of non-reversing events are thermoset cure and decomposition.

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 has many advantages, including:

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

Dynamic mechanical analysis (DMA)

DMA (or dynamic mechanical thermal analysis (DMTA)) measures the stiffness and damping properties of materials as a function of time, temperature, and frequency by applying a sinusoidal load to a specimen and measuring the resultant deformation, whilst the sample is subjected to a controlled temperature programme. The magnitude of the applied stress and the resultant strain are used to calculate the stiffness (modulus) of the material under stress. The phase lag δ between stress and strain is used to determine tanδ, the damping factor. This technique enables the determination of mechanical (storage (elastic) and loss (damping) modulus) and thermal (e.g., T_g) properties of polymeric materials over a wide range of temperatures (-150 °C to 600 °C) and frequencies (0.01 to 200 Hz) [60, 61]. Modulus values obtained using DMA are not as accurate as conventional mechanical test techniques; measurements are sensitive to specimen dimensions and loading conditions (including clamping forces). DMA is suitable for polymeric materials with stiffness ranging from 1 kPa to 1,000 GPa.

The technique has many advantages, including:

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

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 measures changes in dimension (e.g., expansion or contraction), modulus or viscosity of a material as a function of temperature. The temperature may be increased or decreased linearly or stepwise, kept constant or modulated with a set frequency and amplitude. Operating temperature range is typically -150 °C to 1000 °C. TMA can be used to measure heat deflection and glass transition temperatures, thermal expansion coefficients, and characterise creep and stress relaxation behaviour at different stress and temperature conditions. Thermal transition behaviour can be used as a quality assurance technique for determining the extent of resin cure.

Thermal gravimetric analysis (TGA)

TGA measures weight changes in a material as a function of temperature (or time) under a controlled atmosphere. The sample weight is continuously monitored as the temperature is increased either at a constant rate or through a series of steps. The components of a polymer will decompose at different temperatures, thus producing step changes in weight to enable quantitative measurements of the different components. TGA instruments can operate up to a temperature of 1000 °C. It can provide valuable information on moisture and volatile content.

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Chapter 7

Surface analytical techniques

- Scanning electron microscopy (SEM)
- Electron dispersive x-ray analysis (EDX)
- Infrared spectroscopy (IRS)
- X-ray photoelectron spectroscopy (XPS)
- Auger electron spectroscopy (AES)
- Chromatographic analysis

Introduction

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 chapter provides a 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.

Scanning Electron Microscopy (SEM)

This is the most widely used of the surface analytical techniques. High resolution SEM has proved an invaluable tool for studying surface topography, oxide growth and failure analysis. The technique enables qualitative three-dimensional (3-D) imaging of surface features; however, it does not easily lend itself to quantitative surface roughness characterisation. This can be overcome by complementing SEM investigations with atomic force microscopy (AFM). In SEM, a highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated, the intensity of which is governed by surface topography. The method is suitable for all materials, but non-conducting materials must be given a thin conductive coating (e.g., carbon or gold sputtered), which can alter or mask the true surface morphology. The resolution of topographical features is ~5 nm. SEM is often used to survey a surface before more specialised techniques are employed.

Electron dispersive x-ray analysis (EDX)

EDX involves analysis of the elemental composition of a surface from X-rays emitted upon exposure to a primary beam of electrons. The X-rays emitted are characteristic of the atom from which they originated. 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 2-10 mm and the volume analysed can be as large as several cubic microns. The technique can be used to generate elemental distribution maps of the area of interest, enabling both qualitative (boron to uranium) and quantitative (sodium to uranium) elemental analysis. EDX cannot provide information on chemical bonds, although it can provide information on element depletion and migration because of environmental exposure.

Infrared spectroscopy (IRS)

IRS provides information on molecular structure based on specific frequencies associated with internal vibrations of groups of atoms in molecules. It uses a laser in the infrared region to excite the target material and analyse the frequencies absorbed. The technique makes use of the fact that for polymeric materials the chemical bonds between the atoms in the polymer molecules can vibrate at frequencies in the IR range of the electromagnetic spectrum (i.e., at wave numbers from 100 to 4000 cm^{-1}). The technique can be used to analyse gases, liquids, and solids. Both qualitative and quantitative chemical analysis data can be obtained. It is a key tool for assessing polymer chemistry (e.g., monitoring state of cure and determining environmental effects).

X-ray photoelectron spectroscopy (XPS)

XPS is an analytical technique that measures the energies of photoelectrons emitted from atoms of a sample when it is irradiated with soft (or low energy) X-rays. The technique is surface-sensitive and frequently used for quantitative elemental analysis of fracture surfaces, to determine the effect of surface preparation on surface chemistry and for monitoring chemical changes in adhesive samples. The technique, which can detect all elements with the exceptions of hydrogen and helium, can provide information on chemical structure (e.g., oxidation states) and element distribution present on the surface of any solid material. The technique is surface-sensitive with a maximum operational depth of less than 10 nm with a spatial resolution < 10 mm. The technique can be used in conjunction with inert gas ion sputtering to determine the variation in chemical composition with depth.

Auger electron spectroscopy (AES)

AES is a surface sensitive, non-destructive technique for identifying the elements in the first few atomic layers (~1 to 5 nm) on a specimen surface and can provide quantitative data on the detected elements. High-energy electron beam bombardment of the surface results in the emission of Auger electrons at characteristic discrete energies. 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 (spatial resolution of 0.5 mm). The technique can detect all elements except for hydrogen and helium. 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.

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 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.

Chapter 8

Non-destructive evaluation techniques

- Introduction
- Visual inspection
- Ultrasonics
- Acoustic emission (AE)

Introduction

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

- Visual Inspection
- Ultrasonics (C-scan)
- X-ray Radiography
- Thermography
- Acoustic Emission

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

Visual inspection

After the adhesive is cured, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the bonded area. The trained eye can detect specimen misalignment (i.e., twist and non-parallel edges), unfilled areas and voids. Joints containing adhesive depleted regions around the edges or ends of the bonded area should not be tested. Thick, thin, or uneven bond-line can also be detected visually or by using a micrometer or optical microscope. Checks should also be made on the adhesive fillet to ensure that the fillet complies with test specification. It is also advisable to check that the adhesive is fully cured and has bonded to the surface of the adherend. The texture and hardness can be a clue as to the effectiveness of the curing process.

Several optical microscopy techniques are available for producing visible images of structures or details too small to be visible by the human eye, using an optical microscope (or other magnification tool). Microscopy either involves diffraction, reflection, or refraction of radiation incident upon the subject of study and the subsequent collection of this scattered radiation to build up an image of the surface being inspected. This process may be carried out by wide field irradiation of the sample (e.g., standard light microscopy) or by scanning of a fine beam over the sample (e.g., confocal laser scanning microscopy). The maximum resolution is ~0.2 µm on idealised objects, but normally the resolution is limited to 0.5 µm. At the highest magnifications (where the maximum resolution is possible) the depth of focus is of the order of 1 µm. Contrast enhancement can be achieved through staining different structures with selected dyes.

