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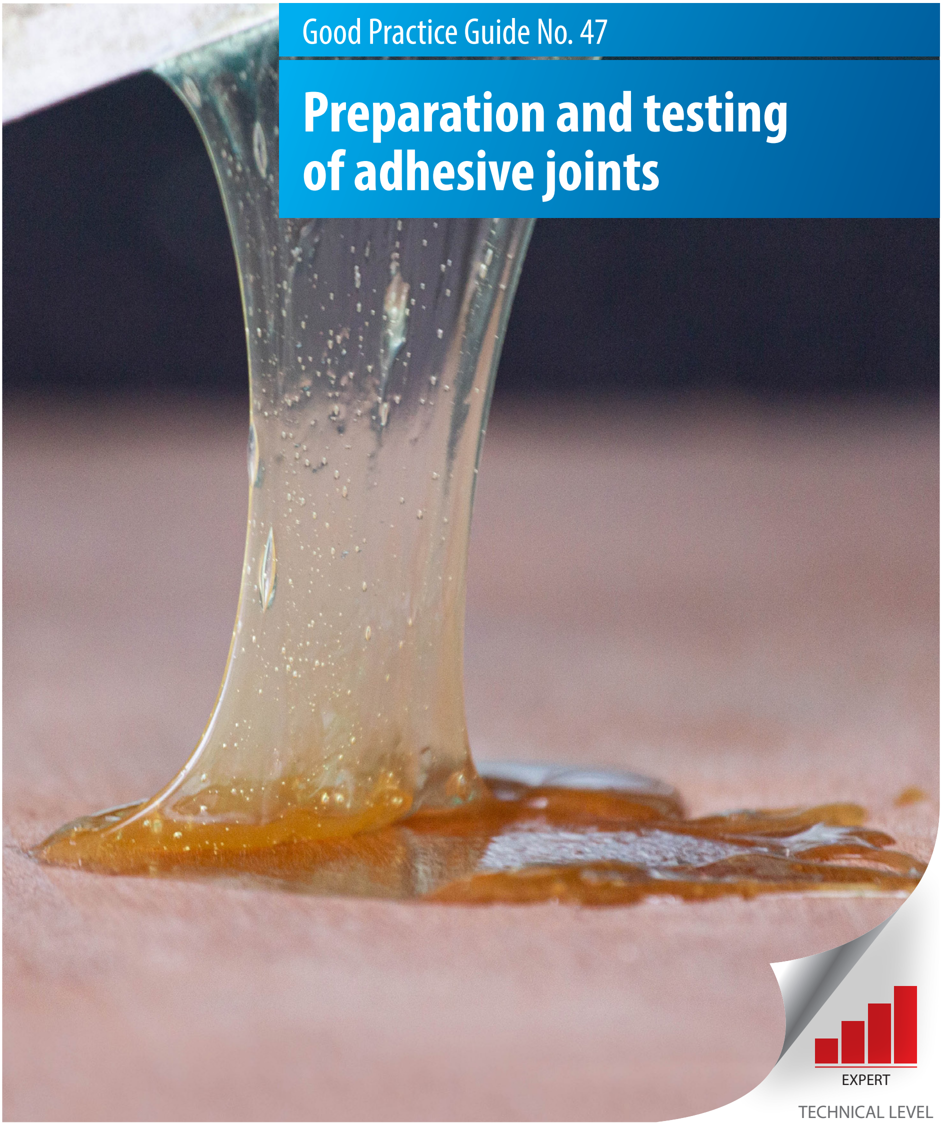
Good Practice Guide No. 47

Preparation and testing of adhesive joints



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Abstract

The reliability (i.e., repeatability and reproducibility) of data obtained from testing adhesive joints can be significantly affected by factors not included in written standards. Factors, such as specimen and machine alignment, processing variables, storage of materials and surface treatments need to be considered in addition to test parameters (e.g., displacement rate) and specimen dimensions that are generally covered in national and international standards. Careful consideration needs to be given to those factors that can significantly influence the strength and long-term performance of adhesive joints. This Good Practice Guide is intended to give guidance on the preparation and testing of adhesive joints, and the use of test methods under ambient and hostile environments (including accelerated ageing) for generating design data and for quality assurance purposes. Static, cyclic fatigue and creep rupture testing are also included. The document is primarily concerned with structural adhesives, but also includes flexible adhesives for structural or semi-structural applications.

The Good Practice Guide also includes the results from a three-stage Round-Robin exercise on *Adhesive Joint Testing* that was conducted in conjunction with UK industry to identify key preparation and testing issues.

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.

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

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

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

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

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

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

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

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

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

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

Structural bond: 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.

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Executive summary

Executive summary

This measurement guide aims to provide guidance to technologists, laboratory personnel and quality assurance personnel on the preparation and testing of adhesive joints for generating design data and for quality assurance purposes. Guidance is provided on static, cyclic fatigue, and creep rupture testing, and testing of adhesive joints in hostile environments (including accelerated testing). The document covers specimen preparation (including adherend machining, surface treatments, joint assembly, and bonding), specimen storage and mechanical testing (including specimen and machine alignment, strain, and displacement measurement). Consideration is given to the effect of material and geometric factors (i.e., bond-line thickness and fillet geometry), and test parameters (i.e., displacement rate) on joint stiffness and strength. The document considers thermal analysis techniques for monitoring cure and non-destructive evaluation (NDE) techniques for inspection of bonded structures.

The document includes a summary of commonly used tensile, peel, cleavage, shear, and fracture toughness test methods, providing guidance on issues relating to specimen requirements, material properties obtained (including associated accuracy), materials selection and amount of material required, costs of specimen preparation and testing, data reduction and suitability for use under creep, fatigue, and environmental conditions. The document, which is primarily concerned with adhesives for structural applications (including toughened and flexible adhesives), covers joints that involve substrates fabricated from either metals or fibre-reinforced thermoset polymer composites. 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 reliably test adhesive joints for producing design data and to enable initial screening of adhesive/adherend/surface treatments. If the intention is to generate design data, then the guide should be used in conjunction with the appropriate structural design codes. The guide assumes some basic knowledge of the materials and techniques involved and is not intended as a textbook.

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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Correct surface preparation is essential for ensuring both initial adhesion and long-term joint durability. Although the guide provides advice on the surface preparation of the commonly used structural materials, it only covers the main steps in each process. It is essential that expert advice from the adhesive manufacturer is obtained and that the detail requirements specified by the manufacturer are completely satisfied.

Where tests are performed to characterise the adhesive material then it is recommended that the surface preparation is as good as possible to minimise premature adhesion failure. Where tests are performed to evaluate a bonding system then the surface preparation procedures for test specimens will need to mirror those for the final bonded component.

The Guide includes the results from a three-stage Round-Robin (R-R) exercise on *Adhesive Joint Testing* that was carried out as part of the assessment to identify key preparation and testing issues. The R-R exercise involved eight industrial companies and the NPL Materials Centre. The results have been analysed using statistical analysis. A good example of an industrial procedure for preparing and testing adhesive joints is presented within the overview of the R-R exercise.

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Chapter 1

Scope

Scope

Adhesive tests can be divided into comparative methods for adhesive and process selection, quantitative methods for generating engineering property data for design purposes, and qualitative methods for assessing long-term (i.e., durability) performance of bonded systems under combined mechanical loading and hostile environments. Although, an extensive range of test methods are available as national and international standards (see Appendix 1), 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, or comparative analyses 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. Lists of standards issued by the American Society for Testing and Materials (ASTM), British Standards Institution (BSI) and International Standards Organisation (ISO) are presented in NPL Report CMMT(A)61 [13].

A major concern with testing adhesive joints is the number of factors, not included in written standards, which can significantly affect the reliability (i.e., repeatability and reproducibility) of test data. Factors, including specimen and machine alignment, processing variables, surface treatment, adhesive storage and joint ageing need to be considered in addition to test and specimen parameters (e.g., displacement rate and dimensions) that are covered in national and international standards. It was evident from a statistical analysis of R-R data carried out in a previous adhesive programme that the uncertainty associated with specimen preparation and testing has a substantial effect on the test results. The large scatter and disparity in results between test houses indicates that several additional factors need to be controlled to ensure reliable data. The situation is further exacerbated when tests are conducted in hostile environments.

The guide is primarily concerned with metals and fibre-reinforced polymer composites bonded with structural adhesives, which includes toughened and flexible adhesives. Guidance is provided on specimen preparation, storage of adherends, adhesives and adhesive joints, and testing and environmental conditioning of adhesive joints. Static, cyclic fatigue and creep rupture testing are covered. Consideration is given to the effect of material and geometric factors on joint stiffness and strength. A list of recommended test methods for determining input data for the design and analysis of bonded joints is given in Appendix 1.

A three-stage R-R exercise was conducted on *Adhesive Joint Testing* in conjunction with adhesive suppliers, end users and test houses within the UK, as part of the assessment to identify key preparation and testing issues. The R-R results, which have been analysed using statistical analysis, are presented in Appendix 2. The R-R exercise involved nine participants (eight industrial companies and NPL).

A brief description of generic procedures for preparing different material surfaces is given in Appendix 3. Appendix 4 provides technical diagrams to construct a bonding fixture for producing single-lap joint specimens. A schematic diagram of the alignment fixture used at NPL for bonding tensile butt joint specimens is shown in Appendix 5.

Organisations that can provide specialist advice are listed at the back of the guide along with relevant standards and publications.

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Chapter 2

Handling and storage of adhesives

- Introduction
- Handling and storage
- Documentation (record handling)
- Measurement of bulk adhesive mechanical properties

Introduction

Several issues relating to the handling and storage of adhesives need to be considered as many of these issues can affect the performance of the material. Issues that need to be considered include:

- Health and safety (COSHH)
- Documentation
- Handling and storage (including moisture and temperature effects)

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.

Handling and storage

Polymeric resins may undergo physical and chemical changes when stored for a considerable length of time or under unsatisfactory storage conditions, thus adversely affecting the performance of the adhesive. For example, adhesives may undergo an increase in viscosity, or films a loss of tack and drapeability. Less flow and, thus poorer wetting of the surface, will occur during cure and gel times of epoxy film adhesives could become shorter. Extended exposure of uncured materials to temperatures and humidity's above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [3]. The overall effect is to reduce the adhesive performance and induce premature failure of adhesively bonded joints.

The safety and product data sheets will provide the user with recommendations regarding storage temperature for an adhesive. Adhesives should be stored in sealed containers to prevent moisture ingress and contamination, and some cases exposure to light. Opening a storage container exposes the adhesive to oxygen and moisture, which may in some cases severely reduce the shelf life of the adhesive. The shelf (storage) life is generally defined as the time that an adhesive can be stored (unopened) without any adverse affects on the cure properties and bond strength of the adhesive. The product sheet will often specify the shelf life of the adhesive for various temperature ranges. An adhesive should not be used beyond the expiry date, which will be indicated on the container. The shelf life of an adhesive can decrease significantly with increasing storage temperature. Unless the manufacturer explicitly states that the adhesive must stored or would benefit from being stored at sub-ambient temperatures then adhesives should be stored at ambient temperatures (18 °C - 25 °C). Adhesives should not be stored below freezing unless recommended by the manufacturer.

Ideally, temperature and humidity in the storage area should be tightly controlled and monitored (i.e., temperature recorders), although this may not always be physically or economically feasible. Standard laboratory conditions are typically 23 ± 2 °C and 50 ± 5 % relative humidity (RH). Refrigerators (or freezers) should be armed with a temperature alarm and the temperature should be monitored continuously to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. The maintenance of the refrigerator units should allow for regular manual or automatic defrosting.

A refrigerated adhesive may be used several times during the shelf life of the product. This will involve the product being taken from a sub-ambient (often sub-zero) temperature and placed at room temperature and allowed to thaw. It is advisable throughout this exercise that the adhesive is enclosed (i.e., remains enclosed in a sealed container) until the temperature has reached room temperature and is ready to be applied to the adherends. Containers that have been opened must be sealed immediately after use. The amount of time at ambient for these adhesives should be minimal, advice on the length of time can be obtained from the adhesive manufacturer. Repeated exposure of the adhesive is a frequent cause of adhesive ageing and deterioration. Sampling of the adhesive before removal from sub-ambient storage may help extend the shelf life of the product, however this may not always be possible for adhesive packed in cartridges.

Exposure to repeated freeze/thaw cycling could limit the shelf life of an adhesive. Exposing a film adhesive that has been stored at sub-ambient temperatures will result in moisture condensing on the surface, which if not allowed to evaporate will compromise the strength and durability of the joint. Entrapped moisture will be released during the cure process, thus compromising the physical and mechanical properties of the adhesive and contribute to the degradation of the bonded surfaces. Routine tests to verify that the physical and mechanical properties of the adhesives are still within specification are recommended, particularly where multiple use of an adhesive has occurred.

Adhesives should be stored in an area specifically dedicated to these materials. The area should be suitable for handling flammable and hazardous materials. It may be necessary to segregate materials depending on the hazardous nature of the material. Materials in these areas should be clearly identified and controlled.

Documentation (record keeping)

It is recommended that an accurate record of the adhesive be maintained, and that routine checks are made each month to ensure that the stored materials are within the time limits recommended by the adhesive manufacturer. A record should include details on the material source, manufacturer’s code number, batch or lot number, form (i.e., one-or two-part, or film, etc.), shelf life, storage temperature and handling history during the shelf life (i.e., dates of use and length of time at room temperature if refrigerated). The report should include test data from acceptance tests and routine quality checks carried out to assess material performance.

Adhesive manufacturers will often supply data sheets (material certification) with their adhesives, normally quoting lap-shear strengths. Data sheets may sometimes include measured adhesive joint strength (i.e., tensile shear, fatigue strength, creep rupture and T-peel data) for a range of temperatures and environmental conditions (i.e., long-term exposure to methanol, gasoline, acetone, acetic acid, or water). The manufacturer usually employs either aluminium or titanium substrates for material qualification. The data supplied may not be easily applied to other surfaces. Few manufacturers provide bulk adhesive data. These properties usually need to be generated in-house (see next Section).

It is recommended that an accurate check as to the quality of an adhesive be carried out on receipt of the material to ensure that material meets the manufacturers specifications, ensuring the product complies with the purchase order and suppliers test reports (see [9]). The colour, viscosity, shelf life, etc. should be verified. It may be necessary to carry out acceptance tests in-house to ensure mechanical properties are in accordance with the material specification. Acceptance tests should be directed towards assuring that the materials are identical from batch to batch. Accelerated environmental tests (i.e., high temperature and humidity) may also need to be carried out to confirm that the durability performance of the material meets the manufacturer specifications. ISO, BSI, and ASTM list various test methods for quality assurance of adhesives (see Appendix 1 and [13]).

Measurement of bulk adhesive mechanical properties

Bulk adhesive tests, which enable the determination of adhesive properties without the need for correcting for adherend effects, are often used to obtain accurate mechanical and physical properties for computer aided design calculations. A cautionary note, steps must be taken to ensure that the adhesive material is representative of the material within the actual bond layer. Recommended procedures for the preparation of bulk specimens of adhesives are given in ISO 15166 [14-15]. The standard considers two-part adhesives cured at ambient or elevated temperatures (Part 1) [14] and single component systems requiring an elevated temperature to cure the adhesive (Part 2) [15] - see also references [16-19]. Bulk adhesive specimens can be cast or machined to the required shape (e.g., dumbbell tensile specimens) - see [10, 12].

It is important that the specimen is free of surface damage (i.e., scratches and nicks). Many liquid and film adhesives can be cast into bulk specimens without the need for machining. To minimise the deleterious effect of surface scratches, which may cause premature failure, the edges and faces of the specimens should be carefully polished to remove any surface defects. Advice on specimen preparation, test methods and standards, and environmental conditioning and testing of bulk adhesives (including referenced standards) are given in references [11-19].

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Chapter 3

Handling and storage of adherends

Before and after machining, adherends sensitive to environmental attack or hygroscopic in nature should be stored in sealed containers to prevent moisture ingress and contamination. It is important that the adherend does not undergo physical or chemical changes whilst being stored. Moisture can alter the chemistry of both the surface and substrate of the adherend, thus compromising the performance of bonded joints. It is recommended that an accurate record of the adherend (including condition and use) be maintained throughout the duration of use the material. Inventory control mechanisms employed for adhesives are also relevant to the storage and handling of adherend materials (see Chapter 2).

Quality documentation should include details on the material source, manufacturer's code number, batch or lot number, form (i.e., sheet or plate), storage conditions, surface conditions of the adherends (including corrosion products, and surface defects such as scratches present on receipt of the material), and the location of each specimen sectioned from the adherend. The inventory report should also include (where necessary) test data (i.e., strength and stiffness properties) for the adherend. Test certificates are often supplied with metallic materials, such as titanium and aluminium alloys, in which an elemental analysis is supplied along with basic tensile property data. These sheets contain information on the materials condition (e.g., descaled, and annealed titanium). Fibre-reinforced polymer composite suppliers provide data sheets on processing conditions, fibre volume fractions and mechanical properties of their products. Often elastic and strength properties are quoted for a range of temperatures, and in some cases for hot/wet conditions (e.g., 70 °C and 85% RH) and other environments. It may be necessary to generate mechanical property data for design purposes. This data should be included in the documentation for the material. Variations in material thickness, and elastic and strength properties can be expected between batches of materials.

Chapter 4

Preparation and assembly of adhesive joints

- Machining and storage of adherends
- Surface preparation of adherends
- Joint assembly
- Curing adhesive
- Quality assurance

Introduction

The reliability of an adhesive joint depends not only on selecting the correct adhesive, but also on the preparation of the adherends, mixing of the adhesive, joint assembly, and the curing process. It is worth noting that a high percentage of failures can be attributed to poor joint manufacture or a lack of understanding of that factors that influence joint performance. These problems can be minimised or eliminated through proper training and education. This chapter examines the key issues relating to the preparation and assembly of adhesive joints.

Machining and storage of adherends

Adherends should be manufactured and/or machined accurately to ensure specimen dimensions meet the specifications of the standard. It is important to ensure that the adherends are free of any edge or surface damage. The surfaces to be bonded must be parallel and flat to ensure uniform, intimate contact, across the entire bond area when the two surfaces are clamped or pressed together. The operator should ensure that during the machining process, no nicks, cuts, or scratches are introduced at the edges or surfaces of the adherends. Surface or edge defects can cause premature failure of the adhesive joint and/or the adherend. It is advisable before preparing the surface to ensure that the adherend sections to be bonded fit together well with the bonded surfaces closely matching (i.e., intimate contact between the two surfaces). Guillotining thin metal sheets is a rapid and low-cost method for producing large quantities of lap-shear test adherends, however the cutting operation can result in bending of the adherends and operators will therefore need to be ruthless by discarding those specimens that fail to meet the specification.

Fibre-reinforced thermoset composites are known to absorb moisture in relatively benign environments. For example, epoxy based composite systems can absorb 0.2 wt. % moisture in a laboratory environment (i.e., 23 °C and 50% RH within 2 to 4 weeks. The presence of moisture in the composite can adversely affect the properties of the adhesive during the cure process, and as a result the joint strength may be compromised. Moisture released from the composite substrate during cure will enter the adhesive and has been known to reduce the glass transition temperature T_g by as much as 20 °C and lower the fracture toughness $\mathbf{G_c}$ of a rubber toughened epoxy adhesive by a factor of 10 [20]. With toughened adhesive formulations, the presence of moisture may inhibit phase separation of the rubber-toughening agent, thus preventing the formation of rubber-toughened particles. Adherends (pre-dried) should therefore be stored in a dry area (i.e., desiccator or sealed container with a suitable desiccant). It is recommended that polymer composites be pre-dried in an oven maintained at 50 ± 2 °C (unless otherwise specified) until the specimen weight reaches a constant value. The temperature of the drying oven should not exceed the maximum operating temperature of the polymeric matrix.

Surface preparation of adherends

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. Many adhesive joint tests have been developed to assess bonded systems (including surface preparation) – see Appendix 1 [13].

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. Appendix 3 provides a brief description of general procedures required for preparing different substrates for adhesive bonding. Many 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 D 2651, ASTM D 2093, BS 7079, BS EN 1840, and BS 7079 [21-25] – see also [26-29]. Advice should be sought on surface preparation from the adhesive manufacturer. **Surface preparation procedures often require potentially hazardous or environmentally damaging chemicals. All preparation should be carried out to COSHH specifications**.