Ultrasonics

Ultrasonic inspection is routinely used for quality assurance purposes and for in-service inspection of engineering structures, particularly composite material systems. It uses high frequency sound energy to interrogate for surface and subsurface discontinuities or flaws. The sound energy is introduced and propagates through the material in the form of waves. The sound waves propagate through the material with attendant loss of energy (attenuation) and are reflected at interfaces. The reflected wave signal is transformed into an electrical signal, which is displayed and then analysed to determine the location, size and orientation of discontinuities or flaws (e.g., cracks or disbonds), and variations in material density. Ultrasonic signals are scattered or reflected from any interface that separates regions of differing acoustic impedance. The reflective signal at the interface becomes smaller as the differences in density between the two medium decreases. Discontinuities or flaws, such as cracks, shrinkage cavities, voids, inclusions, and porosity are detectable using ultrasonic inspection. Thickness and elastic properties can also be measured using ultrasonic techniques.

Ultrasonic C-scan 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. Planar defects, as small as 0.3 mm in size, can be detected and accurately located using ultrasonic techniques. Planar resolution is limited by the ultrasonic transducer diameter. Although a 0.3 mm spatial resolution is possible with many of the high-resolution imaging systems, technical expertise is required to obtain this degree of accuracy. The technique is not suitable for detecting surface contaminants (e.g., oils and grease). Discontinuities that are present immediately beneath the top surface are difficult to detect. This region is called the "dead zone" and is typically 0.1 to 0.25 mm thick. The maximum inspection depth for fibre-reinforced polymer composites is typically 40-50 mm. Discontinuous reinforced systems tend to be difficult to inspect due to attenuation of the ultrasonic signal because of dispersion due to the fillers. Viscoelastic effects in the polymer also contribute to attenuation along with porosity, and damage or defects within composite materials.

X-ray radiography

X-radiography uses localised differences in attenuation under X-ray illumination to provide a cross-sectional picture of the density of a material system. Traditionally images have been recorded on film although increasingly, digital, or real-time recording systems are used. The method is well suited to volume defects and to complex components, which might be difficult to inspect by other methods. X-ray radiography is suitable for detecting the presence of voids and solid inclusions (e.g., backing film) in the adhesive bond-line. It can be used to inspect for debonds in polymer composite joints and between the outer skins and inner core of bonded composite sandwich structures. The technique is also capable of detecting micro-cracks in polymeric materials. X-radiography is not suitable for inspecting joints with metal adherends. Thin debonds and delaminations are difficult to detect because the presence of these defects has minimal effect on the absorption characteristics of polymer materials.

The use of penetrant fluids can enhance the imaging process; however, these fluids can adversely affect the short-term properties and fatigue performance of polymeric materials. The fatigue life can be reduced by a factor of 2 or more. 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.

Thermography

Thermography refers to the detection of defects by measuring differences in thermal response, usually by monitoring infrared emissions using a thermal imaging camera. Thermography 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 CCD cameras.

Acoustic emission (AE)

Acoustic emission (AE) is the transient elastic (stress) waves within a material caused by the release of elastic energy through deformation or fracture. AE may arise from friction (including bearing wear), micro-crack formation, delamination growth and material changes such as corrosion. Although AE is not strictly an NDE technique, it can be used to monitor bonded joints for delaminations and debonds during mechanical testing. AE testing involves the detection of transient elastic waves in the radio frequency range of 1 kHz to 100 MHz propagating through the material microscopic events can be detected if sufficient energy is released. Source location is also possible using multiple sensors attached to the surface or embedded within a structure. AE sensors convert small mechanical movements into electrical signals, which are analysed using computer-based instrumentation. AE can be used to monitor the “state of health” of a structure. The technique relies on the operator having sufficient experience to be able to identify defect types from the AE data.

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[57] BS 7079:2009, General Introduction to Standards for Preparation of Steel Substrates Before Application of Paints and Related Products.

[58] BS EN 13887:2003, Structural Adhesives – Guidelines for the Surface Preparation of Metals and Plastics Prior to Adhesive Bonding.

[59] ISO 11357-1: 2016, Differential Scanning Calorimetry (DSC) – Part 1: General Principles.

[60] ISO 6721-1:2011, Plastics – Determination of Dynamic Mechanical Properties – Part 1: General Principles.

[61] M. J. Lodeiro, D. Mulligan, Cure Monitoring Techniques for Polymer Composites, Adhesives and Coatings, NPL Measurement Good Practice Guide No. 75, 2005.

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Appendix 1

List of test methods and standards

Material Property	Standard/Test Method
Mechanical (Bulk Tests)	
Plastics/Adhesives	
Tension	ISO 527-1 and ISO 527-2, ASTM D638
Compression	ISO 604, ASTM D695
Shear	ISO 15310 (moduli), ASTM D732 (punch test)
Metals	
Tension	ISO 6892-1, ASTM E8
Compression	ASTM E9
Shear	ISO 6892-1 (modulus), ASTM B769 (aluminium)
Composites	
In-plane Tension	ISO 527-4 (multidirectional)/ISO 527-5 (unidirectional)
In-plane Compression	ISO 14126
In-plane Shear	ISO 14129/ISO 15310 (modulus)/ASTM D5379
Through-Thickness Tension	ASTM D7291
Through-Thickness Compression	NPL Draft Procedure
Through-Thickness Shear	ASTM D5379
Mechanical (Adhesive Joint Tests)	
Tensile Butt Joint	ASTM D897, ASTM D2095, BS EN 15870
Shear (torsion)	ISO 11003-1
Shear (TAST)	ISO 11003-2, ASTM D5656
Fracture Toughness	
Mode I (Adhesives)	ISO 25217 (DCB + TDCB)
Mode I (Composites)	ISO 15024/ASTM D5528
Mode II (Composites)	ISO 15114/ASTM D7905
Additional Tests	
Single-Lap Shear	ISO 4587, ASTM D1002, BS EN 1465, BS 5350-C5
Double-Lap Shear	ASTM D3528, BS 5350-C5
T-Peel Test	ISO 11339, ISO 8510-2, ASTM D1876
Climbing Drum Peel Test	ASTM D1781, BS 5350-C13
Floating Roller Peel Test	ISO 4578, ASTM 3167
Wedge Cleavage Test	ISO 10354, ASTM D3433
Compact Tension	ASTM D1062, BS 5350-C1
Effect of Water/Moisture	ISO 62 (water/moisture absorption)/ISO 175
Effect of Chemicals	ISO 175
Effect of Heat Ageing	ISO 216
Test/Conditioning Atmospheres	ISO 291
Failure Patterns	EN 923
Dynamic Mechanical Analysis	ISO 6721
Differential Scanning Calorimetry	ISO 11357

Table A1.1. Test methods for determining input design/analysis data

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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.

Acknowledgements

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Contents

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 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.

Bond-line: 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.

Cure: To set or harden by means of a chemical reaction.

Cure time: Time required to affect a cure at a given temperature.

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

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

Elastomer: A rubbery material that returns to approximately its original dimensions in a short time after undergoing a large amount of deformation.

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

Exothermic: A chemical reaction that emits heat.

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 bond-line.

Fracture toughness: The resistance of a material (or interface) to crack propagation. Defined for different modes of fracture. Mode I is direct tension, Mode II is in-plane shear and Mode III is out-of-plane shear.

Gel: A semi-solid system consisting of a network of solid aggregates in which liquid is held.

Gelation: Formation of a gel.

Glass transition: A 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.

Hygroscopic: Material capable of absorbing and retaining environmental moisture.

ISO: International Standards Organisation.

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

Open time: Time interval from when an adhesive is applied to when the material becomes unworkable.

Peel: Mode of application of a force to a joint in which one or both 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.

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

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

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

Substrate: An adherend, 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.

Tack: The property of an adhesive that enables it to form a bond of measurable strength immediately after adhesive and adherend are brought into contact under low pressure.

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 bond-line.

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

Thermoplastic: A material that can be repeatedly softened by heating.

Traveller: A test specimen used for example to measure moisture content because of environmental conditioning.

Viscosity: Resistance of a liquid material to flow.

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

Wetting: A surface is considered completely wet by a liquid if the contact angle is zero, and incompletely wet if the contact angle has a finite value.

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

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

Executive summary

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 polymer matrix 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 basic knowledge of the materials and techniques involved and is not intended as a textbook. There are several 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 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 both initial adhesion preparation 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 manufacturer is obtained, and 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 (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.

There are several published works, which provide a comprehensive coverage of adhesive technology and preliminary design [1-9]. Other NPL Measurement Good Practice Guides “Preparation and Testing of Bulk Specimens of Adhesives”, “Durability Performance of Adhesive Joints” and “Characterisation of Flexible Adhesives for Design” [10-12] provide advice on issues relating to the preparation and testing of bulk adhesive and adhesive joint specimens, acquisition of design data from bulk specimens, finite element modelling of adhesives, flexible adhesives, and durability testing.

The intention of the guide is to complement these published works. It is recommended that specialist advice be sought from adhesive manufacturers on adhesive selection, use of associated technologies and health and safety requirements.

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Chapter 1

Scope

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. Several of these methods 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 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 bond-line, often referred to as in-*situ* testing. 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 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 (QA) 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 1 shows a list of recommended test methods (see Table A1.1) for determining input data for the design and analysis of bonded joints.

The guide also includes generalised surface preparation techniques for metal and fibre-reinforced polymer matrix composites (PMCs) substrates and a summary of useful surface analytical and thermal analysis techniques. These techniques 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.

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Chapter 2

Measurement of bulk adhesive properties

- Introduction
- Specimen preparation
- Test methods and standards
- Moisture conditioning and testing

Introduction

Bulk adhesive testing is 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 chapter 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.

Specimen preparation

Tests on bulk adhesive specimens can provide reliable elastic and strength property data for design purposes. However, there are several key points that should be considered when producing bulk adhesive specimens [l0-16]:

- 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 ± 2 °C, 50 ± 10 relative humidity (RH)), which can reduce the glass-transition temperature T_g and mechanical propertiese of the adhesive.

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 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 thermal analysis (DMTA) measurements can be used to compare the final state of cure of the materials (see Chapter 6) [17].

Recommended procedures for the preparation of bulk specimens of adhesives are given in ISO 15166 Parts 1 and 2 [13-14]. Part 1 of the standard considers two-part (component) adhesives cured at ambient or elevated temperatures, whilst Part 2 covers single component systems requiring an elevated curing temperature (see also [10-11]). Test specimens may either be moulded or sectioned from manufactured resin plaques. It is important that the specimen is free of surface damage (i.e., scratches and nicks). To minimise the deleterious effects of surface scratches, which may cause premature failure, the edges and faces of the specimens should be carefully polished to remove any surface defects.

Handling adhesives can be hazardous to human health, thus COSHH (Control of Substances Hazardous to Health Regulations) procedures should be followed to minimise operator exposure. A safety data sheet should accompany all adhesive samples (Health and Safety at Work Act 1974). The user prior to handling the material must read this document. The safety data sheet supplied with the adhesive identifies:

- Hazardous ingredients and concentrations - includes regulatory information according to legislative directives.
- Hazard statements, such as H225 (highly flammable liquid and vapour), H315 (causes serious eye irritation) and H319 (causes serious eye irritation), and precautionary statements, such as P260 (do not breathe dust/fume/gas/mist/vapours/spray) and P280 (wear protective gloves/protective clothing/eye protection/face protection). Hazard statements are accompanied by classification (e.g., Flammable liquid. 2 – H225 and Eye irritation. 2 – H315).
- First aid measures (including inhalation, ingestion, skin contact and eye contact), and important symptoms and effects, both acute and delayed (e.g., prolonged skin contact may cause redness and irritation). The severity of the symptoms will vary dependent on the concentration and the length of exposure.
- Fire extinguishing/fighting (suitable and unsuitable extinguishing media, and protective equipment and actions) and accidental release (personal and environmental precautions, and methods for cleaning up spillages or discharges) measures.

- Precautions for safe handling and storage including storage precautions (e.g., keep away from heat, sparks, and open flame) and storage class (e.g., flammable liquid storage).
- Control parameters and occupational exposure limits (short-term and long-term exposure limits).
- Exposure controls (includes engineering measures (e.g., adequate ventilation), respiratory protection, eye, skin and body protection, hygiene measures, respiratory protection, and environmental exposure controls).
- Physical and chemical properties (i.e., colour, odour, melting and boiling points, vapour pressure, flammability, etc.), and stability and reactivity of ingredients, and storage and transportation conditions (e.g., temperature).
- Toxicological information (refer to COSHH) – includes potential health effects and hazardous toxicity data.
- Ecological information, disposal considerations and transport information.

Test methods and standards

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.

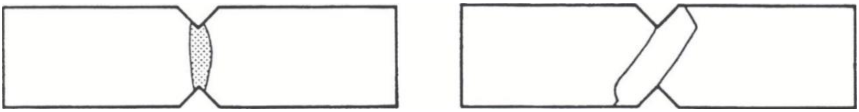
Longitudinal and transverse strain can be measured using either strain gauges, contacting extensometers or video extensometers (non-contact technique) [10]. It is important when using contact techniques, such as strain gauges and extensometers, that the transducer(s) are capable of functioning within the test environment and have suitable response time. Strain gauges are not recommended for adhesive characterisation due to: (i) stiffening effects on lowstiffness materials; (ii) limited range of operation; and (iii) tendency to act as failure initiation sites in 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 is advisable to regularly coat threaded surfaces with a protective grease.

Shear

The V-notched beam method (ASTM D5379 [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 ±45° 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 using 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.

Figure 1. V-notched beam shear fixture and specimen



Thermoplastic – Shear Yielding **Thermoset – Brittle Tensile**

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

Shear strain is measured with biaxial strain gauges (1-2 mm gauge-length) bonded at ±45° 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 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].

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 D695 [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.

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 FRPs) absorb 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 several 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 conditioningof bulk adhesive and testing of adhesive specimens that have been exposed to either hot/humid environments or immersed in water at elevated temperatures.

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; 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 thin specimens to determine the "through-the-thickness" moisture diffusion coefficient.

The International standard 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. 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 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, 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 (sealed container with desiccant) 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 to ensure accurate moisture absorption measurements. To minimise moisture uptake prior to conditioning, specimens are stored in a desiccator at room temperature. It should be noted that under standard laboratory conditions many adhesives absorb significant levels of moisture.

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 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 humidity's 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 rapid in the initial 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 initial 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 conducted at approximately equal intervals of $(\text{time})^{1/2}$. The derived moisture content M can be determined as follows:

$$M = \frac{\left(W_{wet} - W_{dry}\right)}{W_{dry}} \times 100\% \qquad (1)$$

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 throughout the conditioning process (includes temperature, relative humidity, 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 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]:

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

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.