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. Table 1 provides a guide as to the relative quality and cost of various surface treatments. Environmental impact of the process may also need to be assessed when selecting a surface treatment. Clean grit, clean solvent and clean cloths must be used to avoid spreading contamination. The maximum allowable time between surface preparation and bonding or priming metal and composite substrates is dependent on the substrate and the surface treatment (see Table 2).

Surface Treatment	Cost	Quality
None	Low	Low
Solvent Degrease	↓	↓
Vapour Degrease		
Mechanical Abrasion		
Plasma		
Chemical Etch		
Anodising	Expensive	Expensive

Table 1. Relative cost and quality of various surface treatments [9]

Surface Treatment	Cost	Quality
None	1-2 hrs	± 20
Solvent Degrease	1-2 hrs	± 20
Vapour Degrease	1-2 hrs	± 20
Dry Grit-Blasting (Steel)	4 hrs	± 20
Wet Grit-Blasting (Steel)	8 hrs	± 20
Wet Grit-Blasting (Aluminium)	72 hrs	± 20
Chromic Acid Etch (Aluminium)	6 days	± 10
Sulphuric Acid Etch (Stainless Steel)	30 days	± 10
Anodising (Aluminium)	30 days	± 10
Dry Grit + Organosilane (Aluminium)	2-5 hrs	± 5

Table 2. Maximum exposure time between surface preparation or priming metal substrates, and associated variation in tensile shear strength

Surface treatments can be classified as either passive or active [9]. Passive surface treatments (e.g., solvent washing and mechanical abrasion) clean the surface and remove weakly attached surface layers without altering the surface chemistry. Active surface treatments (e.g., anodising, chemical etching, laser, and plasma treatment) alter the surface chemistry (i.e., introduction of functional groups).

The combined effect of solvent and mechanical abrasion (i.e., grit-blasting) treatments is to remove most contaminants and alter the surface topography (increase in surface roughness or bond area). Chemical and plasma treatments also remove contaminants and increase surface roughness; however, these treatments also increase surface reactivity.

Silicon carbide grit-coated paper or other abrasive pads can be used dry or in the presence of a solvent to mechanically abrade the adherend surfaces. It is essential that sufficient time be allowed to ensure the solvent evaporates. Surfaces should be cleaned following abrasion to remove any abrasive material left on the surface. Alternatively, surfaces can be mechanically abraded through dry or wet grit blasting. Grit blasting in addition to removing surface debris and roughening the surface may cause physico-chemical changes, which affect surface energy and wettability [26]. Coarser grit produces rougher surfaces, resulting in lower surface energies, thereby increasing environmental durability of the bonded joint. Differences in initial strength between fine and coarse grits, and types of grit are negligible. Clean grit should always be used for preparing adherend surfaces. To prevent transfer of contamination, airline contamination and contamination of the grit should be avoided. Wet blasting is increasingly being used in preference to dry blasting as the method provides more consistent surface properties [26].

The results presented in Table 2 refer to unconditioned material that has been tested within a short period (e.g., 1-2 weeks) of joint fabrication. The variations in joint strength quoted in Table 2 are optimal values that can be expected under controlled conditions. The uncertainty (or variation) in tensile shear strength can be expected in many cases to increase following exposure to a hostile environment and/or through poor workmanship.

Simple surface treatments (e.g., grit blasting or vapour degreasing) are less prone to human error and therefore the variation in joint strength is unlikely to exceed ± 20%. Bonded joints with chemical surface treatments are more prone to large variations in joint strength, particularly as there are often numerous controlling variables, which need to be strictly controlled.

Primers such as aminopropyltriethoxysilane (or γ-APS) are often applied to: (i) protect the substrate surface prior to bonding; (ii) increase surface wettability; and (iii) and inhibit corrosion [9]. Primers also act as a coupling agent, forming chemical bonds with the adherend and adhesive, thus improving joint strength and environmental durability. Silane coupling agents (e.g., γ-APS) [27] are known to improve durability in the presence of moisture by increasing the water resistance of the oxide layer on the adherend surface. Joint strengths can be very low, however in cases where the silane treatment is poorly controlled. Incorrect application can result in interfacial failure or cohesive failure within the silane coating. Care is needed to ensure a uniform coating, a monomolecular layer thick, is produced across the entire surface area of the adherends to be bonded.

In all cases involving solvents, primers or active chemical surface treatments (e.g., acid and alkaline solutions), extreme care and good laboratory practice should be followed at all times in storing, handling and disposing of these materials (recommended further reading [9]). Personnel need to be trained in handling and using these materials and should wear appropriate protective clothing as the chemicals can be harmful if they come contact with the skin or eyes. Ventilation and spill containment are particularly important safety considerations. Chemical treating solutions should be prepared and stored in containers of glass, ceramic, or chemical resistant plastic. Checks should be made to ensure that the containing vessel is resistant to chemical attack from would be contents (e.g., hydrofluoric acid attacks glass). Stirring rods should be made of the same materials. Contact of the chemical solution with metals during preparation and storage should be avoided.

Continuous monitoring and quality control are required for all chemicals employed in surface treatments, and all containers with chemical agents should be clearly identified with labels. Labelling should be protected from being damaged through chemical contact or handling. Chemical solutions need to be changed regularly to prevent contamination and ensure repeatable concentration [9]. Quality assurance tests are recommended to check concentrations, and levels of contamination (e.g., opacity for checking particulate content, colour and pH for acidic and alkaline solutions).

Treatment tanks or containers with chemical etching solutions (e.g., chromic acid) should be of sufficient size to accommodate the specimens to be treated (i.e., volume of chemical solution should be well in excess of the volume occupied by the component in solution). Tests may need to be carried out to determine the minimum volume required to treat a component. Surfaces to be treated should be completely immersed in all solutions. Treatment tanks used in chemical etching processes should be equipped with temperature controls and should be regularly agitated or stirred to prevent local overheating and to ensure uniform concentration of the chemical etching solution. The tanks should be suitably lined to prevent reaction between the tank and the chemical solution, thus ensuring no adverse effect on the solution used or the surfaces being treated. Rinse tanks should be large enough to accommodate the specimens that have been treated and should be equipped to allow for circulation of clean water to prevent build-up or carryover of materials between batches of specimens.

Metallic surfaces need to be free of corrosive products (e.g., rust) before surface treatment, and the area should be as clean (i.e., free of dust, oil and grease) as possible. These need to be removed prior to surface treatment by mechanical abrasion or dry grit blasting. Vapour degreasing, solvent cleaners, emulsion cleaners or alkaline degreasers can be used when necessary to remove oils and grease from adherend surfaces. A new generation of adhesives have been developed to bond to oily surfaces. For these materials, removal of surface oil and grease is generally not conducive to good joint strength with manufacturers frequently recommending that the user avoid the removal of oil and grease, which participate in the chemical bonding process for these materials.

Immersion of adhesive joints in water at elevated temperatures or exposure to hot/humid environments (e.g., 70 °C and 85% RH) is a commonly used approach for assessing the effectiveness of different surface treatments. Figure 1 compares the relative environmental resistance of different surface treatments aged in water at elevated temperatures.

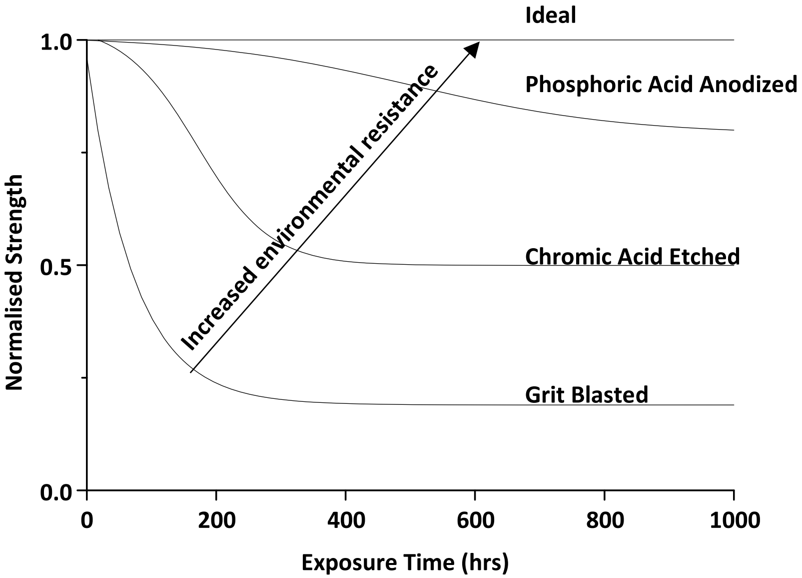

Figure 1. Effect of different surface treatments on the durability of aluminium single-lap joints bonded with an epoxy adhesive immersed in water at elevated temperature

Joint assembly

This section is concerned with issues relating to joint assembly prior to curing the adhesive (i.e., control of bond-line thickness and adhesive fillet and removal of adhesive spew).

Bonding fixture

A bonding fixture is recommended to ensure correct bond length (in the case of lap joints), accurate alignment and uniform bond-line thickness. An example of a bonding fixture for single-lap joints is shown in Appendix 4 [30]. The fixture shown in Appendix 4 enables the overlap length and placement of end tabs to be accurately controlled. The fixture, which allows for up to six individual specimens to be bonded at a time, has no facility to control the adhesive fillet. Alternatively, large test panels (typically 180 mm wide) capable of providing 6 specimens can be made and then cut into specimens. Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e., adherend bending) or that excessive adhesive is forced from the joint due to clamping forces applied to the test specimens.

It may be necessary to check the clamping force applied by the fixture to the specimen during the curing process to ensure that clamping force remains constant and has not relaxed through adhesive flow. Mould release agent or thin polytetrafluorene (PTFE) film will need to be used to guarantee easy release of bonded components from the clamping fixture.

Note 2: End tabs are introduced to single-lap joints 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 (see Figure 2). BS EN 1465 [31] 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. The introduction of end tabs, however, will add to the costs of this quality assurance test.

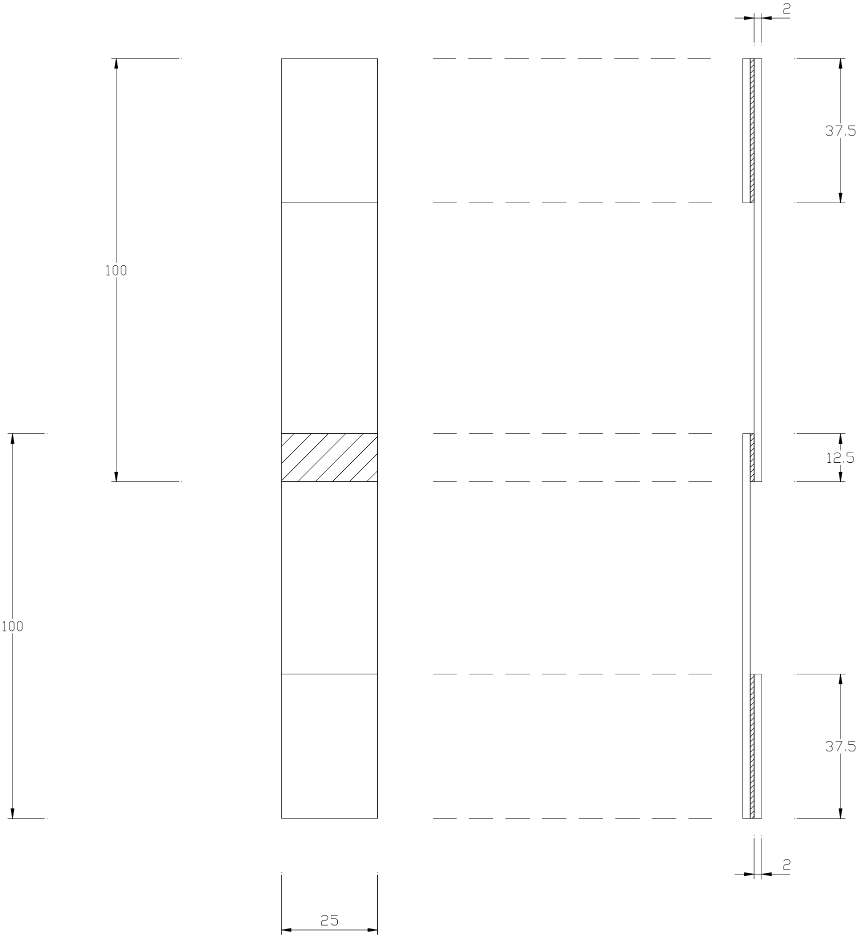

Figure 2. Schematic of single-lap joint (dimensions in mm)

A bonding fixture is essential for adhesive bonding of butt joints (see Appendix 5), as good alignment is key to this method producing data. The cylindrical butt joint (Figure 3) 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 take to ensure good alignment during specimen preparation (i.e., bonding of adherends) and testing. Small misalignment can severely reduce strength data. Variations of the test configuration have been included in ASTM 897 and ASTM D 2095 and BS EN 15870 [32-34]. At present, there is no definitive standard test method.

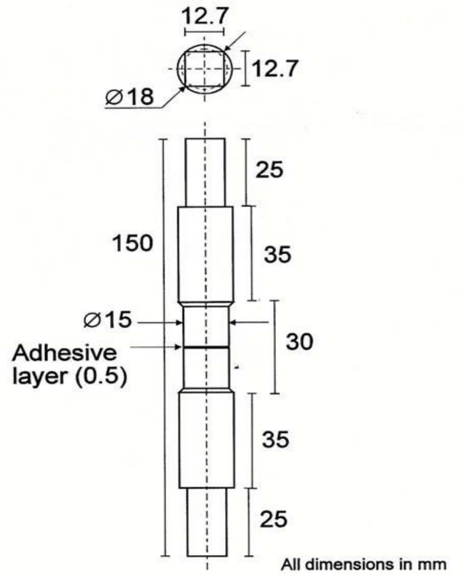

Figure 3. Cylindrical butt joint

Adhesive fillet

Efforts to reduce stress concentrations formed at the bond-line ends of joints may include the use of tapered or bevelled external scarf and radius fillets at the bond-line end (NB. The use of very rigid adherends will not eliminate stress concentrations at the bond-line). Significant increases in the "apparent" shear strength of single-lap joint, compared with square-ended bond-lines, can be achieved through the formation of a fillet or spew at the overlap ends. Further increases in strength may be achieved by rounding the ends of the adherends. The spew also acts as a barrier to water and chemical ingress from the surrounding environment.

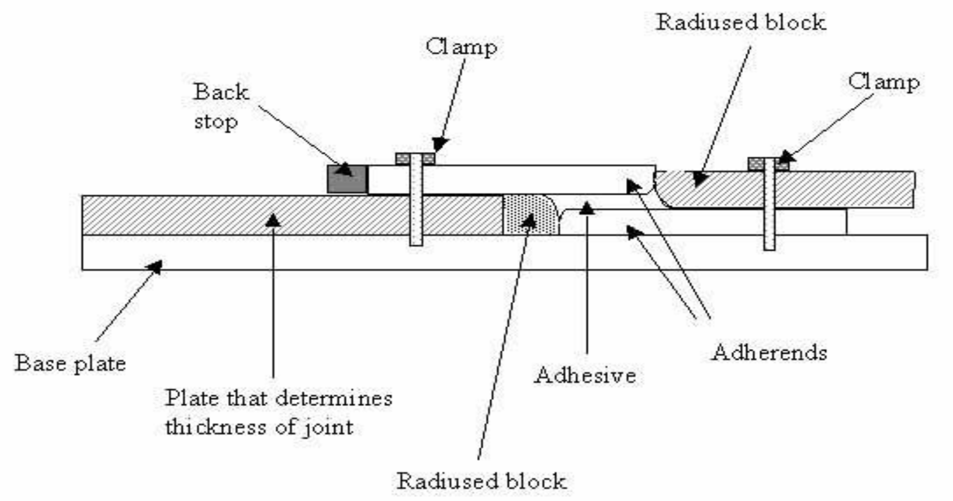

Figure 4. Fixture for controlling single-lap joint fillet

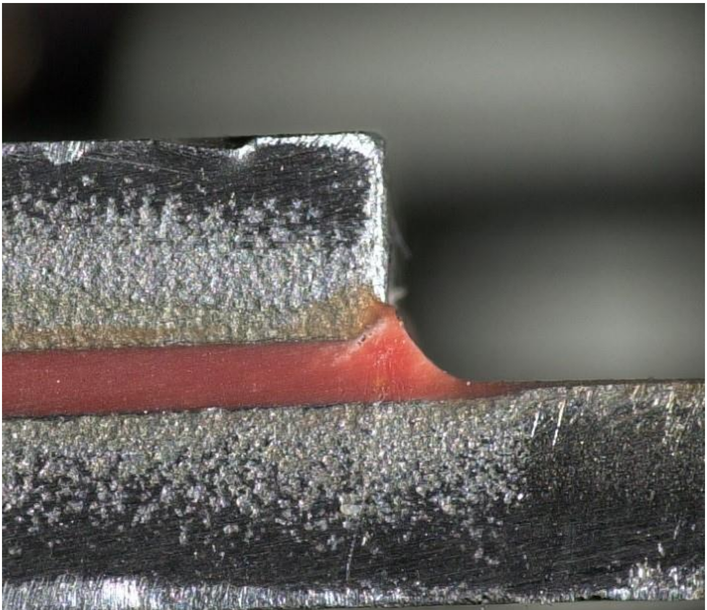

Figure 5. Single-lap joint with a concave fillet

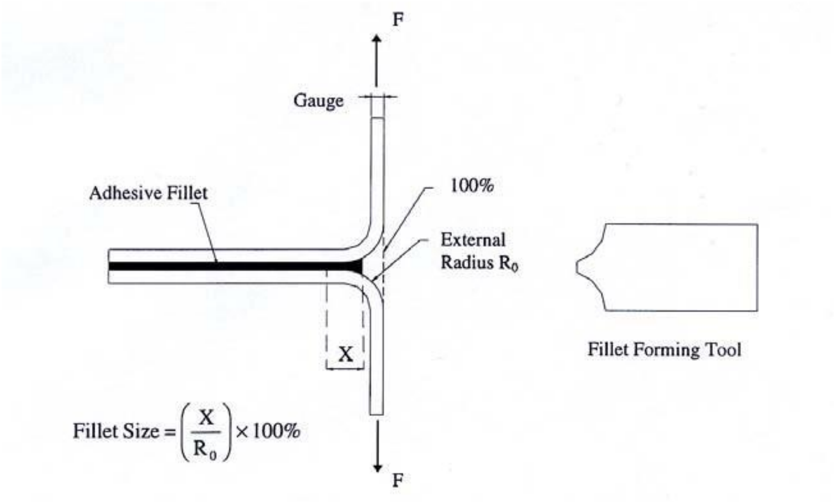

Figure 6. Definition of fillet size in T-peel joint (including fillet forming tool) [35]

Note 3: 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. It is recommended that the Tpeel joint specimen has a 50% adhesive fillet.

Several points are worth noting:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint; as shown in Figure5. The tool can be held in place using heat resistant tape. Figure 6 shows a tool that was used to produce a consistent fillet for T-peel joints bonded with a paste adhesive. The tool can be fabricated from either aluminium or stainless steel coated with release agent.
- Controlling fillet or spew geometry is not always possible as a number of adhesives undergo minimal flow during cure (e.g., flexible adhesives). The high viscosity associated with flexible adhesives prevents adhesive flow, thus making it difficult to control the fillet geometry.
- Ideally, the fillet geometry should be kept constant when producing comparative mechanical property data for different adhesive/adherend systems.
- Strengths of flexible adhesive lap joints remain fairly constant for fillet lengths in excess of 2mm, and therefore for consistency, large fillets should be allowed to form.
- Care needs to be taken to ensure no adhesive is removed from inside the bond area when removing excess adhesive from the joint prior to cure. Removing adhesive from inside the joint will result in localised debonding and poor joint performance.

- Avoid removal of adhesive spew from the ends of joints after cure, as there is the possibility of damaging the joint. It may be convenient to remove spew from the specimen sides to provide a straight edge for aligning in a test machine. This can be achieved using emery paper.