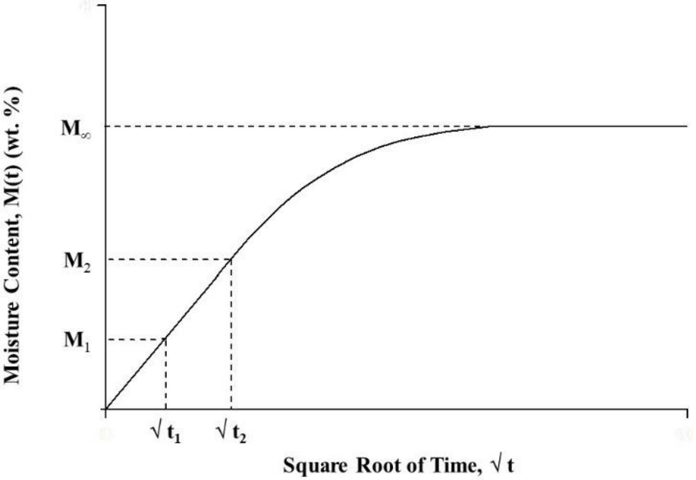


Figure 3. Fickian diffusion curve

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 shorter time to reach than time required to reach full equilibrium. The time required to obtain the last 5% can be longer than the time taken to reach the 95% level. Clearly a 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 ±5%. It is essential that boiler units of humidity cabinets be supplied from a deionised/distilled water reservoir to prevent salt deposits on test specimens, corrosion of bonded joints and scaling of the plumbing.

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 the previous section. 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 1: It is assumed that all absorbed moisture is translated into a change in resin volume. fu 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.

Mechanical testing of 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 represented by a relative humidity of 85% RH and a temperature of 70 °C.

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.

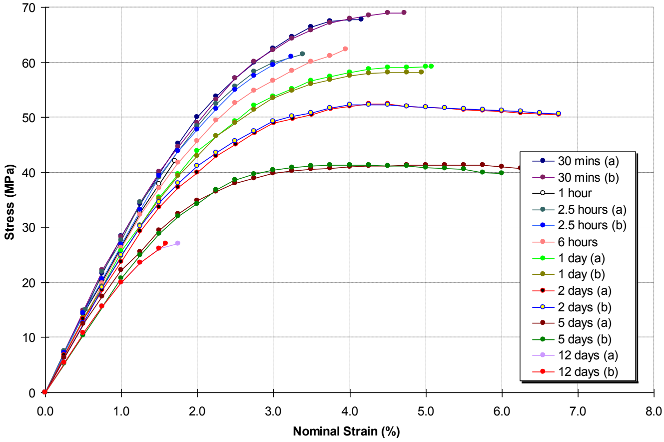


Figure 4. Tensile stress-strain curves for different water immersion periods

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 wi1ing 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, conditioned 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 polythenebag containing a salt solution appropriate to the humidity requirements of the test, are not practical. Traveler specimens 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.

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Chapter 3

Measurement of adherend properties

- Tension
- Compression
- Shear
- Through-thickness (T-T) properties
- Scope

Introduction

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

Tension

ISO 6892-1 [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 ISO 527-4 [31] for multidirectional composite laminates and 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.

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-load 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-tabbed (to prevent failure at the specimen ends). No problem is expected for the use of the test configurations for creep and environmental testing of metals or composites.

Shear

There is no standard at present for determining shear strength of metals. Shear modulus for these materials can be determined from tensile tests. 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 ±45° laminate, 250 mm long, 25 mm wide and 2 mm thick. Specimen preparation and testing are similar to those specified in ISO 527-4 [31]. 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 minimizes 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.

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 Chapter 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.

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Chapter 4

Adhesive Joint Testing

- Introduction
- Tensile properties
- Shear properties
- Quality assurance testing
- General issues relating to environmental testing
- Creep
- Cyclic fatigue
- Basic design considerations

Introduction

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

- The stress distribution within the bond-line tends to be non-uniform in a majority of test joint configurations with stress concentrations existing at the bond-line 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 bond-line 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.

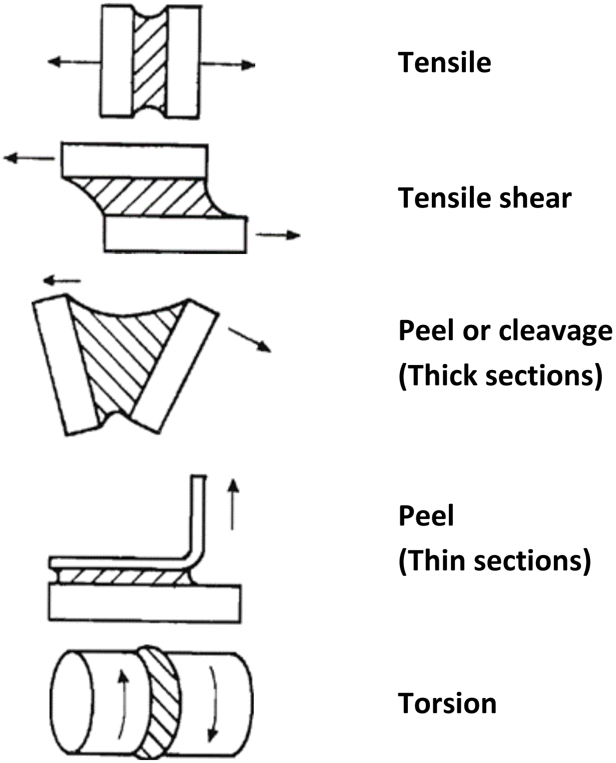

Figure 5. Basic loading modes experienced by adhesive joints

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.

Tensile properties

The cylindrical butt joint (Figure 6) can be used to test thin bond-line 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 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 D897 [38], ASTM D2095 [39], and BS EN 15870 [40].

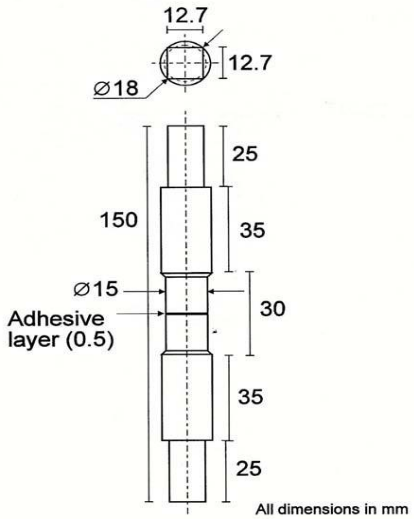

Figure 6. Tensile butt joint

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.**

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 (thick adherend shear test (TAST)). The TAST specimen can be produced by either:

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

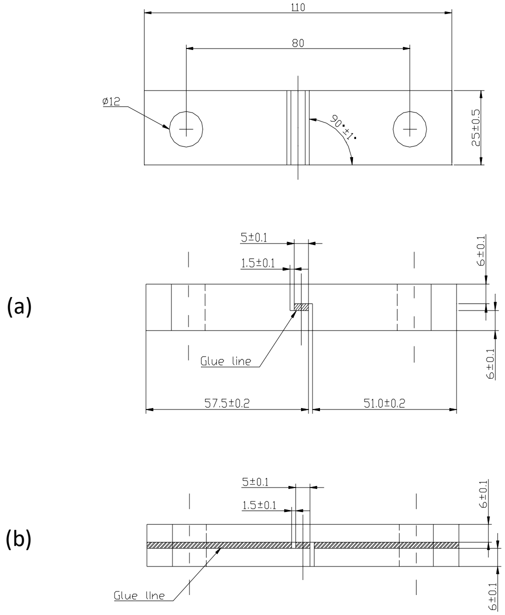

Figure 7. TAST specimen: (a) pre-shaped adherends; (b) bonded sheets (dimensions in mm)

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.