The use of tapered or bevelled external scarf and radius fillets at the bond-line ends will reduce peel and shear stresses induced by eccentricity in the loading path (see Figure 7). These additional features will add considerably to the costs of specimen manufacture.

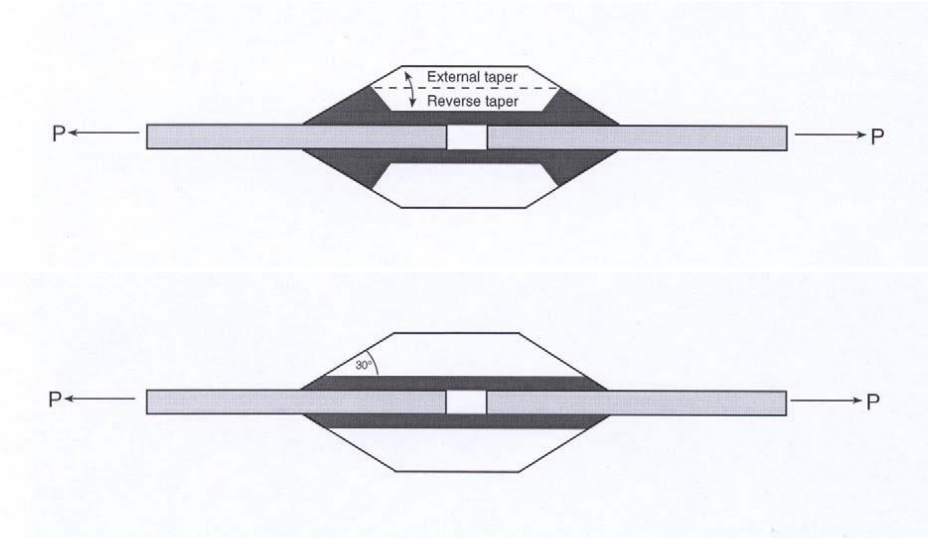

Figure 7. Tapered (bottom) and bevelled (top) strap joints

Bond-line thickness

Bond-line thickness needs to be accurately controlled (i.e., uniform adhesive layer thickness across the entire bonded area) to obtain consistent and reliable joint strength. Also, the method used to control bond-line thickness must not introduce voids or promote void formation in the adhesive otherwise the joint performance will be compromised. It should be noted that the thicker the bond-line the higher the risk of incorporating a high level of voids. In addition, stresses at the corners of the joint tend to be larger as it is difficult to maintain axial loading with a very thick bond-line. Thick adhesive layers can change the cure properties producing internal stresses, thereby reducing short and long-term performance. Conversely too thin a bond-line can result in adhesive starvation and debonding. Optimum bond thickness will depend on the type of adhesive used.

Control of bond-line thickness can be achieved by mechanical means (i.e., separation of adherends physically controlled by the bonding fixture), using thin wire spacers (e.g., stainless steel) inserted between the adherends or by ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1% by mass). Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness. It is essential that wire spacers used to control bond-line thickness are located well within the bonded area away from the specimen edges and regions of high stress concentrations (i.e., ends of joints).

When using glass beads to control bond-line thickness, the distribution of glass beads in the adhesive must be uniform and therefore glass beads should be thoroughly mixed into the adhesive. Mixing should take place before applying the adhesive to the adherend surfaces. Controlling the bond-line thickness of flexible adhesives joints is difficult due to the highly viscous nature of flexible adhesives. The preferred method is to use thin wire spacers, however work carried out at NPL showed that this method of bond-line control was not always reliable.

Curing adhesive

There are several key points that should be considered when curing adhesive joint specimens [10, 12, 14-17]:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint; as shown in Figure5. The tool can be held in place using heat resistant tape. Figure 6 shows a tool that was used to produce a consistent fillet for T-peel joints bonded with a paste adhesive. The tool can be fabricated from either aluminium or stainless steel coated with release agent.
- 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. Visual inspection should be carried out to ensure there is no air entrapment.
- The cure state of the adhesive layer in the adhesive joint should be similar to that of bulk adhesive specimens [14-15]. Failure to achieve similar thermal histories can result in significant differences in material properties. Differences between thermal histories will lead to differences in mechanical properties (See Chapter 2 [12]).
- Temperatures in the adhesive should be monitored throughout the cure cycle. It is recommended that trials be carried out on the adhesive joint using a thermocouple embedded in the adhesive in order to ensure that the temperature within the adhesive layer actually reaches the specified cure temperature.
- Due to differences in thermal mass, joint specimens may heat at different rates than bulk test specimens and therefore the final temperature of the adhesive joint at the end of the cure period can be significantly different to that of the bulk adhesive.

- For heat curing systems, the temperature of the specimen will lag behind the oven temperature, and it may therefore be necessary to elevate the oven temperature when curing joint specimens.
- Adhesives should be fully cured prior to conditioning and testing otherwise an 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, thus causing heat damage.
- Residual thermal stresses may be generated as a result of non-uniform (rapid) cooling, resin shrinkage and thermal expansion coefficient mismatch between the adhesive and adherend. As the joint is cooled down from the cure temperature, residual stresses are frozen in the material.
- As mentioned in Chapter 2, handling adhesives can be hazardous to human health, thus COSHH procedures should be followed to minimise operator exposure. Ovens and work areas should be suitably ventilated, ensuring minimal levels of hazardous vapours/gases in the work area.

It is recommended that the quality documentation should include details on the cure variables (i.e., temperature, pressure, heating and cooling rates and dwell times), and a record of equipment used for curing the adhesive joints and monitoring the temperature within the oven and adhesive joint (i.e., oven type and thermocouples).

Real-time monitoring of material property development in adhesives can be achieved using oscillatory rheometry or ultrasonic methods. Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA), can provide useful information relating to adhesive composition and final state of cure [17, 36-37] – see also NPL Measurement Good Practice Guide No. 32 [38].

Differential scanning calorimetry (DSC)

DSC is used to determine the heat flow associated with material transitions as a function of time and temperature or changes in heat capacity using minimal amounts of material. The technique provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions caused by phase changes, melting, oxidation and environmental degradation. The technique involves slowly heating a small sample (typically 5 mg to 10 mg) of material and measuring the heat absorbed or emitted by the sample as a function of temperature compared to a reference material. ISO 11357 [36] specifies methods for the thermal analysis of polymeric materials, including composite materials using DSC.

DSC can be used to measure the lowering of T_g with increasing moisture content for neat resins (including adhesives) and fibre-reinforced polymer composites. The technique has many advantages, including:

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

The disadvantages of DSC include:

- Incorrect transition may be assigned to T_g
- T_g is not detectable for all materials

Heating rates of 10 °C/minute or 20 °C/minute are typically used for T_g determination [38]. The reported T_g is generally independent of heating rate, however this is not the case for the first order transitions. A higher heating rate will broaden such transitions (e.g., curing or melting), which will reduce the accuracy of analysis.

Dynamic mechanical analysis (DMA)

DMA (or dynamic mechanical thermal analysis (DMTA)) enables the determination of transition temperatures, storage modulus and loss modulus of the sample over a wide range of temperatures (-150 °C to 800 °C), frequencies (0.01 to 200 Hz) or time, by free vibration, and resonant or non-resonant forced vibration techniques (ISO 6721 [37]). Flexural samples are popular, whereby the sample is mounted on a clamp and then subjected to sinusoidal changes in strain (or stress) while undergoing a change in temperature. DMTA is suitable for adhesives and laminated composites with stiffness values ranging from 1 kPa to 1,000 GPa.

Tension, compression, and flexure loading configurations can be employed. Three-point flexure specimens are typically 40 mm in length, 10 mm wide and 2 mm thick. Specimens can be up to 50 mm in length, 15 mm wide and 7 mm thick. DMTA can be used to measure heat distortion temperature, and thermal expansion and contraction (i.e., coefficient of thermal expansion) under dynamic or isothermal heating conditions. Thermal transition behaviour can be used as a quality assurance technique for determining the extent of resin cure.

The technique has many advantages, including:

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

The disadvantages of DMTA include:

- Poor reproducibility of modulus
- Each test is relatively time consuming

The selection of heating rate will have a significant influence on the results from DMTA [38]. The recommended heating rate is 3 °C/minute for routine testing. A lower heating rate reduces temperature variations within the specimen, while a higher heating rate allows quicker testing of specimens. It is difficult to make general recommendations for the applied strain on the specimen. The major requirement is that the strain should be in the linear elastic region for the material. Ideally, this should be determined for the material by determining the modulus at a range of strains and determining the elastic limit from the results. In practice, a strain of less than 0.5 % should be suitable for most materials.

General comments on thermal analysis techniques

The analysis of material by any 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 in a thermal analysis technique. 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. If the difference in T_g is greater than 5 °C between the first and second runs, then a third run is recommended. It is important that the material used for DSC and DMTA samples is representative of the adhesive within the bonded structure. The suggested method for preparing samples is to place the adhesive between two flat, smooth, parallel metal adherends (i.e., stainless steel), which have been coated with a thin layer of lubricant or release agent to prevent adhesion.

Quality assurance

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 (see [9]). 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 techniques are available for the non-destructive inspection of adhesive joints:

- Ultrasonic C-scan
- X-radiography
- Thermography

Note 4: There is no NDE technique that can provide a quantitative assessment of joint strength. Ultrasonic C-scan technique is particularly suited to the detection of planar type defects (e.g., debonds and delaminations) normal to the incident beam. Voids and porosity in the adhesive and adherends are also detectable. The minimum size of voids and delaminations detectable with C-scan is approximately 2 mm. The technique is not suitable for detecting surface contaminants (e.g., oils and grease).

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

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

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

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

Specimens should be stored in a desiccator (unless otherwise specified) to prevent moisture ingress, which can degrade the adhesive joint. Adhesive joints have been known to deteriorate under standard laboratory conditions (10% strength loss within 3-6 months). It is recommended that an accurate record be maintained for all batches of adhesive joints.

Documentation should include details on date of manufacture, precursor materials (i.e., adhesive and adherends), description of method of joint fabrication and assembly (including processing variables - curing time, temperature, and pressure), surface preparation and specimen dimensions (including width, bond length, adhesive layer and adherend thickness).

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Chapter 5

Preparation and assembly of adhesive joints

- Test Machine and specimen alignment
- Gripping of specimens
- Strain and displacement measurement techniques
- Mechanical testing
- Cyclic loading
- Creep
- Environmental conditioning and testing
- Conclusions

Introduction

This chapter considers the affect of test parameters (i.e., test machine alignment, load train stiffness, methods of gripping test machines, accuracy of load and displacement transducer) on the accuracy and reliability of strength and long-term performance of adhesive joints. Guidance is provided on the main factors that need to be controlled when carrying out mechanical testing. Consideration is given to various loading modes (i.e., static, cyclic fatigue and creep) and environmental conditions (i.e., elevated humidity and temperature). Appendix 1 provides a summary of commonly used adhesive joint test methods and related standards [13].

Test machine and specimen alignment

The test machine should have high lateral rigidity and accurate alignment between the upper and lower gripping faces. The load train should be as short and as stiff as possible (i.e., no universal joints included). If the grips are articulated, as in the case of universal joints, then the specimen may be subjected to large bending and twisting loads, resulting in reduce joint strength. Avoid eccentric acting forces. Small lateral (1 to 2 mm) or angular (1 to 2 degrees) offsets in the loading train can lead to additional shear and bending stresses, resulting in premature joint failure. It is worth noting that the slope of the load-displacement response can be similar for poor and well-aligned specimens.

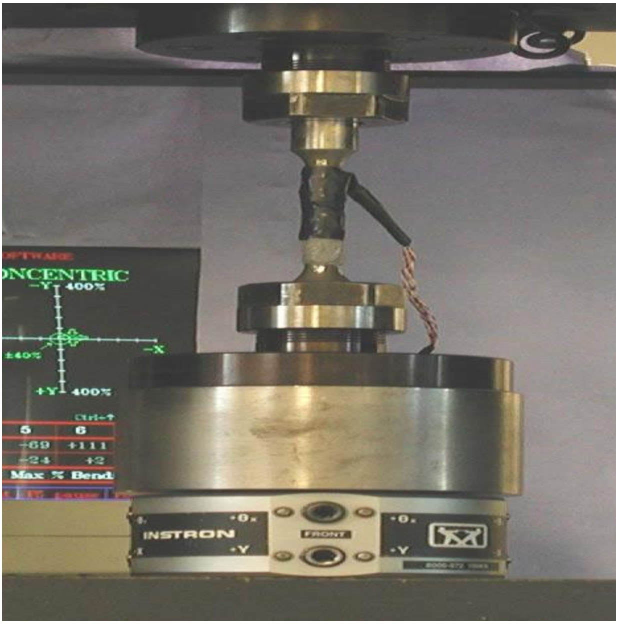

Figure 8. Alignment specimen with strain gauges

It is recommended that the alignment of the test machine and the test specimen be checked at the centre of the gauge length using a strain gauged coupon specimen [39-40]. Alignment specimens can be in the form of a rectangular or circular bar (see Figure 8). These specimens need to be accurately machined to ensure errors in parallelism are < 0.2 mm/m and in concentricity (lateral offset) of 0.03 mm [39]. Strain gauges are bonded to the surface of the alignment specimen to monitor alignment and bending strains. Bending strains should be less than 3 to 5% of the average axial strain.

Use a device to ensure that the specimens are positioned in the grips in a repeatable manner. An alignment fixture can also be included in the loading train to minimise angular and lateral offset between the upper and lower machine grips or loading rods. The alignment cell is attached to the upper or lower crosshead of the test frame; whichever is the most convenient. Commercial alignment cells are available that allow lateral movement, tilt and rotation of the machine grip or loading rod (see Figure 8).

Gripping of specimens

Grips for holding test specimens to be loaded in tension should be attached to the test frame so that the major axis of the test specimen coincides with the direction of pull through the centreline of the gripping assembly. The centre line of the specimen should be aligned with the axis of the loading fixtures to avoid bending and asymmetric loading. It is important than when loading test specimens in the grips that no lateral or angular offset is introduced to the specimen. Avoid rotating the grips during gripping operation. If one of the grips is articulated, this should be tightened first to prevent the specimen being subjected to large bending and twisting loads during tightening. Care should be taken to avoid axially stressing the specimen whilst the grips are being tightened. Any pre-stressing of the specimen should be kept to a minimum. Grips should be slowly tightened with any induced loads removed by progressively adjusting the crosshead position. The applied load on the specimen should be zero at the onset of testing. It may be necessary to use a device (i.e., metal spacer) during the test set-up to ensure good alignment and repeatable test results, as often the specimen width is less than the width of the mechanical grips.

Manual or servo-hydraulic grips can be used to hold specimens during testing. Wedge-action grips are recommended as the lateral force (i.e., pressure) applied to the test specimen in the gripping regions increases as the axial load applied to the specimen increases. Gripping pressure should be sufficient to prevent specimen slippage throughout the duration of the test, but not excessive to initiate failure of the specimen at the grips. For cyclic loading, it is essential that fretting in the gripped region be prevented to avoid the possibility of premature failure.

Strain and displacement measurement techniques

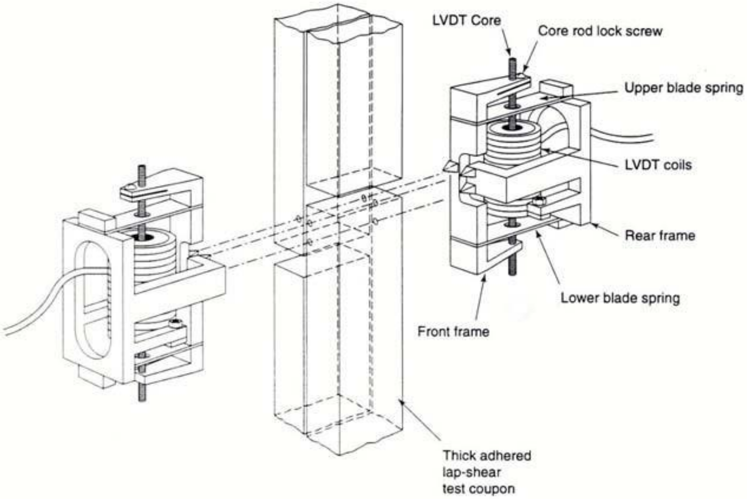

Figure 9. TAST specimen with extensometers for measuring shear deformation

Several contact and non-contact techniques are available for measuring strain and displacement. This section considers the use of contact extensometers, video extensometers, electronic speckle pattern interferometry (ESPI), strain gauges and crosshead movement for measuring strain and displacement under ambient and hostile environments, and static and cyclic fatigue loading conditions [10, 19]. Apart from strain mapping techniques (i.e., ESPI), the measured strain will be an average strain in the bond-line. Also, strain gauges only measure strain at the location of the gauge.

Contact extensometers

Contact extensometers are the preferred method for measuring strain and displacement, and hence stiffness of bonded joints. It is recommended that two extensometers, attached to opposite faces of the specimen, be used to measure displacement [10]. Any bending of the specimen will be apparent from diverging displacement readings. It is recommended that the individual transducer readings be recorded so that the quality of the test data can be checked. Errors due to minor bending are minimised by taking the average measurement of the two displacement transducers. To minimise inclusion of adherend deflection in the measurement the contact points should be as close to the bond layer as possible.

Figure 9 shows two extensometers attached to a thick adherend shear test (TAST) specimen for measuring shear deformation. The three-point contact minimises rotation of the extensometers. Knife-edged tensile extensometers, as described in reference [10], can be used if extensometer straddles the bond-line. The deformation of the adherends needs to be accounted for when analysing the data, but where the stiffness of the adherends is very much greater than that of the adhesive layer then corrections may be minimal.

Where adherends are flexible, it is advisable to support the weight of the extensometer because allowing the extensometer to hang unsupported from the specimen may cause bending and introduce contact stresses. The contact forces should be sufficient to prevent slippage between the extensometer and the specimen, but not large enough to cut or nick the specimen surface causing the specimen to fail prematurely. It may be necessary to remove extensometers attached to a specimen prior to failure to prevent the possibility of the extensometer sustaining damage during failure. Failure can be a violent event, releasing considerable energy, thereby damaging, or even destroying the extensometer.

An extensometer should be capable of measuring the change in gauge length with an accuracy of 1% of the applied displacement or better (i.e., equivalent to ± 0.5 μm for 10 % strain over a typical bond thickness of 0.5 mm). It is important that the extensometers can operate satisfactorily within the test environment (i.e., temperature and humidity), and that these devices are resistant to chemical attack when used in hostile environments. Precautions may need to be taken to insulate the leads to prevent moisture ingress.

Non-contact extensometers

Non-contact or optical extensometers (e.g., video extensometers) are available, which avoid contact damage and can be used up to failure, since there is no possibility of damage to the extensometer. Video extensometers are not particularly suited to measuring small strains (e.g., movements of a few μm), which limits their applicability to structural adhesive joints. Furthermore, measurement is normally only possible at one side of the joint so that bending cannot be evaluated. However, video extensometers have been used in tests on joints bonded with flexible adhesives where deflections are larger. Measurements of joint stress-strain curves have been in reasonably good agreement with contact extensometer results. Some systems provide capabilities for dot location measurements (facilitating strain mapping).

The technique relies on a remote camera monitoring the separation of two marks or lines inscribed on the test specimen, which define the gauge length. The change in separation of the two lines is recorded throughout the test. The gauge marks should be approximately equidistant from the mid-point, and the measured distance between the marks should be measured to an accuracy of 1%, or better. Gauge marks should not be scratched, punched, or impressed on the specimen in any way that may cause damage to the specimen. It is advisable to ensure that there is a sharp contrast in colour between the specimen surface and the gauge marks. The lines should be as narrow as possible. There are no temperature restrictions as video extensometers can be located outside the test chamber.

Strain gauges

Currently there are no standard tests using strain gauges to monitor strain in the adhesive layer. However, structural monitoring capabilities, where strain-sensing devices are embedded in materials, are the subject of research in many organisations. Strain gauges can be attached to adherends and will measure the strain in these adherends. The usefulness of such measurements may be limited except in the cases where changes in joint performance are manifested in measurable changes in the adherend strain. One such application is back-face strain gauging of thin lap-shear joints where crack growth in the adhesive layer can be monitored through strains measured by gauges bonded to the external surface of the adherends at the overlap. Strain gauges are occasionally used for monitoring strain in bonded structures (e.g., four-point bending of sandwich structures).