ISO 11003-2 [41] specifies a specimen with an overall length of 110 mm, a width of25 mm and overlap length of 5 mm. The ISO standard recommends an adherend thickness of 6 mm and a bond-line 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.

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].

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 [42-45]. End tabs, cut from the same material as the adherend sections, are adhesively bonded to the specimen. The end tabs, which are typically 37.5 mm in length, 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.

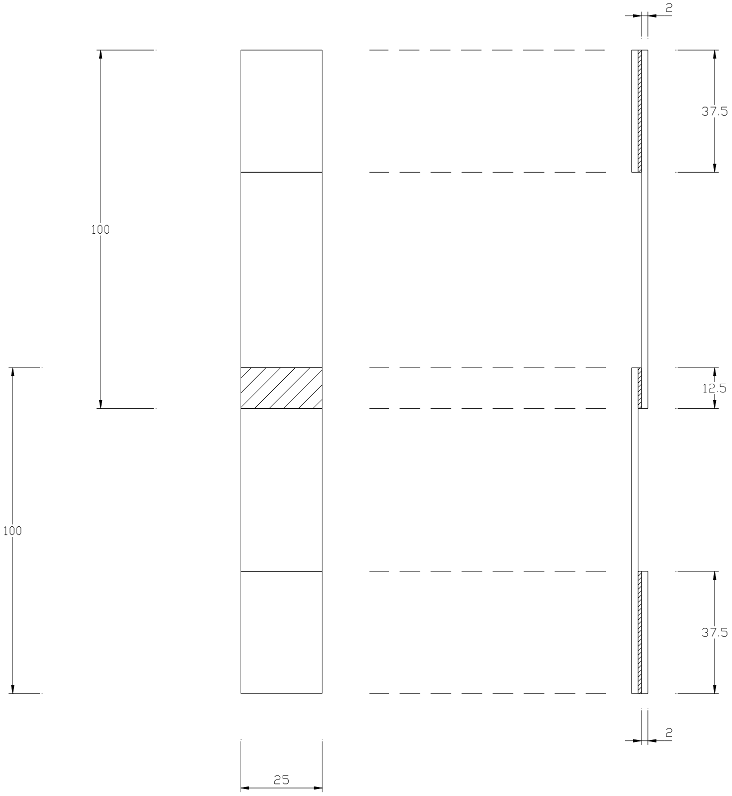

Figure 8. Schematic of single-lap joint (dimensions in mm)

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]:

$$L = \frac{\sigma_Y t}{1.5\,\tau} \tag{3}$$

where σ is the yield stress of the adherend, τ = average shear strength of the adhesive and t is the 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 fibre-reinforced plastics (FRPs). 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].

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 Ro 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.

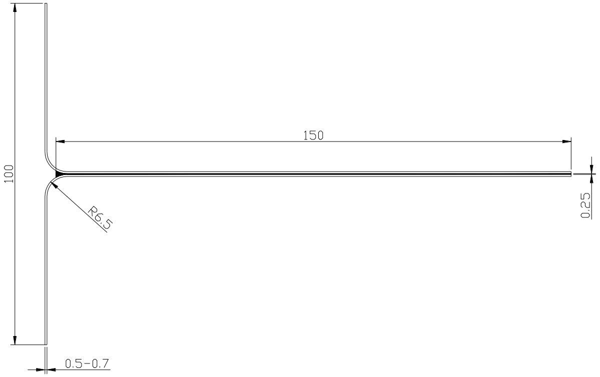

Figure 9. Schematic T-peel joint (dimensions in mm)

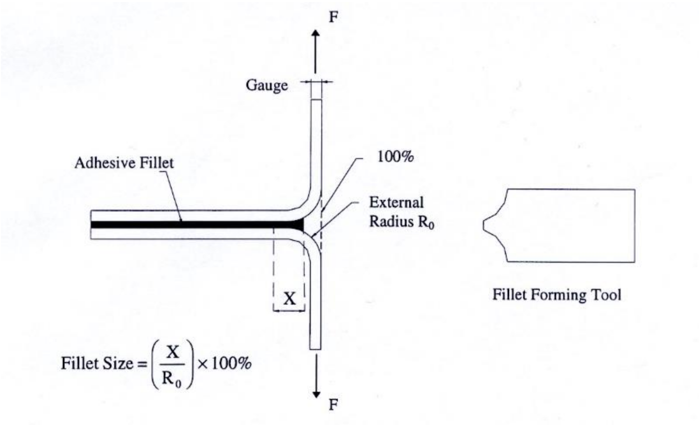

Figure 10. Definition of fillet size in T-peel joint [51] – including fillet forming tool

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 Chapter 5, whilst Chapter 7 provides a brief summary of surface and chemical characterisation techniques. The standard 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 (Chapter 2). 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 ISO 62 [26]). For non-equilibrium moisture levels, the moisture distribution is non-uniformly distributed; as shown in Figure 11.

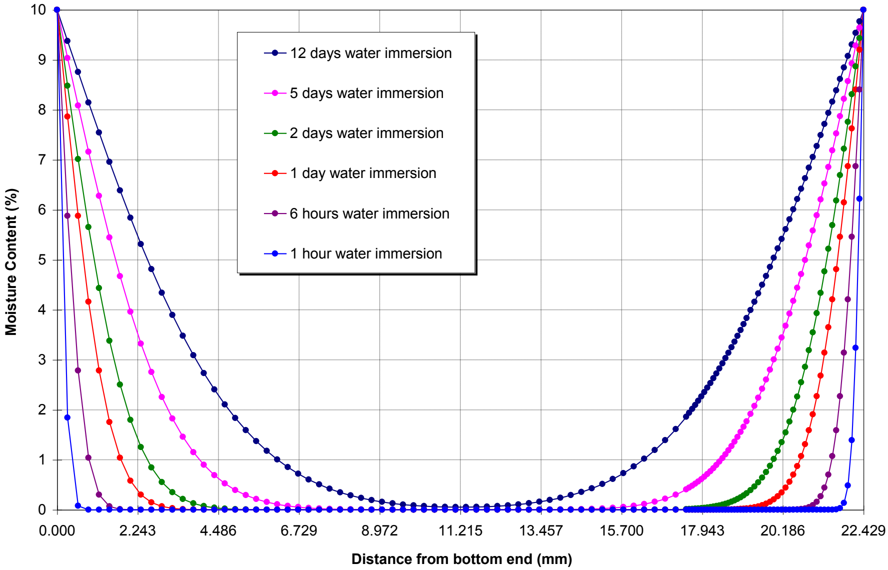

Figure 11. Typical moisture concentration distributions along an adhesive layer

Creep

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. Specimens are removed at regular intervals to assess strength reduction.
- **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:

1. Servo-hydraulic test machines.
2. Dead-weight and lever creep testing machines.
3. A screw jack in series with a load cell (see Figure 12); and
4. Self-stressing fixture where specimens are placed in either a tube equipped with a calibrated spring system (see Figure 12) for loading specimens or a circular ring.

Figure 12. Screw jack machines (left) and a self-stress tube (right)

The use of a servo-hydraulic test machine is not an economic option in most cases. 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 12), 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 (ISO 14615 [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 shown in Figure 12 can load 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 lifetime 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 to accurately determine time-to-failure. Specimens loaded by springs can often be in an unstressed state for a considerable period (overnight or weekends) before the failed joint is replaced (by a “dummy” specimen) and the loading train is 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 system evaluated. Typically, these are between 10 and 50% of the short-term strength of the joint. It is recommended that the sustained stress in an adhesive bonded joint under service conditions should be kept below 25% of the short-term strength of the joint. The onset of failure tends to be catastrophic. In general, damage and stiffness loss prior to crack initiation is minimal. The time involved in crack formation tends to be far greater than the time associated with crack propagation. Methods for accelerating the testing process that use mechanical loading tend to use stress levels that are significantly higher than stress levels used in design, thus the limiting design strains are reached in shorter times than in actual service.

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) specimens per stress level with five stress levels per condition should provide a reasonable number of data points. 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).

Cyclic fatigue

The fatigue properties of a bonded joint are a function of the joint geometry and adhesives, and therefore cannot be determined from the 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). Fatigue data are normally obtained at the highest frequency possible to minimise the duration of tests. The uncertainty in life expectancy at any stress level is typically an order of magnitude.

Restrictions on test frequency can arise from test equipment limitations (response time), time dependent processes and hysteretic (self-generated) 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. The upper frequency limit will be dependent upon the thermal conductivity of the adherend/adhesive system, mode of loading and specimen size. Trials may be necessary to determine the upper frequency limit.

It is recommended that the temperature rise of the material surface be kept to a minimum. 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, although the latter is beyond the budget of most industrial facilities. The temperature resolution is ~1 °C for the two methods.

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 13), thus improving creep and fatigue performance. It also provides added protection from environmental attack. Increasing the bond-line 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.

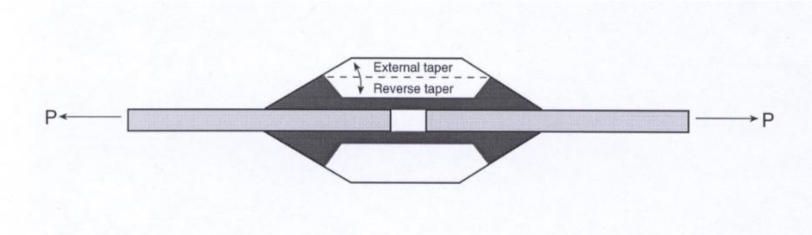

Figure 13. Bevelled strap joint

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 14) for adhesive joints can be approximated (rule of thumb) by a straight line fit as follows:

$$\frac{P_{MAX}}{P_0} = 1 - klogNf \tag{4}$$

where k is the slope, N_f is the number of cycles to failure, P_{MAX} is the maximum load applied to the specimen, P_0 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 (Figure 15) has a far better fatigue performance than tests where peel stresses are the major cause of failure.

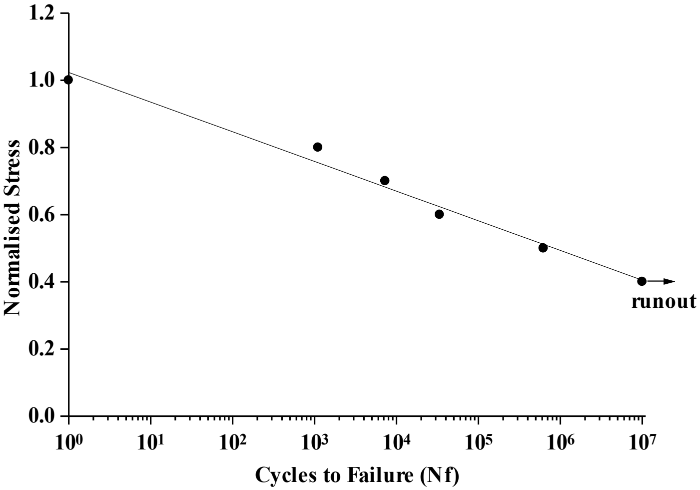

Figure 14. Normalised S-N curve for a tapered-strap joint

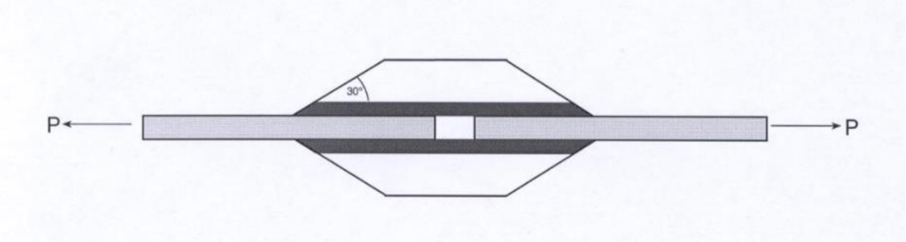

Figure 15. Tapered-strap joint

Joint Configuration	*k value*
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)	0.013

Table 1. Typical k values for bonded joints tested at room temperature

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.

Chapter 5

Surface preparation of adherends

- Introduction
- Steel
- Zinc coated steel
- Stainless steels
- Aluminium alloys
- Titanium alloys
- Fibre-reinforced polymer composites
- General comments

Introduction

Surface preparation is recognised as the most critical step in the adhesive bonding process and considerable adhesive joint testing is performed to optimise surface treatment. The selection of surface treatment is largely dependent on the required strength and durability of the joint, although economic considerations, such as costs and time involved in preparation, also play a role in the selection process. Correct surface preparation is essential for good joint strength and maintaining long-term structural integrity of bonded joints. Unsatisfactory surface preparation will result in the bond failing adhesively and unpredictably at the adhesive/adherend interface.

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 affects 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 chapter 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 ISO 17212, ASTM D2093, ASTM D2651, BS 7079 and BS EN 1840 [54-58]. Advice should be sought on surface preparation from the adhesive manufacturer.

Note 1: After completion of the surface preparation process, the adherends must not be exposed to physical handling or uncontrolled atmospheric environments to prevent surface contamination prior to bonding. It is advisable that bonding be performed immediately following surface treatment to maximise performance. 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 should be carried out to COSHH specifications.

Steel

The recommended method for preparing steel substrates for bonding is as follows [1]:

- 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.

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 [1]:

- 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 and then dry the surface.
- Apply a suitable primer, if required by the adhesive manufacturer.

Stainless steels

The recommended steps to be taken in preparation of stainless-steel surfaces is as follows [1]:

- 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).

Aluminium alloys

The recommended steps to be taken in preparation of aluminium alloy surfaces are [1]:

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

Titanium alloys

The recommended steps to be taken in preparation of aluminium alloy surfaces are [1]:

- Degrease with a suitable solvent (e.g., acetone or methyl ethyl ketone (MEK)).
- Grit blast - remove dust/debris by oil-free air blast or by vacuum cleaner.
- Acid etchthe surface.
- Rinse and dry.
- Chemically stabilise the oxide layer.

Fibre-reinforced polymer matrix composites

All fibre-reinforced polymer 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 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 bond-line, resulting in a weakened and porous adhesive layer. T_g and fracture toughness can be significantly lowered because 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 [1]:

- 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 affect the physical and mechanical properties of the outer layer of the cured laminate.