Strain gauges are generally limited to the measurement of strains less than 10%. Biaxial rosettes are available for measuring longitudinal and lateral strains. Large strain gauges are preferable as alignment and handling is easier, and they average out local strain variations. Local strain variations can cause premature failure of the strain gauges. Correct alignment of strain gauges is important, as significant errors can be caused by careless application of the strain gauges to the specimen. Errors of 15% can occur from a 2° misalignment [41].

The adhesive used to bond the strain gauge should be capable of withstanding the test environment for the complete duration of the test [11]. Most adhesives are sensitive to moisture (and other chemicals), which can often preclude bonding prior to specimen conditioning. Moisture attack of an adhesive and strain gauges will occur from the top, edges and in the case of polymeric materials through the substrate beneath the gauge. The situation is exacerbated at elevated temperatures. It is therefore important to ensure that the adhesive selected for bonding the strain gauge and associated electrical wiring is suitably encapsulated.

The strain gauges are usually bonded to the specimen following moisture conditioning (i.e., immersion in water or exposure to humid environments). However, bonding the strain gauge to the specimen may require heat and pressure, which will induce drying out of the conditioned specimen. To avoid drying out, room temperature curing anaerobic adhesives have been used and have proved satisfactory for bonding strain gauges to moisture conditioned specimens. For hot/wet conditions, a high temperature anaerobic adhesive can be used provided the application temperature does not result in thermal damage to the adhesive joint (i.e., adhesive and adherend). Although anaerobic adhesives have good moisture, solvent and temperature degradation resistance, these adhesives are known to attack certain plastics. Hence, precautions need to be taken when selecting these materials for use with plastics or fibre-reinforced polymer composites. Cyanoacrylates (or super glues), which are sensitive to surface moisture and low pH levels, are unsuitable for environmental testing. Strain gauge manufacturers can provide information on adhesive selection and procedures for protecting strain gauges.

For cyclic loading, it is essential that the fatigue life of the strain gauges, over the operating strain levels, should be well more than the life expectancy of the test component. Autogenous (self-generated) heating can degrade the mechanical properties of the adhesive bond between strain gauges and the specimen. This can result in small errors in strain measurement, thus requiring correction of the data to account for the temperature rise. Measurements should also be carried out to determine the magnitude of creep within the strain gauge adhesive.

Crosshead displacement

An approximate measurement of strain and hence stiffness can be obtained from measuring the crosshead displacement of the test frame [10]. The strain is the ratio of crosshead displacement and the initial grip separation. Hence, any slippage within the loading train will produce errors in the strain measurement. The strain values obtained from crosshead measurements will differ from the actual strain in the central region of the specimen.

Stiffness measurements directly obtained from the crosshead movement need to be corrected account for the stiffness of the loading train. This can be a difficult task as the specimen size and geometry, and the deformation behaviour of the specimen need to be considered [14]. Given the small adhesive layer deflections that occur even at large strains owing to the thin bond-lines, the accuracy of strains determined using crosshead displacements must be considered suspect and used only for qualitative purposes.

Electronic speckle pattern interferometry (ESPI)

Electronic speckle pattern interferometry (ESPI) is a non-contact technique capable of measuring and monitoring non-uniform strain fields at high resolution. The system can measure the deformation and thus the strain under mechanical and/or thermal loads along the three material axes (i.e., 3-D strain measurement). ESPI systems can measure local deformation with a resolution of 0.1 μm, equivalent to 200 microstrain for a 0.5 mm thick bond. The technique needs minimal specimen preparation and can inspect areas ranging from 25 mm^2 to 600 mm^2, but capital outlay for equipment is generally prohibitive for most test facilities. The technique can be used to measure strain distributions in complex geometries, and for checking finite element analysis. Further details of the technique with illustrated case studies are given in reference [42]. Interferometry techniques are not routine and are thus unlikely to be suitable for mass screening programmes. Similarly, the technique may not be suitable for cyclic testing.

Mechanical testing

The strength or stiffness measured in mechanical tests form only part of the useful data that can be obtained. The mode of failure (i.e., cohesive failure in the adhesive or interfacial failure) and degree of cohesive and adhesive failure should be recorded for each specimen. Optical microscopy or scanning electron microscopy may be required to analyse the fracture morphology, particularly if failure is close to the adhesive-adherend interface. Standards often recommend that results from specimens where failure occurs in the adherend should be discarded and replaced. However, unless the reason for adherend failure can be traced to inherent flaws in the test adherend that are unlikely to be replicated in the bonded component then these tests can still provide some valuable information.

The effects of test environment need to be considered. The operator should ensure that the equipment used to load and monitor (i.e., extensometers and load cell) the specimen is unaffected by the test environment. It may be necessary to thermally insulate load cells and use molybdenum grease to ensure moving parts in test fixtures do not seize whilst testing. It is recommended that the loading fixtures be fabricated from stainless steel to avoid environmental attack.

Number of test specimens

A minimum number of five specimens should be tested for each batch of specimens. If a greater precision of the mean value is required, then the number of specimens tested should be increased (see ISO 2602 [43]).

Specimen dimensions

Specimen dimensions and crack length need to be accurately measured, as small measurement errors can translate into large variations in strength or fracture toughness, particularly if the calculation includes squares or cubes terms of the measured parameter (see Table 3). The uncertainty in strength or fracture toughness calculation is compounded where there is more than one term (i.e., width, thickness, and crack length, etc), each with an associated uncertainty. The bond line thickness, being a very small dimension, tends to be the dimension where accuracy and precision of dimensional measurement are most critical. Measurements at different locations should be carried out to check the uniformity of bond-line thickness.

Vernier callipers or travelling microscope are recommended for measuring specimen width and bond length, and a micrometer or travelling microscope for measuring specimen thickness. A travelling microscope should be used to measure crack length.

Dimensional Error	Linear Error	Squared Error	Cubed Error
± 1	± 1	± 2	± 3
± 5	± 5	± 10	± 16
± 10	± 10	± 21	± 33

Table 3. Associated uncertainty with measurement error (percentage)

Speed of testing

Polymeric adhesives are viscoelastic materials; that is their mechanical properties (strength and stiffness) are sensitive to the rate at which they are loaded (or more accurately the strain rate). Standards relating to testing of adhesive joints infrequently specify the speed or rate of testing required, but instead specify that the test joint be loaded at a constant stress or strain rate and to ensure that failure is achieved in a prescribed period (typically 60 to 90 seconds). This introduces a degree of subjectivity into the selection of test conditions. Where specimens have different bond-line thickness then strain rates may vary between specimens leading to greater uncertainties in the results. For comparative measurements, it is recommended that all joints be tested at comparable strain rates. This can be achieved by ensuring the ratio of test speed over bond thickness is approximately the same for each test specimen. The standard requirement to fail the joint in the prescribed time is convenient for testing but may not impose strain rates relevant to the design requirement.

A series of trials to failure are recommended to ascertain the test speed required to meet the strain rate or time limit specified in the standard. It is therefore advisable that 4 or 5 additional specimens be prepared with each batch of specimens for this purpose. It should be noted that the small gauge length, due to the thin bond line, leads to relatively high rates of strain in the adhesive at moderate test speeds. This needs to be considered when comparing joint specimen tests with bulk specimen properties.

Adherend property and geometric effects

Altering the geometry of a bonded joint will invariably cause changes to occur in the stress and strain distribution within the adhesive layer. These differences can have a profound effect on the stress concentrations and consequently the load-capacity and long-term performance of the joint. Currently, there are no well-established design procedures for predicting failure behaviour or relating changes in material and geometric parameters to joint strength. Finite element analysis (FEA) enables the prediction of the effects of changing joint geometry parameters on stress/strain levels in the structure. Thus, joints can be designed to minimise stress concentrations. With accurate material properties data, relevant materials models, and reliable failure criteria the strength of any joint under any stress state could be predicted. However, the state of the art is not at this stage yet. Research on this is continuing.

Adherend Thickness/Overlap Length	Load/Width (N/mm)
CR1 Mild Rolled Steel	
1.5 mm/12.5 mm	334 ± 11
2.5 mm/12.5 mm	354 ± 10
2.5 mm/25.0 mm	428 ± 38
2.5 mm/50.0 mm	633 ± 63
5251 Aluminium Alloy	
1.6 mm/12.5 mm	191 ± 14
3.0 mm/12.5 mm	325 ± 28
6Al-4V-Titanium Alloy	
2.0 mm/12.5 mm	457 ± 52
Unidirectional T300/924 Carbon/Epoxy	
2.0 mm/12.5 mm	369 ± 41
Plain Woven Fabric (Tufnol 10G/40)	
2.5 mm/12.5 mm	275 ± 28
2.5 mm/25.0 mm	454 ± 27
2.5 mm/50.0 mm	511 ± 32
5.1 mm/12.5 mm	327 ± 27

Table 4. Failure load per unit width for AV119 epoxy single-lap joints

Bond-line Thickness (mm)	Bond Thickness (mm)		
	12.5	25.0	50
0.31	585 ± 55	1,162 ± 21	1,988 ± 215
0.52	562 ± 36	1,344 ± 49	2,199 ± 166
0.71	498 ± 24	1,050 ± 59	1,689 ± 162
1.10	455 ± 29	880 ± 105	1,707 ± 27

Table 5. Failure load (N) for mild steel/elastomer single-lap joints

The results presented in Tables 4 and 5 clearly indicate that the joint strength is dependent on the adherend material, adherend thickness, bond-line thickness and bond length. Table 4 compares the failure load per unit width (N/mm) for various single-lap joint configurations and materials bonded with an epoxy adhesive [44-46]. Table 5 demonstrates the sensitivity of single-lap strength on bond-line thickness and bond length. The joints consisted of mild steel sections bonded with an elastomer adhesive. Small variations in bond-line thickness can result in significant changes in bond strength. For comparative studies, careful consideration should therefore be given to ensuring that the stress and strain distributions (i.e., maximum peel and shear stresses at the ends of the joint) for different systems are at least similar.

As a first approximation for single-lap joints, adherends with similar products of stiffness and thickness should form joints with similar strengths if failure is cohesive in the adhesive layer, i.e.:

$$E_1 t_1 = E_2 t_2$$

w here *E* is the longitudinal stiffness of the adherend, *t* is the adherend thickness, and subscripts 1 and 2 refer to materials 1 and 2.

For the measurement of the adhesive properties, steel adherends are recommended because of the material's high stiffness. For ambient tests, suitable steels are XC18 and 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.

Cyclic loading

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.

Creep

Creep tests are performed to assess the extension of joints under load to predict long-term behaviour or to assess the long-term strength of joints under load. The first requirement needs high precision extensometry to monitor joint extension and the tests must be performed under stable environmental conditions (temperature and humidity) to avoid artefacts in the measurement. These tests could, in theory, be performed using any of the loading options outlined below but for highest accuracy options 1 or 2 would be preferred. This section addresses the requirements of creep failure (or creep rupture tests).

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 (Figure 10); and
4. Self-stressing fixture where specimens are placed in either a tube equipped with a calibrated spring system (Figure 10) for loading specimens or a circular ring.

Figure 10. 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 10), 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. 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 10 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 tested. Typically, these are between 10 and 50% of the short-term strength of the joint. It is generally 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).

Environmental conditioning and testing

To determine the effectiveness of different adhesive systems, processing variables and surface treatments, it may be necessary to expose adhesively bonded joints to various environmental and loading conditions that simulate actual service conditions. The resistance of the bonded structure to degradation agents often becomes apparent within a short period. In some circumstances only a few hours of exposure may lead to catastrophic failure or seriously compromise the structural integrity of the joint (see also Measurement Good Practice Guide Number 28 [18]).

The two predominant factors in climatic exposure are humidity and temperature. The severity of these two factors will depend on geographical location and need to be considered in assessing allowable strengths. A list of possible service environments is listed below:

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

Engineering structures are exposed to various combinations of these environments in service, often resulting in complex synergistic degradation of the material. It is recommended that when comparing the effects of surface treatments, adhesive systems, and processing variables on the durability of joints in hostile environments, that all specimens are prepared and conditioned together to account for any variability in the conditioning environment. Also, control specimens are recommended to check improvements in joint performance.

A listing of fatigue, creep and environmental standards are presented in NPL Report CMMT(A) 61 [13] along with a summary of environmental conditioning procedures, covering the scope of the standard, relevant materials, environmental conditions, and specimen requirements. Several NPL reports [46-50] cover the use of statistical analysis for analysing durability data.

Degradation agent

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

Moisture (water) degradation is probably the major cause of in-service failure in bonded structures. The ubiquitous nature of water combined with the ability to penetrate the adhesive structure poses considerable problems. With good design it is possible to significantly reduce the rate of moisture diffusing to the adhesive/adherend interfaces. This problem is further exacerbated at elevated temperatures and mechanical stress. Hot and humid environments can often cause rapid loss of strength in metal-epoxy joints within a short duration (i.e., 2 years) with catastrophic consequences. Failure invariably occurs at the adhesive/adherend interface.

The major cause of strength loss in adhesively bonded metal joints is associated with interfacial degradation through water-substrate interaction rather than weakening of the adhesive. Water can degrade the strength of adhesive joints through hydration of metal oxide layers. Corrosion products, such as rust, at the interface are considered a post-failure phenomenon. The presence of water in epoxy adhesives results in plasticisation (essentially softening) of the polymeric material. At temperatures below the glass transition temperature T_g, polymer property reduction is reversible upon dehydration, whereas above T_g, matrix properties are permanently degraded. Surface treatments, such as phosphoric acid anodisation and organosilane primer coatings will bestow joints with improved moisture resistance.

Accelerated conditioning

The degree of degradation that occurs in adhesive structures when in service is linked directly with the amount of moisture absorbed. The moisture absorption kinetics of polymer systems will differ widely and change with physical ageing. The approach often adopted, is to define a constant relative humidity environment that will produce a moisture level that is representative of a bonded structure that has been exposed to a real-life environment at a particular geographical location. The variability of a natural environment, that is daily, monthly, or seasonal changes in temperature and humidity are known to be a major factor in determining both the final moisture equilibrium level in the material and the distribution of moisture in the outer surface layers. The military consider the worst worldwide environment to be represented by a relative humidity of 85% and a temperature of 70°C.

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

The usual approach used to accelerate moisture uptake is to increase the diffusivity of the adhesive by elevating the temperature of the conditioning environment. However, there is a temperature limit to which most polymer resins can be raised without affecting a change in the mechanism by which moisture is absorbed. Above this critical temperature, there is a strong possibility that the degradation mechanism is altered.

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

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

Note 5: Although accelerated ageing is widely used, a full understanding of the effects is not yet available and there is no agreement within industry as to a satisfactory accelerated test.

Chapter 6

Conclusions

Conclusions

The two most important criteria when selecting a test method are the availability of a standard test method and the ability of the test method to produce consistent and reliable engineering data for a range of service conditions. When planning a test strategy the following should be considered:

- Selection of appropriate test for ranking and screening, design data and QA/QC (i.e., conformance to design standards/codes) purposes.
- Knowledge of existing standards/legislative requirements.
- An understanding of the material (i.e., adherend and adhesive) characteristics, test method and the test data generated. An awareness of the mechanical and physical properties of the adherend, adhesive and the joined system are invaluable in understanding the test results and for trouble-shooting.
- Cost of fabrication and testing/test facility requirements.
- Knowledge of the factors that affect reliability of the test data.

Note 6: Many of the test methods/standards are limited to providing comparative data on adhesive systems and surface treatments, and are unsuitable for generating design data.

Independent of the test method selected, correct surface preparation is essential for ensuring both initial adhesion and long-term joint durability. Although the guide provides advice on the surface preparation of the commonly used structural materials, it only covers the main steps in each process. **It is essential that expert advice from the adhesive manufacturer is obtained and that the detailed requirements specified by the manufacturer are completely satisfied**. Special care is needed to ensure that bond-line thickness is uniform and free of voids, and that the method used to control the bond-line thickness does not compromise the strength or durability of the joint. Assume the degradation process is irreversible and commences on completion of the cure cycle.

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Appendix 1

Test methods

List of test Methods

Material Property	Standard/Test Method
Bulk Plastic/Adhesive Tests	
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
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	ISO 4587, ASTM D3528, BS 5350-C15
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 D3167
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

Adhesive joint testing

The four main loading modes of bonded joints are (see Figure A1.1):

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

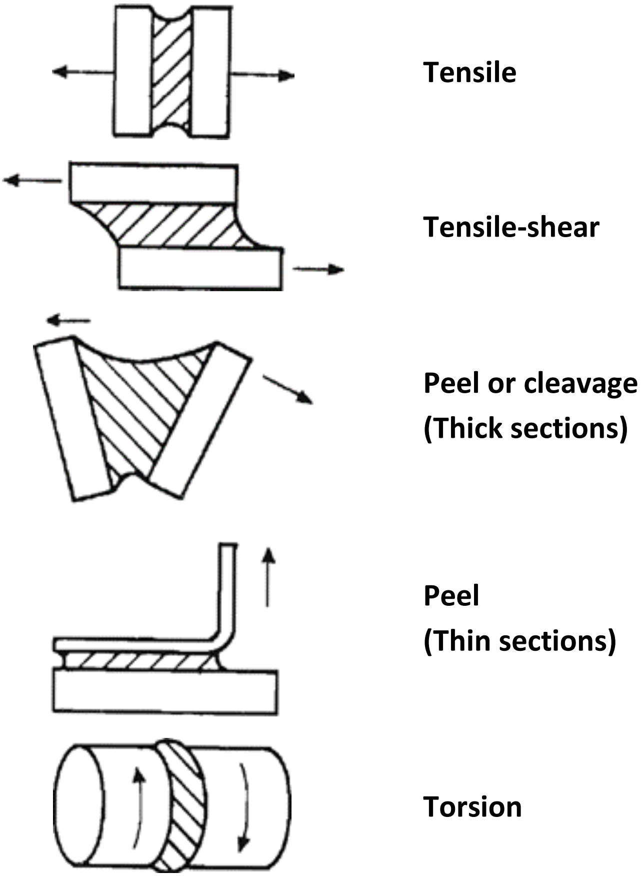

Figure A1.1. Basic loading modes experienced by adhesive joints

Tables A1.2 to A1.19 in Appendix 1 provide detail information on adhesive joint test methods.