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.

Chapter 6

Thermal Analysis Techniques

- Introduction
- Differential scanning calorimetry (DSC)
- Dynamic Mechanical Analysis (DMA)
- Thermal Mechanical Analysis (TMA)
- Thermal gravimetric analysis (TGA)

Introduction

Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), can provide useful information relating to the composition and degradation (e.g., effects of moisture ingress on T_g) of adhesives. The analysis of material by any thermal analysis technique is complicated by changes in the material during testing. For adhesive materials, there will most likely be further curing or loss of moisture, when subjected to heating during thermal analysis. In some cases, these changes will prevent the accurate determination of the T_g. Repeat measurements need to be taken to check completeness of cure. It is important that the material assessed is representative of the adhesive within the bonded structure. This section briefly examines the suitability of using thermal analysis techniques for characterising environmental degradation of adhesives.

Differential scanning calorimetry (DSC)

DSC measures temperatures and heat flows associated with thermal transitions in a material (see ISO 11357-1 [59]). Operating temperature range is typically -180 °C to 700 °C, or higher. 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. DSC can be used to measure T_g and degree of cure, however it is not particularly suitable for determining the effect of moisture on T_g as the small samples tend to dry out during testing.

Modulated DSC offers improved resolution and sensitivity not possible using other techniques. The heat flow signal is composed of several parts (i.e., reversing, and non-reversing events). Conventional DSC can only measure and illustrate the sum of those parts in a single signal, whereas with MDSC multiple signals are generated in a single experiment so that individual components of the total heat flow signal can be displayed and analyzed independently. Typical reversing events are glass transitions and examples of non-reversing events are thermoset cure and decomposition.

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 has many advantages, including:

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

Dynamic mechanical analysis (DMA)

DMA (or dynamic mechanical thermal analysis (DMTA)) measures the stiffness and damping properties of materials as a function of time, temperature, and frequency by applying a sinusoidal load to a specimen and measuring the resultant deformation, whilst the sample is subjected to a controlled temperature programme. The magnitude of the applied stress and the resultant strain are used to calculate the stiffness (modulus) of the material under stress. The phase lag δ between stress and strain is used to determine tanδ, the damping factor. This technique enables the determination of mechanical (storage (elastic) and loss (damping) modulus) and thermal (e.g., T_g) properties of polymeric materials over a wide range of temperatures (-150 °C to 600 °C) and frequencies (0.01 to 200 Hz) [60, 61]. Modulus values obtained using DMA are not as accurate as conventional mechanical test techniques; measurements are sensitive to specimen dimensions and loading conditions (including clamping forces). DMA is suitable for polymeric materials with stiffness ranging from 1 kPa to 1,000 GPa.

The technique has many advantages, including:

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

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 measures changes in dimension (e.g., expansion or contraction), modulus or viscosity of a material as a function of temperature. The temperature may be increased or decreased linearly or stepwise, kept constant or modulated with a set frequency and amplitude. Operating temperature range is typically -150 °C to 1000 °C. TMA can be used to measure heat deflection and glass transition temperatures, thermal expansion coefficients, and characterise creep and stress relaxation behaviour at different stress and temperature conditions. Thermal transition behaviour can be used as a quality assurance technique for determining the extent of resin cure.

Thermal gravimetric analysis (TGA)

TGA measures weight changes in a material as a function of temperature (or time) under a controlled atmosphere. The sample weight is continuously monitored as the temperature is increased either at a constant rate or through a series of steps. The components of a polymer will decompose at different temperatures, thus producing step changes in weight to enable quantitative measurements of the different components. TGA instruments can operate up to a temperature of 1000 °C. It can provide valuable information on moisture and volatile content.

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Chapter 7

Surface analytical techniques

- Scanning electron microscopy (SEM)
- Electron dispersive x-ray analysis (EDX)
- Infrared spectroscopy (IRS)
- X-ray photoelectron spectroscopy (XPS)
- Auger electron spectroscopy (AES)
- Chromatographic analysis

Introduction

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 chapter provides a 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.

Scanning Electron Microscopy (SEM)

This is the most widely used of the surface analytical techniques. High resolution SEM has proved an invaluable tool for studying surface topography, oxide growth and failure analysis. The technique enables qualitative three-dimensional (3-D) imaging of surface features; however, it does not easily lend itself to quantitative surface roughness characterisation. This can be overcome by complementing SEM investigations with atomic force microscopy (AFM). In SEM, a highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated, the intensity of which is governed by surface topography. The method is suitable for all materials, but non-conducting materials must be given a thin conductive coating (e.g., carbon or gold sputtered), which can alter or mask the true surface morphology. The resolution of topographical features is ~5 nm. SEM is often used to survey a surface before more specialised techniques are employed.

Electron dispersive x-ray analysis (EDX)

EDX involves analysis of the elemental composition of a surface from X-rays emitted upon exposure to a primary beam of electrons. The X-rays emitted are characteristic of the atom from which they originated. 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 2-10 mm and the volume analysed can be as large as several cubic microns. The technique can be used to generate elemental distribution maps of the area of interest, enabling both qualitative (boron to uranium) and quantitative (sodium to uranium) elemental analysis. EDX cannot provide information on chemical bonds, although it can provide information on element depletion and migration because of environmental exposure.

Infrared spectroscopy (IRS)

IRS provides information on molecular structure based on specific frequencies associated with internal vibrations of groups of atoms in molecules. It uses a laser in the infrared region to excite the target material and analyse the frequencies absorbed. The technique makes use of the fact that for polymeric materials the chemical bonds between the atoms in the polymer molecules can vibrate at frequencies in the IR range of the electromagnetic spectrum (i.e., at wave numbers from 100 to 4000 cm^{-1}). The technique can be used to analyse gases, liquids, and solids. Both qualitative and quantitative chemical analysis data can be obtained. It is a key tool for assessing polymer chemistry (e.g., monitoring state of cure and determining environmental effects).

X-ray photoelectron spectroscopy (XPS)

XPS is an analytical technique that measures the energies of photoelectrons emitted from atoms of a sample when it is irradiated with soft (or low energy) X-rays. The technique is surface-sensitive and frequently used for quantitative elemental analysis of fracture surfaces, to determine the effect of surface preparation on surface chemistry and for monitoring chemical changes in adhesive samples. The technique, which can detect all elements with the exceptions of hydrogen and helium, can provide information on chemical structure (e.g., oxidation states) and element distribution present on the surface of any solid material. The technique is surface-sensitive with a maximum operational depth of less than 10 nm with a spatial resolution < 10 mm. The technique can be used in conjunction with inert gas ion sputtering to determine the variation in chemical composition with depth.

Auger electron spectroscopy (AES)

AES is a surface sensitive, non-destructive technique for identifying the elements in the first few atomic layers (~1 to 5 nm) on a specimen surface and can provide quantitative data on the detected elements. High-energy electron beam bombardment of the surface results in the emission of Auger electrons at characteristic discrete energies. 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 (spatial resolution of 0.5 mm). The technique can detect all elements except for hydrogen and helium. 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.

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 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.