Tension test methods

Advantages	Disadvantages
Yields tension, torsion, and compression Stiffness/strength Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for environmental and cyclic testing (QA only)	Qualitative adhesive property data only Strain measurements difficult High shear/peel stresses at bond-line edges Strength sensitive to spew fillet Sensitive to specimen misalignment Limited to thick and rigid adherends Failure attributed to peel stresses Special bonding fixture required

Table A1.2. Tensile butt joint test method (see Figure 3)

Peel test methods

Advantages	Disadvantages
Yields peel strength Straightforward/economic • Specimen fabrication • Testing • Data reduction Discriminates between surface treatments and adhesives Suitable for In-situ environmental (QA only) and cyclic testing	Limited to thin flexible adherends Not suitable for generating design data Large uncertainties in measurements No allowance for large adherend deformation Sensitive to fillet dimensions

Table A1.3. T-Peel test method (see Figures 6 and A1.2)

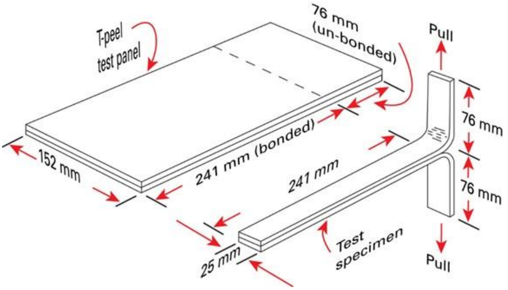

Figure A1.2. T-peel specimen

Advantages	Disadvantages
Yields • Peel torque • Skin (facing) stiffness of honeycomb sandwiches Laminated or honeycomb sandwich structures Straightforward/economic • Testing • Data reduction	Special test fixture required Limited to: • Laminated or honeycomb sandwich structures • Low stiffness skins (facings) Large and expensive specimens Comparative data/service correlation unknown Unsuitable for fatigue testing Unsuitable for in-situ environmental testing

Table A1.4. Climbing drum peel test method (see Figure A1.3)

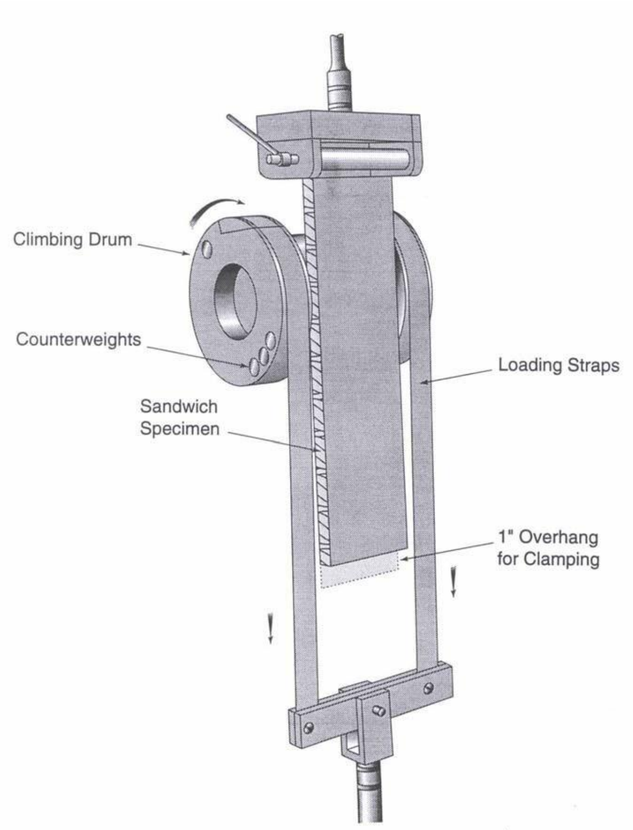

Figure A1.3. Climbing drum test of sandwich structure

Advantages	Disadvantages
Yields peel strength Compatible with metals and PMCs Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction	Special test fixture required Limited to rigid-to-flexible adherends No allowance for large adherend deformation Comparative data/service correlation unknown Unsuitable for fatigue testing Unsuitable for in-situ environmental testing

Table A1.5. Floating roller method (see Figure A1.4)

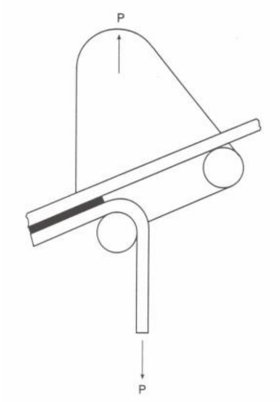

Figure A1.4. Floating roller method

Cleavage Test Methods

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

Table A1.6. Wedge cleavage or Boeing wedge test (see Figure A1.5)

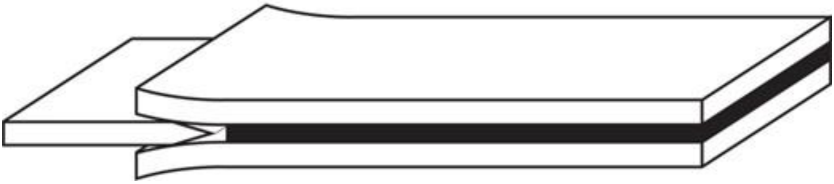

Figure A1.5. Wedge cleavage test

Advantages	Disadvantages
Yields fracture strength Compatible with metals and PMCs Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction Suitable for environmental/fatigue testing (QA only)	Special test fixture required (moderate cost) Special bonding fixture required Limited to rigid adherends Not suitable for generating design data

Table A1.7. Compact tension test (see Figure A1.6)

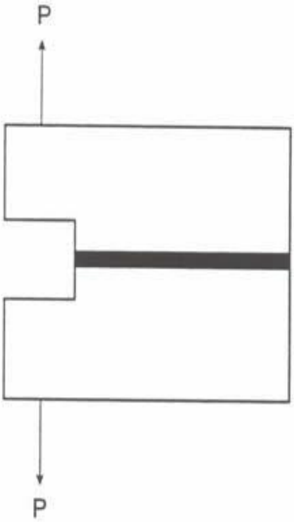

Figure A1.6. Compact tension test

Shear test methods

Advantages	Disadvantages
Yields "apparent" shear strength	Geometry dependent
Compatible with metals, plastics, and PMCs	Limited to rigid adherends
Straightforward/economic	Not suitable for generating design data
• Specimen fabrication	High shear/peel stresses at bond-line ends
• Testing	Moderate to high bending moments
• Data reduction	Failure attributed to peel stresses
Suitable for environmental and cyclic testing (QA only)	Special bonding fixture required
	Large uncertainties in measurements

Table A1.8. Single/double-lap joint tests (see Figures 2 and A1.7)

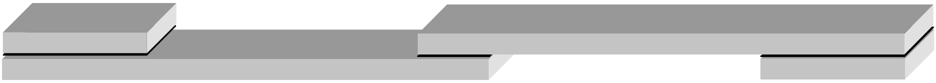

Figure A1.7. Single-lap test specimen

Advantages	Disadvantages
Yields "apparent" shear strength	Geometry dependent
Compatible with metals, plastics, and PMCs	Limited to rigid adherends
Straightforward/economic	Not suitable for generating design data
• Specimen fabrication	Elevated shear and peel stresses at bond-line ends
• Testing	Moderate to high bending moments
• Data reduction	Failure attributed to peel stresses
Suitable for environmental and cyclic testing (QA only)	Special bonding fixture required
	Large uncertainties in measurements

Table A1.9. V-notched beam shear tests (see Figure A1.8)

Figure A1.8. V-notched beam shear test

Advantages	Disadvantages
Bulk Resin Shear modulus attainable for all polymeric materials Shear strength attainable for thermoplastics **Adhesive Joints** Yields shear modulus and strength Suitable for compliant adhesives **Additional Points** Low material requirements Data reduction is straightforward Suitable for use under environmental conditions Suitable for creep testing	**Bulk Resin** Shear modulus attainable for all polymeric materials Shear strength attainable for thermoplastics **Adhesive Joints** Yields shear modulus and strength Suitable for compliant adhesives **Additional Points** Low material requirements Data reduction is straightforward Suitable for use under environmental conditions Suitable for creep testing

Table A1.10. Arcan shear tests (see Figure A1.9)

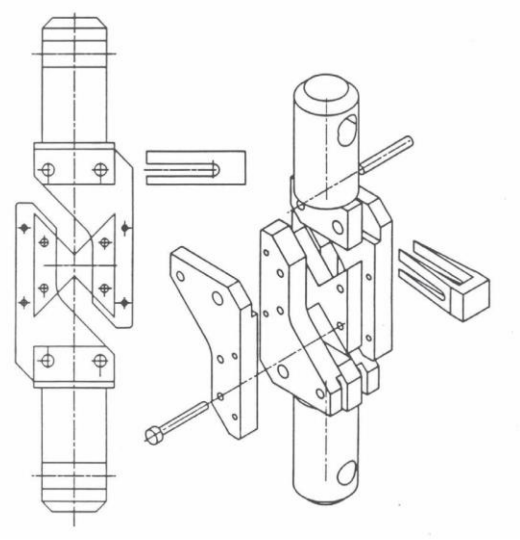

Figure A1.9. Arcan shear test

Advantages	Disadvantages
Bulk Resin	**Bulk Resin**
Shear modulus attainable	Strain gauges/extensometers required
Shear strength attainable for thermoplastics	Brittle polymers - tensile failure occurs
Adhesive Joints	Axial strains need to be monitored
Yields shear strength and shear modulus	**Adhesive Joints**
Compatible with metals and composites	Special bonding fixture required
Additional Points	Non-uniform shear stress state
Suitable for use under environmental conditions	Small strains - difficult to measure accurately
Suitable for cyclic/creep testing	**Additional Points**
	Accurate specimen machining required
	Torsion facility required
	Stress concentrations present at end grips
	Complex data reduction (non-linear stress/strain)
	No existing national or international standards

Table A1.11. Torsion butt tests (see Figure 3)

Advantages	Disadvantages
Yields shear strength and shear modulus Compatible with all metals, plastics, and PMCs Stress state relatively uniform over bond-line Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction Suitable for environmental testing	Complex/expensive extensometers (2 off) required Special bonding fixture required Accurate specimen machining required Stress concentrations present at bond-line ends Stress analysis difficult Limited fatigue capability

Table A1.12. Thick adherend shear tests (see Figure 3)

Mode I Fracture Toughness Test Methods

Advantages	Disadvantages
Yields mode I fracture toughness Compatible with metals and PMCs Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for environmental and cyclic testing (QA only)	Complex/expensive extensometers (2 off) required Special bonding fixture required Accurate specimen machining required Stress concentrations present at bond-line ends Stress analysis difficult Limited fatigue capability

Table A1.13. Double cantilever beam (DCB) tests (see Figure A1.10)

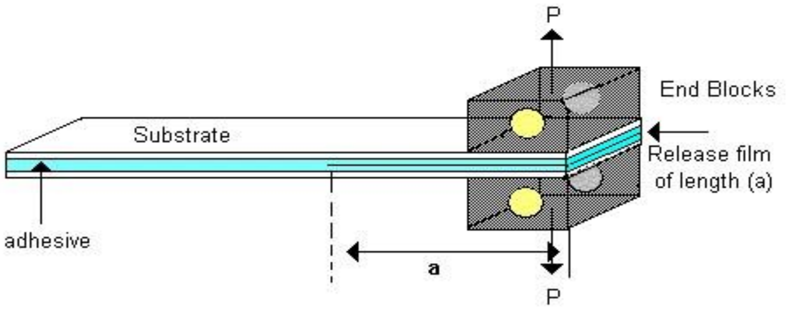

Figure A1.10. Double cantilever beam (DCB) specimen

Advantages	Disadvantages
Yields Mode I fracture toughness Compatible with metals Adaptable to thin adherends Constant compliance Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for cyclic/environmental testing (QA only)	Limited to rigid adherends/brittle adhesives Flexural test fixture required Unsuitable for generating design data Analysis required to account for • Shear deformation • Large beam deflection Non-linear load-displacement Pre-cracking difficult Crack extension measurements difficult Moderate-large measurement uncertainties No existing national/international standard

Table A1.14. Tapered double cantilever beam (TDCB) tests (see Figure A1.10)

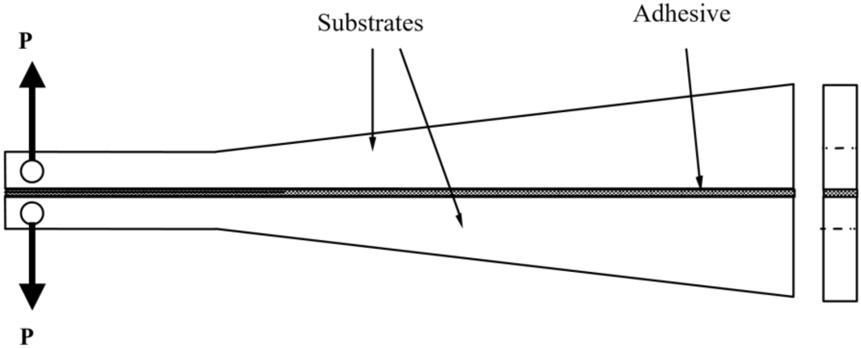

Figure A1.11. Double cantilever beam (DCB) specimen

Mode II fracture toughness test methods

Advantages	Disadvantages
Yields mode II fracture toughness Compatible with metals and PMCs Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for cyclic/environmental testing (QA only)	Limited to rigid adherends Large specimens required Not suitable for generating design data Special test fixture required Special bonding fixture required Moderate to large uncertainties in measurements

Table A1.15. End loaded split (ELS) and end notched flexure (ENF) Tests (see Figures A1.12 and A1.13)

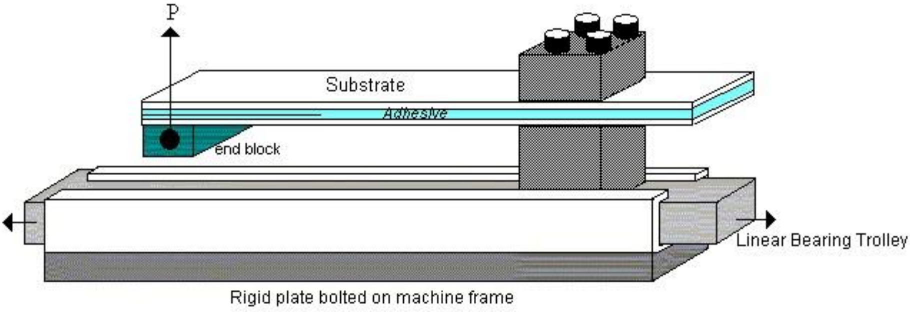

Figure A1.12. End loaded split (ELS) test

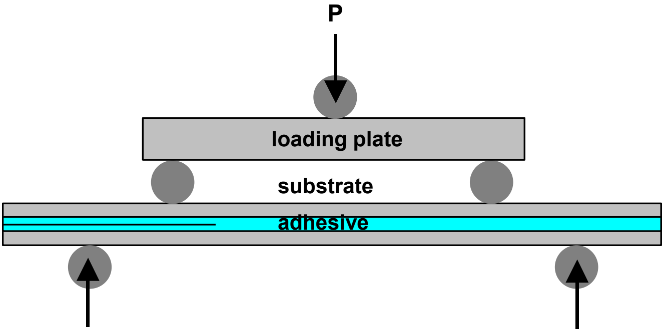

Figure A1.13. End notched flexure (ENF) test

Test Method	Tensile Butt Joint	T-Peel	Climbing Drum	Floating Roller Method
Mechanical Properties Obtained	Tensile strength/modulus	Peel strength	Peel strength/skin stiffness	Peel strength
Material Quantity Requirements per specimen	Low	Low	High	Low
Typical Specimen Dimensions (mm)	Diameter 15-25 mm Adherend thickness 12-15	Bond length 150 Width 25 Adherend thickness 0.5-1.0 Arm length 50	Long adherend 300 Short adherend 240 Width 25 Adherend thickness 0.5-5.0	Flexible adherend 250 Rigid adherend 200 Width 25 Adherend thickness 0.5-1.6
Materials Suitable for Testing	1 - 6	1, 4 and 6 Flexible-flexible adherend	Flexible-rigid adherend 1 - 6 and sandwich structures	Flexible-rigid adherend 1 - 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	High	Low - moderate
Cost of Testing/Specimen	Low	Low	Low - moderate	Low - moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Extensometer	None	None	Extensometer (2 off)
Test Equipment and Fixture Requirements	Universal test machine and end grips	Universal test machine and end grips	Special test fixture Universal test machine and end grips	Special test fixture Universal test machine and end grips
Fatigue performance	Limited	unsuitable	Unsuitable	Unsuitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Unsuitable	Unsuitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	Large uncertainty (> 30%)	To be determined	To be determined
Standards	ASTM D897 ASTM D2095 BS EN 15870	ISO 11339 ISO 8510-2 ASTM D1876	ASTM D1781 BS 5350-C13	ISO 4578 ASTM D3167

Table A1.16. Tensile and peel test methods

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

Test Method	**Wedge Cleavage**	**Compact Tension**	**DCB**	**TDCB**
Mechanical Properties Obtained	Fracture energy	Cleavage strength	Mode I fracture toughness	Mode I fracture toughness
Material Quantity Requirements/Specimen	Low	high	Low	High
Typical Specimen Dimensions (mm)	Length 200 Width 25 Adherend thickness 2	Length 25 Width 25 Adherend thickness 12	Length 356 Width 25 Adherend thickness 6.35	Length 240 Width 25 Adherend thickness 12.8
Materials Suitable for Testing	1	1 - 6	1 - 6	1 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Low	Moderate - High
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding and bonding jig	Surface preparation	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Travelling microscope or video camera	Extensometer for crack opening displacement	Travelling microscope or video camera	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Self-stressed	Universal test machine and loading fixture	Universal test machine and loading fixture	Universal test machine and loading fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	Large uncertainty	Moderate
Standards	ISO 10354 ASTM D3433	ASTM D1062 BS 5350-C1	ISO 25217	ISO 25217

Table A1.17. Cleavage and Mode I fracture toughness test methods

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

Test Method	Single-Lap	Double-Lap	V-Notched Beam	Arcan
Mechanical Properties Obtained	Shear strength	Shear strength	Shear strength/modulus	Shear strength/modulus
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 100 Width 25 Adherend thickness 2 Overlap length 25	Length 100 Width 25 Adherend thickness 12 Overlap length 25	Length 76 Width 20 Notch width 12 Adherend thickness 5	Length 52 Width 40 Notch width 12 Adherend thickness 6
Materials Suitable for Testing	1, 3 and 6	1, 3 and 6	1, 3 and 6	1, 3 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Moderate	Moderate
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	None	None	Shear extensometer Strain gauges	Shear extensometer Strain gauges
Test Equipment and Fixture Requirements	Universal test machine and loading grips	Universal test machine and loading grips	Universal test machine and loading fixture	Universal test machine and loading fixture
Fatigue performance	Limited	Suitable	Unsuitable	Suitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	Moderate	Low (30%)	Low - moderate (10-20%)	Low - moderate (10-20%)
Standards	ISO 4587 ASTM D1002 BS EN 1465 BS 5350-C3	ISO 4587 ASTM D3528 BS 5350-C13	ASTM D5379 (bulk resin)	None

Table A1.18. Shear test methods

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

Test Method	**TAST**	**Torsion Butt Joint**	**Napkin Ring**	**ENF**
Mechanical Properties Obtained	Shear strength/modulus	Shear strength/modulus	Shear strength	Mode II fracture energies
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 110 Width 5 Adherend thickness 26 Overlap length 5	Diameter 15-25 Adherend thickness 12-15		Length 356 Width 25 Adherend thickness 6
Materials Suitable for Testing	1- 6	1 - 6	1 - 6	1 and 6
Cost of Specimen Fabrication/Preparation	Low-moderate	Low-moderate	Moderate-high	Low
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Extensometers (2 off)	Rotary extensometer	Rotary extensometer	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Universal test machine	Universal test machine and loading fixture	Torsion test machine and loading fixture	Universal test machine and flexure fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	To be determined	Large uncertainty (30%)
Standards	ISO 11003-2 ASTM D5656	None	ASTM E229	None

Table A1.19. Shear and Mode II fracture toughness test methods

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

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Appendix 2

Round-robin adhesive joint evaluation exercise

Overview

A major concern with testing adhesive joints is the number of factors, not included in written standards, which can significantly affect the reliability (i.e., repeatability and reproducibility) of test data. Factors, including specimen and machine alignment, processing variables, surface treatment, adhesive storage and joint ageing need to be considered in addition to test and specimen parameters (e.g., displacement rate and dimensions) that are covered in national and international standards. It was evident from a statistical analysis of R-R data carried out in Performance of Adhesive Joints project PAJ3 on data from the ADH programme that the uncertainty associated with specimen preparation and testing has a substantial effect on the test results. The large scatter and disparity in results between test houses indicates that several additional factors need to be controlled in order to ensure reliable data. The situation is further exacerbated when tests are conducted in hostile environments.

One of the objectives of PAJex3 was to address these issues in a systematic manner in order to identify key variables. The approach was to run hands-on training sessions at the work place of participating organisations in conjunction with a R-R exercise, involving nine (9) participating organisations (including NPL). The industrial organisations, which participated in the R-R evaluation exercise, are listed below:

- Caswell Adhesives
- Corus
- Ford (UK)
- Honeywell Normalair-Garrett
- MIRA
- Permabond
- Stranger Science and Environment
- 3M (UK)

The R-R exercise consisted of three stages:

Stage 1: Six single-lap joint specimens manufactured at NPL were supplied to each organisation. The participants were requested to test the specimens in accordance with BS EN 1465 (Adhesives – Determination of Tensile Lap Shear Strength of Rigid-to-Rigid Bonded Assemblies) [33]. Permabaond supplied both the adhesive (two part epoxy E32) and bright mild steel test adherends (1.5 mm thick). Adhesive storage, handling and cure, and surface preparation were carried out in accordance with the procedures specified by Permabond (see Technical Requirements). Testing was carried out unsupervised by the participating organisations.

Stage 2: A second batch of specimens (6 off) was supplied to each participating organisation. Again, specimens were to be prepared and tested to the procedures used in Stage 1. The difference this time was that NPL personnel were present to witness the tests (i.e., observer status only) and to ask questions on in-house practices relating to the preparation and testing of adhesive joints. NPL provided a 1-hour presentation on "Good Practice for Preparation and Testing of Adhesive Joints", which was followed by a discussion.

Stage 3: Adherend and adhesive materials were supplied to each participating organisation. Members were requested to prepare and test specimens, unsupervised, to the procedures used in Stages 1 and 2.

After each stage, data and specimens were returned to NPL for statistical analysis. The results of the R-R evaluation exercise along with observations and comments from participants are presented below.

The authors would like to acknowledge the contributions of Caswell Adhesives, Corus, Ford (UK), Honeywell Normalair-Garrett, MIRA (Motor Industry Research Agency), Permabond, Stranger Science and Environment, and 3M (UK) for their contribution to the R-R exercise and to this Measurement Good Practice Guide.

Technical requirements

Each participant received a technical information sheet for Permabond E32 (two part, room temperature curing epoxy adhesive) and copies of BS EN 1465 and Permabond Test Method 9 "Determination of Shear Strength of Adhesives Using Single Overlap Joints". The technical information sheet provided with the adhesive includes a description of the two part adhesive, details on the physical properties of the adhesive (i.e., colour, viscosity, specific gravity and mix ratio of resin to hardener), recommended service temperature range, cure speed, typical lap shear strength, storage and handling instructions, and directions for use. Permabond Test Method 9 includes procedures for adherend surface preparation; test joint assembly and testing, and expression of results. In Stage 3, participants were requested to complete a form detailing surface preparation, bond-line thickness control and cure temperature (see below).

In addition, participants were requested to complete a test report for each stage of the R-R exercise. The report form identifies the test site, operator, test machine, load cell and calibration details, date of test, and temperature and humidity of the test laboratory. Operators were required to include specimen identify and details on specimen dimensions (i.e., width, overlap length and bond thickness), loading rate, maximum load, strength, mode of failure and difficulties encountered for each specimen.

Permabond test method 9

Introduction

The procedure (Determination of Shear Strength of Adhesives Using Single Overlap Joints (Issued: 30/10/00)) described below conforms to the basic requirements of BS5350: Part C5 - Single Overlap Technique. Its purpose is to ensure that adhesives conform to the specified strength requirements for their intended applications.

Apparatus

- Calibrated tensometer of suitable load capability.
- Controlled temperature environment as specified.
- Degreasing solvent (acetone, MEK, IPA) & paper towel.
- Grit blasting cabinet with 60/80 standard alumina (ex Vixen Surface Treatments) or rotary abrading table with both 100 and 600 grade 'wet and dry' discs.
- Bright mild steel test adherends of dimension 100x25x1.5 mm unless specified otherwise. (10 test pieces are required for F/V products and 6 for epoxy products).
- Suitable quantity of clean 1¼-inch fold-back clips.

Adherend surface preparation

- Examine the test adherends prior to preparation and dispose of any grossly distorted sections.
- Remove excess dirt and oil by wiping with a dry paper towel. Remove final film of oil with the aid of a solvent soaked towel.
- The end square inch of the adherends should be treated by one of the following methods:

    a) Grit blast with the air pressure set to 20 psi blast the adherends until a uniform finish is achieved.

    b) Abrade on a rotary table using firstly 100 grade 'wet and dry' followed by 600 grade to obtain a uniform, polished surface. Care must be taken to ensure the edges of the adherends are not bevelled or corners rounded during abrasion, as this will affect the ultimate performance of the adhesive under test. Immediately wipe the abraded surface dry to prevent corrosion.

- Degrease the prepared adherends with solvent and paper towel. Repeat this process if necessary to ensure all oil and contaminants are removed.

Test piece assembly

- To regulate the 12.75 ± 0.25 mm overlap, carefully mark the test-piece at the correct distance avoiding contamination of the bond area, then clearly mark the test-pieces with a suitable reference.
- Where the bond line regulation is specified, apply two pieces of wire to the overlap areas of the adherends and secure with the adhesive tape on the reverse side of each adherend.
- Apply the sufficient adhesive components in the specified manner to the surface of the adherends.
- Standing the adherends on their longest edges on top of a sheet of polythene placed on a flat surface, bring the adherends together with light finger pressure, to form the specified overlap.
- Without lifting from the surface or moving the assembly excessively, apply a 'foldback' clip centrally to the overlap area to secure it during the early stages of cure. A second 'fold-back' clip may be applied should it be necessary to move the test assembly to the specified curing environment. It is important that the bonded adherends are prevented from excessive relative movement during this transfer stage and the early stages of curing.
- After the bonded adherends have cured for the specified period, place the test-pieces in the environment where they are to be tested. Allow time for the temperature equilibration.

Testing

- Inspect all test assemblies carefully and discard those that are excessively misaligned or those, which do not have their bond area completely, filled.
- Set up a tensometer with wedge grip jaws and test each assembly with a jaw separation rate of 6 mm/minute. Record the maximum breaking load.

Expression of results

- Examine the faces of the separated adherends for irregularities and check overlap length. Further test assemblies will need to be constructed to replace those rejected.
- The value for shear strength should be calculated as follows:

  a) Calculate the mean of the breaking force in kilo Newtons (kN) to three significant figures.

  b) Express this mean in Mega Pascals (MPa) by dividing it by the overlap in square millimetres, i.e.:

$$\frac{kN \times 1000}{Area\ in\ mm^2} = MPa\ (expressed\ to\ 1\ decimal\ place)$$

For the standard overlap, multiply the value in kN by 3.137 to convert to MPa

c) Measure the spread of results by calculating the coefficient of variance (CoV). The CoV is simply the standard deviation divided by the mean.

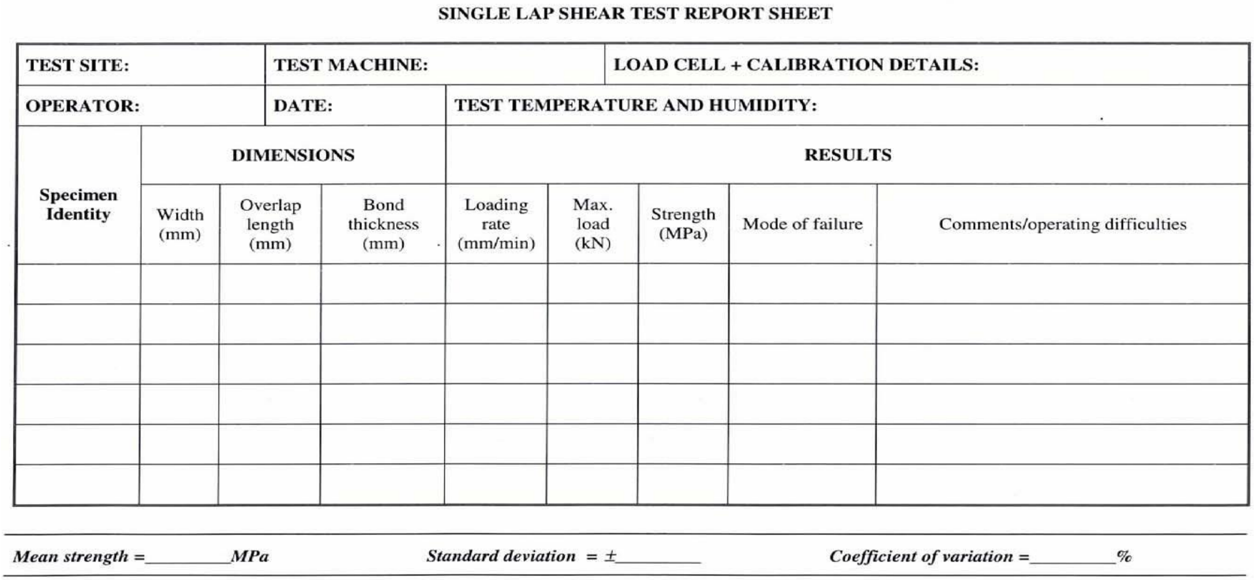
Stage 3: Specimen preparation details

In Stage 3, participants were requested to provide details on the following

Method of adherend/end tab abrasion	
Type of solvent used for degreasing	
Method of specimen alignment during cure	
Cure temperature	
Weight of ballotini balls used	

Single lap shear test report

The following data sheet was completed for each stage of the R-R evaluation exercise.



SINGLE LAP SHEAR TEST REPORT SHEET

TEST SITE:

TEST MACHINE:

LOAD CELL + CALIBRATION DETAILS:

OPERATOR:

DATE:

TEST TEMPERATURE AND HUMIDITY:

Specimen Identity	DIMENSIONS			RESULTS				
	Width (mm)	Overlap length (mm)	Bond thickness (mm)	Loading rate (mm/min)	Max. load (kN)	Strength (MPa)	Mode of failure	Comments/operating difficulties

Mean strength =_________*MPa*

Standard deviation = ±_________

Coefficient of variation =_________%

Test results and discussion

The results of the R-R programme on testing of adhesive joints, which were analysed according to ISO 5725-1 [51], clearly showed a need for formalised guidance on manufacturing and testing of adhesive joints. A summary of the results for Stages 1, 2 and 3 is presented in Tables A2.1 and A2.2, and Figure A2.1 with the individual results from each participant given in Tables A2.3 to A2.5 (end of Appendix 2). The R-R exercise has shown that the most crucial steps in the manufacturing process were:

- Surface treatment of the adherends.
- Adhesive preparation (i.e., mixing and adding ballotini glass balls).
- Joint assembly (including clamping).
- Cure conditions.

Note A2.1: The level of training, experience and motivation of technical staff can play a significant role in the quality of test results.

Stage	Repeatability Conditions		Reproducibility Conditions		Mean Results
	S_r	r	S_R	R	
1	7.2	20.2	7.5	21.1	6542
2	5.5	15.4	7.5	21.1	6652
3	11.5	32.2	34.2	95.6	5160

Table A2.1. Repeatability, reproducibility and mean values for stages 1 to 3

Definitions of *Sr*, *r*, S_R and *R* taken directly from reference [51].

Repeatability value *r*: The value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie within a probability of 95%. Repeatability refers to variance of test results within a batch of specimens tested at a single site.

Reproducibility value *R*: The value below which the absolute difference between two single test results obtained under reproducibility conditions may be expected to lie within a probability of 95%. Reproducibility refers to variance between test results from different sites where the specimens are prepared from the same material and tested to the same procedure.

Repeatability standard deviation S_r: The standard deviation of test results obtained under repeatability conditions.

Reproducibility standard deviation S_R: The standard deviation of test results obtained under repeatability conditions.

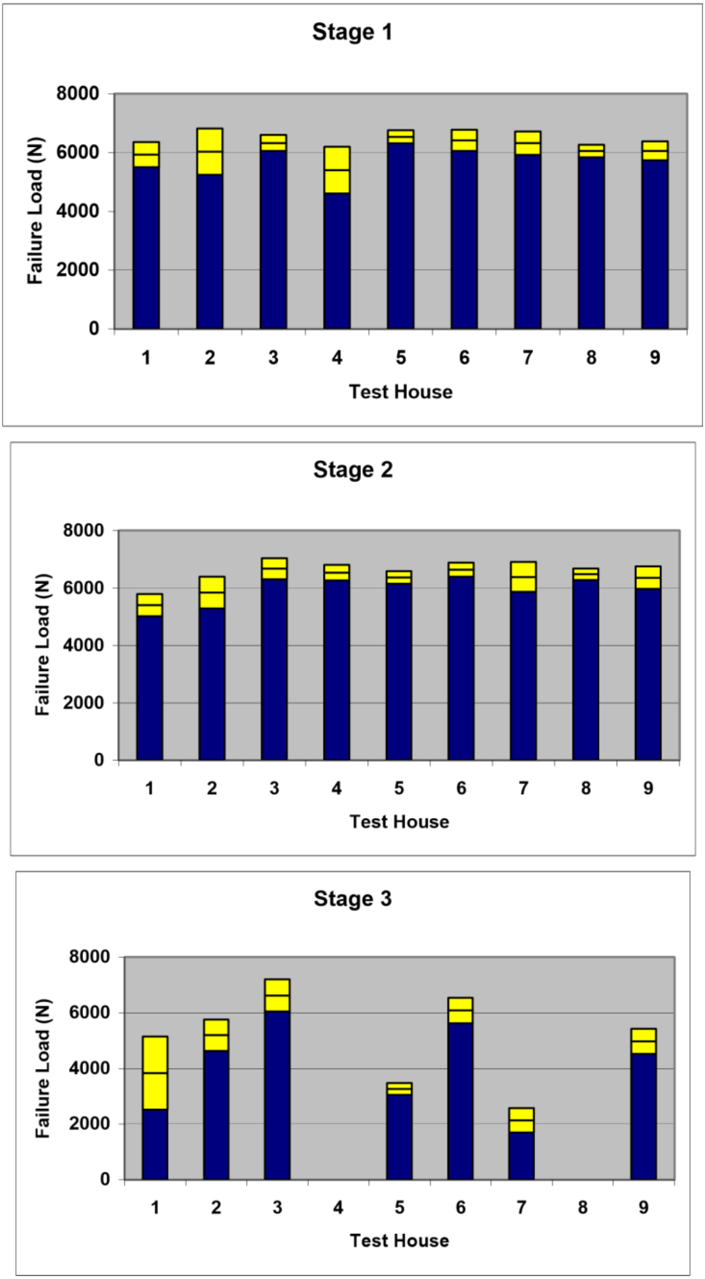


Figure A2.1 Test results (mean ± 1 standard deviation)

The results shown in Table A2.1 show good repeatability within laboratories and good reproducibility between laboratories for Stages 1 and 2. Both repeatability and reproducibility were markedly worse for Stage 3, as to be expected. Tables A2.2 provides details on manufacturing and test parameters used by the various laboratories/test houses in Stage 3.

Parameter	comments
Method of adherend/end tab abrasion	SiC (wet/dry, 60/80 and 180 emery paper) Grit-blasted (240 grit)
Type of solvent used for degreasing	Acetone (commonly used), MEK, IMS and n-heptane
Method of specimen alignment clamping during cure	Support fixture, static weight, and bulldog clips
<u>Cure Conditions</u>	
Cure temperature (°C)	60
Cure time (hrs)	1
Weight of ballotini balls used	1 wt. % (mixed or sprinkled)
Bond-line thickness (mm)	Ranging from 0.20 to 1.00 (typically 0.25 mm)
Test speed (mm/min)	Ranging from 1 to 6
<u>Test Conditions</u>	
Temperature (°C)	Ranging from 22 to 23
Relative humidity	Ranging from 45 to 53 (or not stated)
Failure mode	Cohesive/adhesive (typically 70%/30%)
Additional Observations • Test house 1 observed adhesive not fully cured (i.e., pliable/soft). Two untested specimens were post-cured for 1 hr at 60 °C. Large scatter in strength values for first batch of specimens tested prior to post-curing. • Test house 5 observed lumpy adhesive during joint preparation, which may explain the low joint strengths that were obtained. • Test house 7 observed that the adhesive was gritty (and possibly crystallised), and hence this may have contributed to the large variation in bond-line thickness between specimens (0.25 to 1 mm) and low joint strengths. • Test house 9 removed spew from several specimens after curing the joints, which may have contributed to lower strength values observed.	

Table 2.2. manufacturing and test parameters used by participants in stage 3

The R-R exercise highlighted the need for revision of standard test methods to accommodate non-standard geometries and to remove existing ambiguities. The standard method used in the exercise was open to interpretation and invariably trial specimens were required to obtain the correct machine settings.

A number of concerns raised in relation to the preparation and testing of the single-lap joint are presented below:

- Two-part adhesive needs to be carefully mixed, ensuring correct ratios of hardener to adhesive and that the mixture is uniform. When using glass ballotini balls for controlling bond-line thickness ensure the balls are unformly distributed throughout the adhesive (i.e., do not sprinkle).
- Fillet geometry is known have a major effect on the joint strength, however there is no specification for fillet geometry in BS EN 1465. The fillet should be shaped before cure and/or during cure. The spew should not be removed after cure.
- There is no guidance in BS EN 1465 (or most standards) on methods for controlling bond-line thickness or removing adhesive from the specimen edges in BS EN 1465. The lack of comparative data in the published literature also makes it difficult to select a suitable method for controlling bond-line thickness. Failure to control bond-line thickness can result in low bond strength (see Table A2.5). Temperature of adhesive layer should be monitored to ensure that cure temperature is reached. Do not assume that the oven temperature and the temperature of the adhesive in the joint are the same (see Chapter 4).
- End tabs are introduced to single-lap joints 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 (see Figure 2). BS EN 1465 does not specify the use of end tabs. The introduction of end tabs, however will add to the costs of this quality assurance test.
- Test rate is not specifically stated, but instead the standard specifies that the test joint be loaded at a constant stress or strain rate and that failure be achieved in specified period of time (65 ± 20 s). Hence, there is a need for trials to determine the rate necessary to complete tests within the specified time. At least 2-3 specimens may need to be tested in order to determine a suitable test rate. Test rates varied from 1 to 10 mm/min in Stages 1 and 2 and between 1 and 6 mm/min for Stage 3.
- Standard software format for reporting test data for different test machines could be a step forward in reducing test costs and time.
- BS EN 1465 fails to account for specimens manufactured from materials other than steel or using non-standard dimensions (adherend thickness and bond length, etc.). The standard could be more versatile by providing guidance in these cases.

Site	Failure Load (N)		
	Individual	Mean	
1	5638 6638 6683 6698 6060 6435	6359	427 (6.7)
2	5290 7179 7561 6938 7098 6839	6817	789 (11.6)
3	6514 6906 6213 6695 6401 6860	6598	271 (4.1)
4	6730 5000 5370 6700 6760 6650	6202	797 (12.9)
5	7010 6390 6800 6940 6740 6660	6757	221 (3.6)
6	7129 6821 6585 6417 7262 6430	6774	360 (5.3)
7	6810 6440 6900 7146 6957 6059	6719	398 (5.9)
8	6442 6117 6377 5933 6493 6243	6268	214 (3.4)
9	6200 6020 6340 6980 6400 6360	6383	323 (5.1)

Table A2.3. As-received stage 1 sesults

Site	**Failure Load (N)**		
	Individual	**Mean**	
1	5603 5760 5910 5145 6143 6203	5794	390 (6.7)
2	6413 6750 5820 5873 7103 -	6392	555 (8.7)
3	6450 7369 7329 7285 6783 6991	7035	366 (5.2)
4	7220 6660 6800 6810 6400 6910	6800	271 (4.0)
5	6650 6920 6440 6320 6450 6710	6582	220 (3.3)
6	6854 7162 6864 6476 6873 7117	6886	245 (3.6)
7	7589 7211 7079 7013 6255 6313	6910	525 (7.6))
8	6601 6854 6389 6541 6770 6911	6678	201 (3.0)
9	6950 7400 6390 6640 6340 6780	6750	393 (5.8)

Table A2.4. As-received stage 2 results

Site	**Failure Load (N)**		
	Individual	**Mean**	
1	3393 6203 1153* 6105 4875	5144*	427 (6.7)
2	5700 5438 6038 5888 4915 6570	5758	560 (9.7)
3	7130 6839 6321 7822 7306 7817	7206	581 (8.1)
4	-	-	-
5	3710 3110 3420 3620 3450 3520	3472	207 (6.0)
6	6193 5955 7153 6960 6385 6587	6539	456 (7.0)
7	2070 2790 2530 2800 3160 2070	2570	436 (16.7)
8	-	-	-
9	5520 5780 5320 4900 6040 4980	5423	447 (8.2)

Table A2.5. As-received stage 3 results

** Outlier value removed for analysis purposes*

Appendix 3

Surface preparation of adherends

Introduction

Appendix 3 provides a brief description of generic procedures for preparing different material surfaces [1-9, 21-29].

Steel

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

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

Zinc coated steel

Galvanised steel surfaces are less prone to rusting in most environments than untreated steels and when treated with a suitable surface preparation the adhesive-zinc interface is stronger than the steel-zinc interface. This section provides details on the method to be used for preparing galvanised steel substrates for bonding: Degrease with a suitable solvent (e.g., acetone or methyl ethyl ketone (MEK)).