Chapter 8

Non-destructive evaluation techniques

- Introduction
- Visual inspection
- Ultrasonics
- Acoustic emission (AE)

Introduction

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

- Visual Inspection
- Ultrasonics (C-scan)
- X-ray Radiography
- Thermography
- Acoustic Emission

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

Visual inspection

After the adhesive is cured, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the bonded area. The trained eye can detect specimen misalignment (i.e., twist and non-parallel edges), unfilled areas and voids. Joints containing adhesive depleted regions around the edges or ends of the bonded area should not be tested. Thick, thin, or uneven bond-line can also be detected visually or by using a micrometer or optical microscope. Checks should also be made on the adhesive fillet to ensure that the fillet complies with test specification. It is also advisable to check that the adhesive is fully cured and has bonded to the surface of the adherend. The texture and hardness can be a clue as to the effectiveness of the curing process.

Several optical microscopy techniques are available for producing visible images of structures or details too small to be visible by the human eye, using an optical microscope (or other magnification tool). Microscopy either involves diffraction, reflection, or refraction of radiation incident upon the subject of study and the subsequent collection of this scattered radiation to build up an image of the surface being inspected. This process may be carried out by wide field irradiation of the sample (e.g., standard light microscopy) or by scanning of a fine beam over the sample (e.g., confocal laser scanning microscopy). The maximum resolution is ~0.2 μm on idealised objects, but normally the resolution is limited to 0.5 μm. At the highest magnifications (where the maximum resolution is possible) the depth of focus is of the order of 1 μm. Contrast enhancement can be achieved through staining different structures with selected dyes.

Ultrasonics

Ultrasonic inspection is routinely used for quality assurance purposes and for in-service inspection of engineering structures, particularly composite material systems. It uses high frequency sound energy to interrogate for surface and subsurface discontinuities or flaws. The sound energy is introduced and propagates through the material in the form of waves. The sound waves propagate through the material with attendant loss of energy (attenuation) and are reflected at interfaces. The reflected wave signal is transformed into an electrical signal, which is displayed and then analysed to determine the location, size and orientation of discontinuities or flaws (e.g., cracks or disbonds), and variations in material density. Ultrasonic signals are scattered or reflected from any interface that separates regions of differing acoustic impedance. The reflective signal at the interface becomes smaller as the differences in density between the two medium decreases. Discontinuities or flaws, such as cracks, shrinkage cavities, voids, inclusions, and porosity are detectable using ultrasonic inspection. Thickness and elastic properties can also be measured using ultrasonic techniques.

Ultrasonic C-scan 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. Planar defects, as small as 0.3 mm in size, can be detected and accurately located using ultrasonic techniques. Planar resolution is limited by the ultrasonic transducer diameter. Although a 0.3 mm spatial resolution is possible with many of the high-resolution imaging systems, technical expertise is required to obtain this degree of accuracy. The technique is not suitable for detecting surface contaminants (e.g., oils and grease). Discontinuities that are present immediately beneath the top surface are difficult to detect. This region is called the "dead zone" and is typically 0.1 to 0.25 mm thick. The maximum inspection depth for fibre-reinforced polymer composites is typically 40-50 mm. Discontinuous reinforced systems tend to be difficult to inspect due to attenuation of the ultrasonic signal because of dispersion due to the fillers. Viscoelastic effects in the polymer also contribute to attenuation along with porosity, and damage or defects within composite materials.

X-ray radiography

X-radiography uses localised differences in attenuation under X-ray illumination to provide a cross-sectional picture of the density of a material system. Traditionally images have been recorded on film although increasingly, digital, or real-time recording systems are used. The method is well suited to volume defects and to complex components, which might be difficult to inspect by other methods. X-ray radiography is suitable for detecting the presence of voids and solid inclusions (e.g., backing film) in the adhesive bond-line. It can be used to inspect for debonds in polymer composite joints and between the outer skins and inner core of bonded composite sandwich structures. The technique is also capable of detecting micro-cracks in polymeric materials. X-radiography is not suitable for inspecting joints with metal adherends. Thin debonds and delaminations are difficult to detect because the presence of these defects has minimal effect on the absorption characteristics of polymer materials.

The use of penetrant fluids can enhance the imaging process; however, these fluids can adversely affect the short-term properties and fatigue performance of polymeric materials. The fatigue life can be reduced by a factor of 2 or more. 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.

Thermography

Thermography refers to the detection of defects by measuring differences in thermal response, usually by monitoring infrared emissions using a thermal imaging camera. Thermography 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 CCD cameras.

Acoustic emission (AE)

Acoustic emission (AE) is the transient elastic (stress) waves within a material caused by the release of elastic energy through deformation or fracture. AE may arise from friction (including bearing wear), micro-crack formation, delamination growth and material changes such as corrosion. Although AE is not strictly an NDE technique, it can be used to monitor bonded joints for delaminations and debonds during mechanical testing. AE testing involves the detection of transient elastic waves in the radio frequency range of 1 kHz to 100 MHz propagating through the material microscopic events can be detected if sufficient energy is released. Source location is also possible using multiple sensors attached to the surface or embedded within a structure. AE sensors convert small mechanical movements into electrical signals, which are analysed using computer-based instrumentation. AE can be used to monitor the “state of health” of a structure. The technique relies on the operator having sufficient experience to be able to identify defect types from the AE data.

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Useful contacts

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Appendix 1

List of test methods and standards

Material Property	Standard/Test Method
Mechanical (Bulk Tests)	
Plastics/Adhesives	
Tension	ISO 527-1 and ISO 527-2, ASTM D638
Compression	ISO 604, ASTM D695
Shear	ISO 15310 (moduli), ASTM D732 (punch test)
Metals	
Tension	ISO 6892-1, ASTM E8
Compression	ASTM E9
Shear	ISO 6892-1 (modulus), ASTM B769 (aluminium)
Composites	
In-plane Tension	ISO 527-4 (multidirectional)/ISO 527-5 (unidirectional)
In-plane Compression	ISO 14126
In-plane Shear	ISO 14129/ISO 15310 (modulus)/ASTM D5379
Through-Thickness Tension	ASTM D7291
Through-Thickness Compression	NPL Draft Procedure
Through-Thickness Shear	ASTM D5379
Mechanical (Adhesive Joint Tests)	
Tensile Butt Joint	ASTM D897, ASTM D2095, BS EN 15870
Shear (torsion)	ISO 11003-1
Shear (TAST)	ISO 11003-2, ASTM D5656
Fracture Toughness	
Mode I (Adhesives)	ISO 25217 (DCB + TDCB)
Mode I (Composites)	ISO 15024/ASTM D5528
Mode II (Composites)	ISO 15114/ASTM D7905
Additional Tests	
Single-Lap Shear	ISO 4587, ASTM D1002, BS EN 1465, BS 5350-C5
Double-Lap Shear	ASTM D3528, BS 5350-C5
T-Peel Test	ISO 11339, ISO 8510-2, ASTM D1876
Climbing Drum Peel Test	ASTM D1781, BS 5350-C13
Floating Roller Peel Test	ISO 4578, ASTM 3167
Wedge Cleavage Test	ISO 10354, ASTM D3433
Compact Tension	ASTM D1062, BS 5350-C1
Effect of Water/Moisture	ISO 62 (water/moisture absorption)/ISO 175
Effect of Chemicals	ISO 175
Effect of Heat Ageing	ISO 216
Test/Conditioning Atmospheres	ISO 291
Failure Patterns	EN 923
Dynamic Mechanical Analysis	ISO 6721
Differential Scanning Calorimetry	ISO 11357

Table A1.1. Test methods for determining input design/analysis data

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