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

Stainless steel

The recommended steps to be taken in preparation of stainless steel surfaces are:

- Degrease to remove oils and lubricants present on the substrate surface.
- Grit blast the surface ensuring the protective 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:

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

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

Titanium alloys

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

- Degrease surface with a suitable solvent (e.g., acetone or MEK).
- Grit blast
- Remove dust/debris by either brushing, by oil-free air blast or by vacuum cleaner.
- Acid etch the surfaces.
- Rinse and dry.
- Chemically stabilise oxide layer.

Fibre-reinforced polymer composites

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 fibre-reinforced polymer composites is as follows:

- Grit blast
- Remove dust/debris by either brushing, by oil-free air blast or by vacuum cleaner.
- Acid etch the surfaces.
- Rinse and dry.
- Chemically stabilise oxide layer.

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 [28]. 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 affects the physical and mechanical properties of the outer layer of the cured laminate. If the peel-ply contains a release agent, the adherend should be cleaned with a solvent and dried in a stream of dry nitrogen.

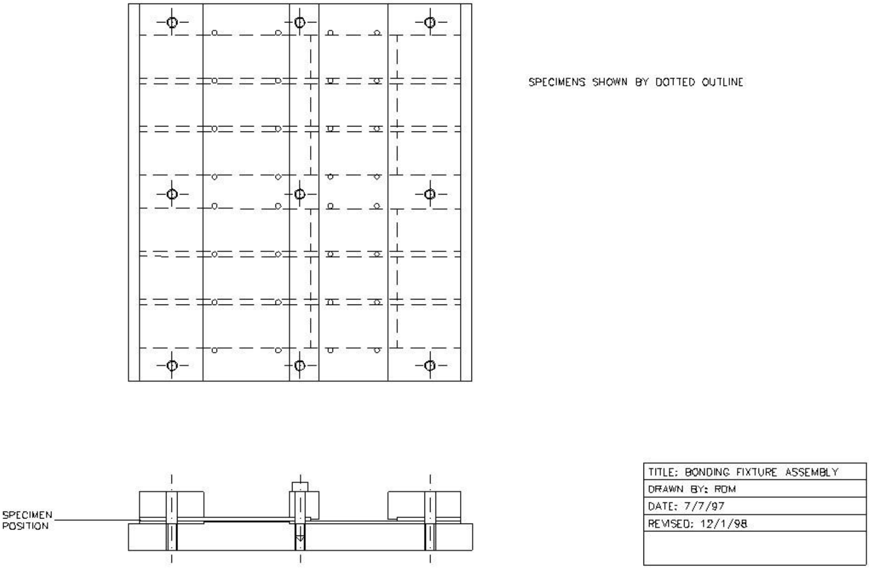
Note A3.1: All fibre-reinforced plastic composites absorb small amounts of moisture from the atmosphere when exposed to ambient levels of humidity (i.e., standard laboratory conditions). For ambient temperature curing adhesive systems, the presence of moisture in the composite adherend may pose no problems, however it is advisable, to dry the adherends prior to bonding to prevent reductions in glass transition temperature T_g and fracture toughness G_c.

Appendix 4

Bonding fixture for single-lap joints

Introduction

Appendix 4 provides technical diagrams to construct a bonding fixture for producing single-lap joint specimens (see Figure 2). This fixture was used to produce the specimens for Stages 1 and 2 of the R-R exercise reported in Appendix 2.



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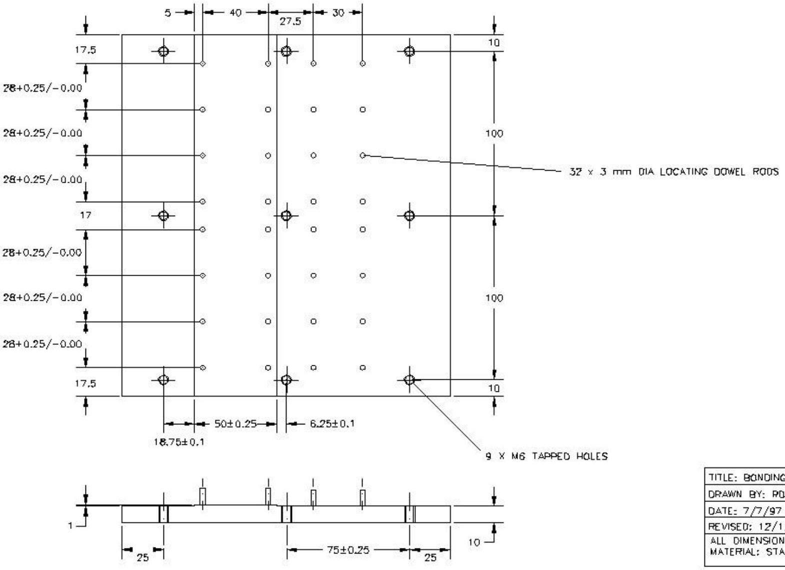
REVISED: 12/1/98



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REVISED: 12/1/98
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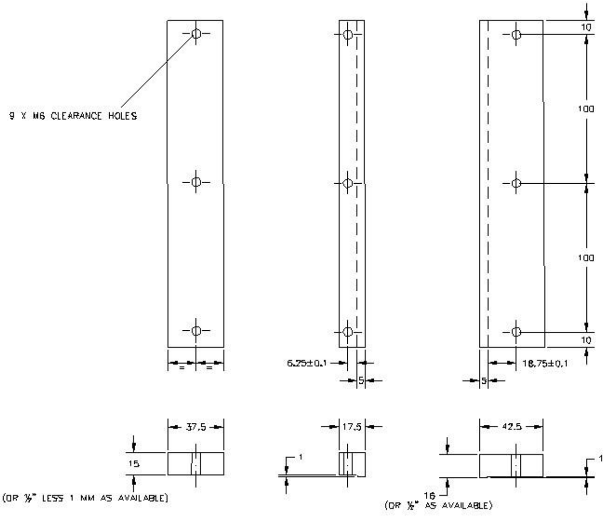
MATERIAL: STAINLESS STEEL



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REVISION 1: 12/1/98
REVISION 2: 9/2/98
ALL DIMENSIONS IN MM
MATERIAL: STAINLESS STEEL

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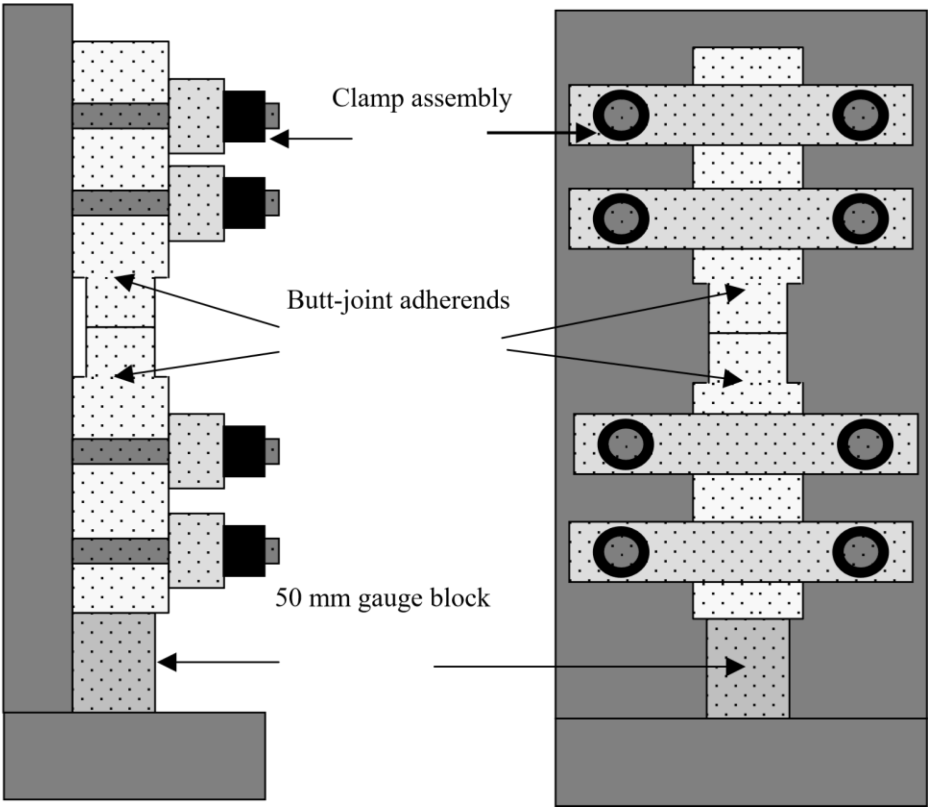
Appendix 5

Alignment fixture for bonding tensile butt joints

Alignment fixture for bonding tensile butt joints

The diagram shown below is a schematic of the alignment fixture used at NPL for bonding tensile butt joint specimens (see also Figure 3).



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Abstract

The reliability (i.e., repeatability and reproducibility) of data obtained from testing adhesive joints can be significantly affected by factors not included in written standards. Factors, such as specimen and machine alignment, processing variables, storage of materials and surface treatments need to be considered in addition to test parameters (e.g., displacement rate) and specimen dimensions that are generally covered in national and international standards. Careful consideration needs to be given to those factors that can significantly influence the strength and long-term performance of adhesive joints. This Good Practice Guide is intended to give guidance on the preparation and testing of adhesive joints, and the use of test methods under ambient and hostile environments (including accelerated ageing) for generating design data and for quality assurance purposes. Static, cyclic fatigue and creep rupture testing are also included. The document is primarily concerned with structural adhesives, but also includes flexible adhesives for structural or semi-structural applications.

The Good Practice Guide also includes the results from a three-stage Round-Robin exercise on *Adhesive Joint Testing* that was conducted in conjunction with UK industry to identify key preparation and testing issues.

Acknowledgements

This document is an updated version of Good Practice Guide No. 47 (originally produced in a *Performance of Adhesives Joints* project as part of the BEIS funded *Materials Measurement Programme*). Good Practice Guide No. 47 has been updated as part of a review of composite and adhesive GPGs within NPL's NMS programme funded by BEIS. Support and input from colleagues at NPL are recognised and was greatly appreciated throughout the project's lifetime. The advice and guidance from Gareth McGrath (project supervisor on behalf of BEIS), the Adhesives programme Industrial Advisory Group and industrial participants in the *Adhesive Joint Testing* Round-Robin exercise are gratefully acknowledged.

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.

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

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

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

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

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

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

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

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

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

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

Structural bond: 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 personnel and quality assurance personnel on the preparation and testing of adhesive joints for generating design data and for quality assurance purposes. Guidance is provided on static, cyclic fatigue, and creep rupture testing, and testing of adhesive joints in hostile environments (including accelerated testing). The document covers specimen preparation (including adherend machining, surface treatments, joint assembly, and bonding), specimen storage and mechanical testing (including specimen and machine alignment, strain, and displacement measurement). Consideration is given to the effect of material and geometric factors (i.e., bond-line thickness and fillet geometry), and test parameters (i.e., displacement rate) on joint stiffness and strength. The document considers thermal analysis techniques for monitoring cure and non-destructive evaluation (NDE) techniques for inspection of bonded structures.

The document includes a summary of commonly used tensile, peel, cleavage, shear, and fracture toughness test methods, providing guidance on issues relating to specimen requirements, material properties obtained (including associated accuracy), materials selection and amount of material required, costs of specimen preparation and testing, data reduction and suitability for use under creep, fatigue, and environmental conditions. The document, which is primarily concerned with adhesives for structural applications (including toughened and flexible adhesives), covers joints that involve substrates fabricated from either metals or fibre-reinforced thermoset polymer composites. 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 reliably test adhesive joints for producing design data and to enable initial screening of adhesive/adherend/surface treatments. If the intention is to generate design data, then the guide should be used in conjunction with the appropriate structural design codes. The guide assumes some basic knowledge of the materials and techniques involved and is not intended as a textbook.

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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Correct surface preparation is essential for ensuring both initial adhesion and long-term joint durability. Although the guide provides advice on the surface preparation of the commonly used structural materials, it only covers the main steps in each process. It is essential that expert advice from the adhesive manufacturer is obtained and that the detail requirements specified by the manufacturer are completely satisfied.

Where tests are performed to characterise the adhesive material then it is recommended that the surface preparation is as good as possible to minimise premature adhesion failure. Where tests are performed to evaluate a bonding system then the surface preparation procedures for test specimens will need to mirror those for the final bonded component.

The Guide includes the results from a three-stage Round-Robin (R-R) exercise on *Adhesive Joint Testing* that was carried out as part of the assessment to identify key preparation and testing issues. The R-R exercise involved eight industrial companies and the NPL Materials Centre. The results have been analysed using statistical analysis. A good example of an industrial procedure for preparing and testing adhesive joints is presented within the overview of the R-R exercise.

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Chapter 1

Scope

Scope

Adhesive tests can be divided into comparative methods for adhesive and process selection, quantitative methods for generating engineering property data for design purposes, and qualitative methods for assessing long-term (i.e., durability) performance of bonded systems under combined mechanical loading and hostile environments. Although, an extensive range of test methods are available as national and international standards (see Appendix 1), 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, or comparative analyses 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. Lists of standards issued by the American Society for Testing and Materials (ASTM), British Standards Institution (BSI) and International Standards Organisation (ISO) are presented in NPL Report CMMT(A)61 [13].

A major concern with testing adhesive joints is the number of factors, not included in written standards, which can significantly affect the reliability (i.e., repeatability and reproducibility) of test data. Factors, including specimen and machine alignment, processing variables, surface treatment, adhesive storage and joint ageing need to be considered in addition to test and specimen parameters (e.g., displacement rate and dimensions) that are covered in national and international standards. It was evident from a statistical analysis of R-R data carried out in a previous adhesive programme that the uncertainty associated with specimen preparation and testing has a substantial effect on the test results. The large scatter and disparity in results between test houses indicates that several additional factors need to be controlled to ensure reliable data. The situation is further exacerbated when tests are conducted in hostile environments.

The guide is primarily concerned with metals and fibre-reinforced polymer composites bonded with structural adhesives, which includes toughened and flexible adhesives. Guidance is provided on specimen preparation, storage of adherends, adhesives and adhesive joints, and testing and environmental conditioning of adhesive joints. Static, cyclic fatigue and creep rupture testing are covered. Consideration is given to the effect of material and geometric factors on joint stiffness and strength. A list of recommended test methods for determining input data for the design and analysis of bonded joints is given in Appendix 1.

A three-stage R-R exercise was conducted on *Adhesive Joint Testing* in conjunction with adhesive suppliers, end users and test houses within the UK, as part of the assessment to identify key preparation and testing issues. The R-R results, which have been analysed using statistical analysis, are presented in Appendix 2. The R-R exercise involved nine participants (eight industrial companies and NPL).

A brief description of generic procedures for preparing different material surfaces is given in Appendix 3. Appendix 4 provides technical diagrams to construct a bonding fixture for producing single-lap joint specimens. A schematic diagram of the alignment fixture used at NPL for bonding tensile butt joint specimens is shown in Appendix 5.

Organisations that can provide specialist advice are listed at the back of the guide along with relevant standards and publications.

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Chapter 2

Handling and storage of adhesives

- Introduction
- Handling and storage
- Documentation (record handling)
- Measurement of bulk adhesive mechanical properties

Introduction

Several issues relating to the handling and storage of adhesives need to be considered as many of these issues can affect the performance of the material. Issues that need to be considered include:

- Health and safety (COSHH)
- Documentation
- Handling and storage (including moisture and temperature effects)

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.

Handling and storage

Polymeric resins may undergo physical and chemical changes when stored for a considerable length of time or under unsatisfactory storage conditions, thus adversely affecting the performance of the adhesive. For example, adhesives may undergo an increase in viscosity, or films a loss of tack and drapeability. Less flow and, thus poorer wetting of the surface, will occur during cure and gel times of epoxy film adhesives could become shorter. Extended exposure of uncured materials to temperatures and humidity's above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [3]. The overall effect is to reduce the adhesive performance and induce premature failure of adhesively bonded joints.

The safety and product data sheets will provide the user with recommendations regarding storage temperature for an adhesive. Adhesives should be stored in sealed containers to prevent moisture ingress and contamination, and some cases exposure to light. Opening a storage container exposes the adhesive to oxygen and moisture, which may in some cases severely reduce the shelf life of the adhesive. The shelf (storage) life is generally defined as the time that an adhesive can be stored (unopened) without any adverse affects on the cure properties and bond strength of the adhesive. The product sheet will often specify the shelf life of the adhesive for various temperature ranges. An adhesive should not be used beyond the expiry date, which will be indicated on the container. The shelf life of an adhesive can decrease significantly with increasing storage temperature. Unless the manufacturer explicitly states that the adhesive must stored or would benefit from being stored at sub-ambient temperatures then adhesives should be stored at ambient temperatures (18 °C - 25 °C). Adhesives should not be stored below freezing unless recommended by the manufacturer.

Ideally, temperature and humidity in the storage area should be tightly controlled and monitored (i.e., temperature recorders), although this may not always be physically or economically feasible. Standard laboratory conditions are typically 23 ± 2 °C and 50 ± 5 % relative humidity (RH). Refrigerators (or freezers) should be armed with a temperature alarm and the temperature should be monitored continuously to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. The maintenance of the refrigerator units should allow for regular manual or automatic defrosting.

A refrigerated adhesive may be used several times during the shelf life of the product. This will involve the product being taken from a sub-ambient (often sub-zero) temperature and placed at room temperature and allowed to thaw. It is advisable throughout this exercise that the adhesive is enclosed (i.e., remains enclosed in a sealed container) until the temperature has reached room temperature and is ready to be applied to the adherends. Containers that have been opened must be sealed immediately after use. The amount of time at ambient for these adhesives should be minimal, advice on the length of time can be obtained from the adhesive manufacturer. Repeated exposure of the adhesive is a frequent cause of adhesive ageing and deterioration. Sampling of the adhesive before removal from sub-ambient storage may help extend the shelf life of the product, however this may not always be possible for adhesive packed in cartridges.

Exposure to repeated freeze/thaw cycling could limit the shelf life of an adhesive. Exposing a film adhesive that has been stored at sub-ambient temperatures will result in moisture condensing on the surface, which if not allowed to evaporate will compromise the strength and durability of the joint. Entrapped moisture will be released during the cure process, thus compromising the physical and mechanical properties of the adhesive and contribute to the degradation of the bonded surfaces. Routine tests to verify that the physical and mechanical properties of the adhesives are still within specification are recommended, particularly where multiple use of an adhesive has occurred.

Adhesives should be stored in an area specifically dedicated to these materials. The area should be suitable for handling flammable and hazardous materials. It may be necessary to segregate materials depending on the hazardous nature of the material. Materials in these areas should be clearly identified and controlled.

Documentation (record keeping)

It is recommended that an accurate record of the adhesive be maintained, and that routine checks are made each month to ensure that the stored materials are within the time limits recommended by the adhesive manufacturer. A record should include details on the material source, manufacturer’s code number, batch or lot number, form (i.e., one-or two-part, or film, etc.), shelf life, storage temperature and handling history during the shelf life (i.e., dates of use and length of time at room temperature if refrigerated). The report should include test data from acceptance tests and routine quality checks carried out to assess material performance.

Adhesive manufacturers will often supply data sheets (material certification) with their adhesives, normally quoting lap-shear strengths. Data sheets may sometimes include measured adhesive joint strength (i.e., tensile shear, fatigue strength, creep rupture and T-peel data) for a range of temperatures and environmental conditions (i.e., long-term exposure to methanol, gasoline, acetone, acetic acid, or water). The manufacturer usually employs either aluminium or titanium substrates for material qualification. The data supplied may not be easily applied to other surfaces. Few manufacturers provide bulk adhesive data. These properties usually need to be generated in-house (see next Section).

It is recommended that an accurate check as to the quality of an adhesive be carried out on receipt of the material to ensure that material meets the manufacturers specifications, ensuring the product complies with the purchase order and suppliers test reports (see [9]). The colour, viscosity, shelf life, etc. should be verified. It may be necessary to carry out acceptance tests in-house to ensure mechanical properties are in accordance with the material specification. Acceptance tests should be directed towards assuring that the materials are identical from batch to batch. Accelerated environmental tests (i.e., high temperature and humidity) may also need to be carried out to confirm that the durability performance of the material meets the manufacturer specifications. ISO, BSI, and ASTM list various test methods for quality assurance of adhesives (see Appendix 1 and [13]).

Measurement of bulk adhesive mechanical properties

Bulk adhesive tests, which enable the determination of adhesive properties without the need for correcting for adherend effects, are often used to obtain accurate mechanical and physical properties for computer aided design calculations. A cautionary note, steps must be taken to ensure that the adhesive material is representative of the material within the actual bond layer. Recommended procedures for the preparation of bulk specimens of adhesives are given in ISO 15166 [14-15]. The standard considers two-part adhesives cured at ambient or elevated temperatures (Part 1) [14] and single component systems requiring an elevated temperature to cure the adhesive (Part 2) [15] - see also references [16-19]. Bulk adhesive specimens can be cast or machined to the required shape (e.g., dumbbell tensile specimens) - see [10, 12].

It is important that the specimen is free of surface damage (i.e., scratches and nicks). Many liquid and film adhesives can be cast into bulk specimens without the need for machining. To minimise the deleterious effect of surface scratches, which may cause premature failure, the edges and faces of the specimens should be carefully polished to remove any surface defects. Advice on specimen preparation, test methods and standards, and environmental conditioning and testing of bulk adhesives (including referenced standards) are given in references [11-19].

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Chapter 3

Handling and storage of adherends

Before and after machining, adherends sensitive to environmental attack or hygroscopic in nature should be stored in sealed containers to prevent moisture ingress and contamination. It is important that the adherend does not undergo physical or chemical changes whilst being stored. Moisture can alter the chemistry of both the surface and substrate of the adherend, thus compromising the performance of bonded joints. It is recommended that an accurate record of the adherend (including condition and use) be maintained throughout the duration of use the material. Inventory control mechanisms employed for adhesives are also relevant to the storage and handling of adherend materials (see Chapter 2).

Quality documentation should include details on the material source, manufacturer's code number, batch or lot number, form (i.e., sheet or plate), storage conditions, surface conditions of the adherends (including corrosion products, and surface defects such as scratches present on receipt of the material), and the location of each specimen sectioned from the adherend. The inventory report should also include (where necessary) test data (i.e., strength and stiffness properties) for the adherend. Test certificates are often supplied with metallic materials, such as titanium and aluminium alloys, in which an elemental analysis is supplied along with basic tensile property data. These sheets contain information on the materials condition (e.g., descaled, and annealed titanium). Fibre-reinforced polymer composite suppliers provide data sheets on processing conditions, fibre volume fractions and mechanical properties of their products. Often elastic and strength properties are quoted for a range of temperatures, and in some cases for hot/wet conditions (e.g., 70 °C and 85% RH) and other environments. It may be necessary to generate mechanical property data for design purposes. This data should be included in the documentation for the material. Variations in material thickness, and elastic and strength properties can be expected between batches of materials.

Chapter 4

Preparation and assembly of adhesive joints

- Machining and storage of adherends
- Surface preparation of adherends
- Joint assembly
- Curing adhesive
- Quality assurance

Introduction

The reliability of an adhesive joint depends not only on selecting the correct adhesive, but also on the preparation of the adherends, mixing of the adhesive, joint assembly, and the curing process. It is worth noting that a high percentage of failures can be attributed to poor joint manufacture or a lack of understanding of that factors that influence joint performance. These problems can be minimised or eliminated through proper training and education. This chapter examines the key issues relating to the preparation and assembly of adhesive joints.

Machining and storage of adherends

Adherends should be manufactured and/or machined accurately to ensure specimen dimensions meet the specifications of the standard. It is important to ensure that the adherends are free of any edge or surface damage. The surfaces to be bonded must be parallel and flat to ensure uniform, intimate contact, across the entire bond area when the two surfaces are clamped or pressed together. The operator should ensure that during the machining process, no nicks, cuts, or scratches are introduced at the edges or surfaces of the adherends. Surface or edge defects can cause premature failure of the adhesive joint and/or the adherend. It is advisable before preparing the surface to ensure that the adherend sections to be bonded fit together well with the bonded surfaces closely matching (i.e., intimate contact between the two surfaces). Guillotining thin metal sheets is a rapid and low-cost method for producing large quantities of lap-shear test adherends, however the cutting operation can result in bending of the adherends and operators will therefore need to be ruthless by discarding those specimens that fail to meet the specification.

Fibre-reinforced thermoset composites are known to absorb moisture in relatively benign environments. For example, epoxy based composite systems can absorb 0.2 wt. % moisture in a laboratory environment (i.e., 23 °C and 50% RH within 2 to 4 weeks. The presence of moisture in the composite can adversely affect the properties of the adhesive during the cure process, and as a result the joint strength may be compromised. Moisture released from the composite substrate during cure will enter the adhesive and has been known to reduce the glass transition temperature T_g by as much as 20 °C and lower the fracture toughness $\mathbf{G_c}$ of a rubber toughened epoxy adhesive by a factor of 10 [20]. With toughened adhesive formulations, the presence of moisture may inhibit phase separation of the rubber-toughening agent, thus preventing the formation of rubber-toughened particles. Adherends (pre-dried) should therefore be stored in a dry area (i.e., desiccator or sealed container with a suitable desiccant). It is recommended that polymer composites be pre-dried in an oven maintained at 50 ± 2 °C (unless otherwise specified) until the specimen weight reaches a constant value. The temperature of the drying oven should not exceed the maximum operating temperature of the polymeric matrix.

Surface preparation of adherends

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. Many adhesive joint tests have been developed to assess bonded systems (including surface preparation) – see Appendix 1 [13].

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. Appendix 3 provides a brief description of general procedures required for preparing different substrates for adhesive bonding. Many 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 D 2651, ASTM D 2093, BS 7079, BS EN 1840, and BS 7079 [21-25] – see also [26-29]. Advice should be sought on surface preparation from the adhesive manufacturer. **Surface preparation procedures often require potentially hazardous or environmentally damaging chemicals. All preparation should be carried out to COSHH specifications**.

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. Table 1 provides a guide as to the relative quality and cost of various surface treatments. Environmental impact of the process may also need to be assessed when selecting a surface treatment. Clean grit, clean solvent and clean cloths must be used to avoid spreading contamination. The maximum allowable time between surface preparation and bonding or priming metal and composite substrates is dependent on the substrate and the surface treatment (see Table 2).

Surface Treatment	Cost	Quality
None	Low	Low
Solvent Degrease	↓	↓
Vapour Degrease		
Mechanical Abrasion		
Plasma		
Chemical Etch		
Anodising	Expensive	Expensive

Table 1. Relative cost and quality of various surface treatments [9]

Surface Treatment	Cost	Quality
None	1-2 hrs	± 20
Solvent Degrease	1-2 hrs	± 20
Vapour Degrease	1-2 hrs	± 20
Dry Grit-Blasting (Steel)	4 hrs	± 20
Wet Grit-Blasting (Steel)	8 hrs	± 20
Wet Grit-Blasting (Aluminium)	72 hrs	± 20
Chromic Acid Etch (Aluminium)	6 days	± 10
Sulphuric Acid Etch (Stainless Steel)	30 days	± 10
Anodising (Aluminium)	30 days	± 10
Dry Grit + Organosilane (Aluminium)	2-5 hrs	± 5

Table 2. Maximum exposure time between surface preparation or priming metal substrates, and associated variation in tensile shear strength

Surface treatments can be classified as either passive or active [9]. Passive surface treatments (e.g., solvent washing and mechanical abrasion) clean the surface and remove weakly attached surface layers without altering the surface chemistry. Active surface treatments (e.g., anodising, chemical etching, laser, and plasma treatment) alter the surface chemistry (i.e., introduction of functional groups).

The combined effect of solvent and mechanical abrasion (i.e., grit-blasting) treatments is to remove most contaminants and alter the surface topography (increase in surface roughness or bond area). Chemical and plasma treatments also remove contaminants and increase surface roughness; however, these treatments also increase surface reactivity.

Silicon carbide grit-coated paper or other abrasive pads can be used dry or in the presence of a solvent to mechanically abrade the adherend surfaces. It is essential that sufficient time be allowed to ensure the solvent evaporates. Surfaces should be cleaned following abrasion to remove any abrasive material left on the surface. Alternatively, surfaces can be mechanically abraded through dry or wet grit blasting. Grit blasting in addition to removing surface debris and roughening the surface may cause physico-chemical changes, which affect surface energy and wettability [26]. Coarser grit produces rougher surfaces, resulting in lower surface energies, thereby increasing environmental durability of the bonded joint. Differences in initial strength between fine and coarse grits, and types of grit are negligible. Clean grit should always be used for preparing adherend surfaces. To prevent transfer of contamination, airline contamination and contamination of the grit should be avoided. Wet blasting is increasingly being used in preference to dry blasting as the method provides more consistent surface properties [26].

The results presented in Table 2 refer to unconditioned material that has been tested within a short period (e.g., 1-2 weeks) of joint fabrication. The variations in joint strength quoted in Table 2 are optimal values that can be expected under controlled conditions. The uncertainty (or variation) in tensile shear strength can be expected in many cases to increase following exposure to a hostile environment and/or through poor workmanship.

Simple surface treatments (e.g., grit blasting or vapour degreasing) are less prone to human error and therefore the variation in joint strength is unlikely to exceed ± 20%. Bonded joints with chemical surface treatments are more prone to large variations in joint strength, particularly as there are often numerous controlling variables, which need to be strictly controlled.

Primers such as aminopropyltriethoxysilane (or γ-APS) are often applied to: (i) protect the substrate surface prior to bonding; (ii) increase surface wettability; and (iii) and inhibit corrosion [9]. Primers also act as a coupling agent, forming chemical bonds with the adherend and adhesive, thus improving joint strength and environmental durability. Silane coupling agents (e.g., γ-APS) [27] are known to improve durability in the presence of moisture by increasing the water resistance of the oxide layer on the adherend surface. Joint strengths can be very low, however in cases where the silane treatment is poorly controlled. Incorrect application can result in interfacial failure or cohesive failure within the silane coating. Care is needed to ensure a uniform coating, a monomolecular layer thick, is produced across the entire surface area of the adherends to be bonded.

In all cases involving solvents, primers or active chemical surface treatments (e.g., acid and alkaline solutions), extreme care and good laboratory practice should be followed at all times in storing, handling and disposing of these materials (recommended further reading [9]). Personnel need to be trained in handling and using these materials and should wear appropriate protective clothing as the chemicals can be harmful if they come contact with the skin or eyes. Ventilation and spill containment are particularly important safety considerations. Chemical treating solutions should be prepared and stored in containers of glass, ceramic, or chemical resistant plastic. Checks should be made to ensure that the containing vessel is resistant to chemical attack from would be contents (e.g., hydrofluoric acid attacks glass). Stirring rods should be made of the same materials. Contact of the chemical solution with metals during preparation and storage should be avoided.

Continuous monitoring and quality control are required for all chemicals employed in surface treatments, and all containers with chemical agents should be clearly identified with labels. Labelling should be protected from being damaged through chemical contact or handling. Chemical solutions need to be changed regularly to prevent contamination and ensure repeatable concentration [9]. Quality assurance tests are recommended to check concentrations, and levels of contamination (e.g., opacity for checking particulate content, colour and pH for acidic and alkaline solutions).

Treatment tanks or containers with chemical etching solutions (e.g., chromic acid) should be of sufficient size to accommodate the specimens to be treated (i.e., volume of chemical solution should be well in excess of the volume occupied by the component in solution). Tests may need to be carried out to determine the minimum volume required to treat a component. Surfaces to be treated should be completely immersed in all solutions. Treatment tanks used in chemical etching processes should be equipped with temperature controls and should be regularly agitated or stirred to prevent local overheating and to ensure uniform concentration of the chemical etching solution. The tanks should be suitably lined to prevent reaction between the tank and the chemical solution, thus ensuring no adverse effect on the solution used or the surfaces being treated. Rinse tanks should be large enough to accommodate the specimens that have been treated and should be equipped to allow for circulation of clean water to prevent build-up or carryover of materials between batches of specimens.

Metallic surfaces need to be free of corrosive products (e.g., rust) before surface treatment, and the area should be as clean (i.e., free of dust, oil and grease) as possible. These need to be removed prior to surface treatment by mechanical abrasion or dry grit blasting. Vapour degreasing, solvent cleaners, emulsion cleaners or alkaline degreasers can be used when necessary to remove oils and grease from adherend surfaces. A new generation of adhesives have been developed to bond to oily surfaces. For these materials, removal of surface oil and grease is generally not conducive to good joint strength with manufacturers frequently recommending that the user avoid the removal of oil and grease, which participate in the chemical bonding process for these materials.

Immersion of adhesive joints in water at elevated temperatures or exposure to hot/humid environments (e.g., 70 °C and 85% RH) is a commonly used approach for assessing the effectiveness of different surface treatments. Figure 1 compares the relative environmental resistance of different surface treatments aged in water at elevated temperatures.

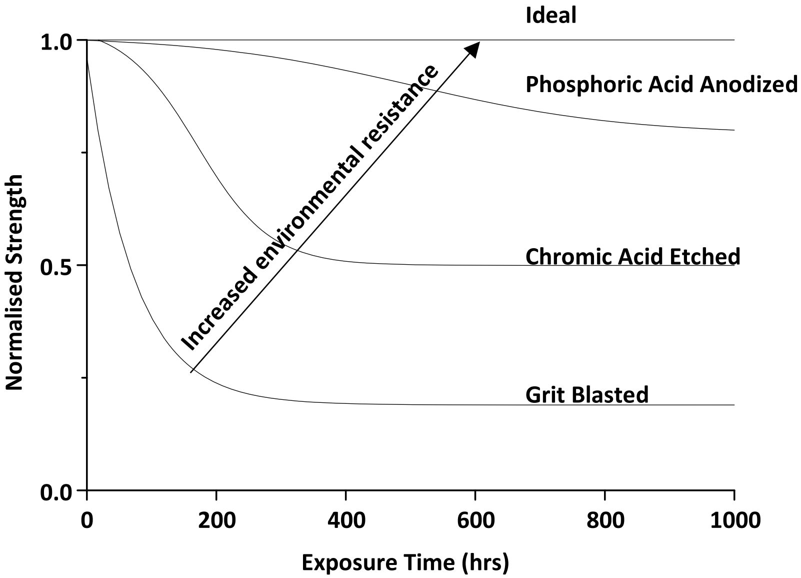

Figure 1. Effect of different surface treatments on the durability of aluminium single-lap joints bonded with an epoxy adhesive immersed in water at elevated temperature

Joint assembly

This section is concerned with issues relating to joint assembly prior to curing the adhesive (i.e., control of bond-line thickness and adhesive fillet and removal of adhesive spew).

Bonding fixture

A bonding fixture is recommended to ensure correct bond length (in the case of lap joints), accurate alignment and uniform bond-line thickness. An example of a bonding fixture for single-lap joints is shown in Appendix 4 [30]. The fixture shown in Appendix 4 enables the overlap length and placement of end tabs to be accurately controlled. The fixture, which allows for up to six individual specimens to be bonded at a time, has no facility to control the adhesive fillet. Alternatively, large test panels (typically 180 mm wide) capable of providing 6 specimens can be made and then cut into specimens. Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e., adherend bending) or that excessive adhesive is forced from the joint due to clamping forces applied to the test specimens.

It may be necessary to check the clamping force applied by the fixture to the specimen during the curing process to ensure that clamping force remains constant and has not relaxed through adhesive flow. Mould release agent or thin polytetrafluorene (PTFE) film will need to be used to guarantee easy release of bonded components from the clamping fixture.

Note 2: End tabs are introduced to single-lap joints 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 (see Figure 2). BS EN 1465 [31] 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. The introduction of end tabs, however, will add to the costs of this quality assurance test.

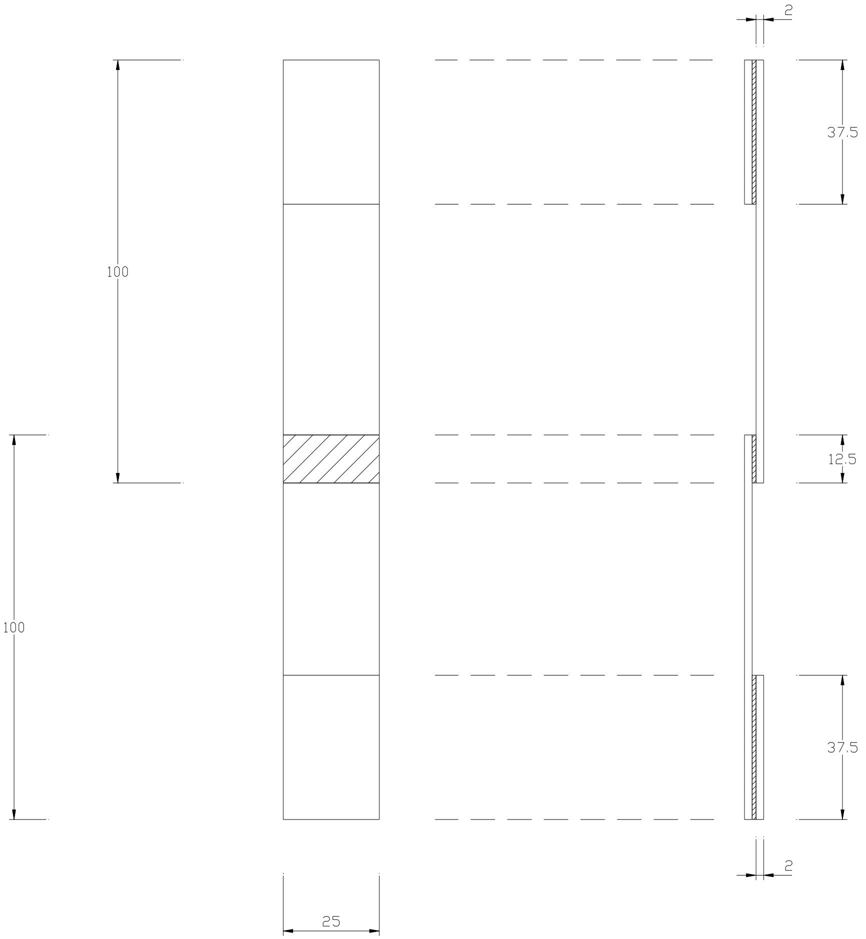

Figure 2. Schematic of single-lap joint (dimensions in mm)

A bonding fixture is essential for adhesive bonding of butt joints (see Appendix 5), as good alignment is key to this method producing data. The cylindrical butt joint (Figure 3) 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 take to ensure good alignment during specimen preparation (i.e., bonding of adherends) and testing. Small misalignment can severely reduce strength data. Variations of the test configuration have been included in ASTM 897 and ASTM D 2095 and BS EN 15870 [32-34]. At present, there is no definitive standard test method.

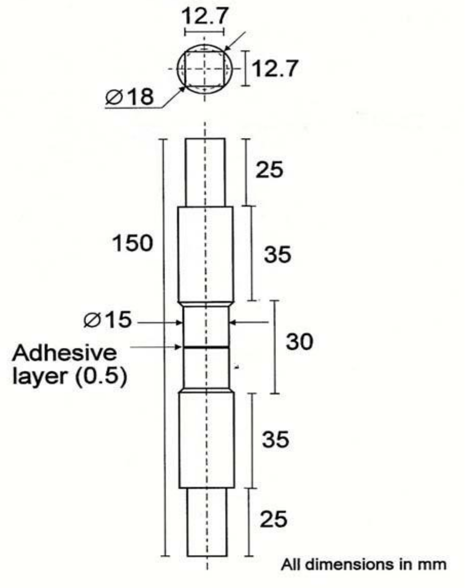

Figure 3. Cylindrical butt joint

Adhesive fillet

Efforts to reduce stress concentrations formed at the bond-line ends of joints may include the use of tapered or bevelled external scarf and radius fillets at the bond-line end (NB. The use of very rigid adherends will not eliminate stress concentrations at the bond-line). Significant increases in the "apparent" shear strength of single-lap joint, compared with square-ended bond-lines, can be achieved through the formation of a fillet or spew at the overlap ends. Further increases in strength may be achieved by rounding the ends of the adherends. The spew also acts as a barrier to water and chemical ingress from the surrounding environment.

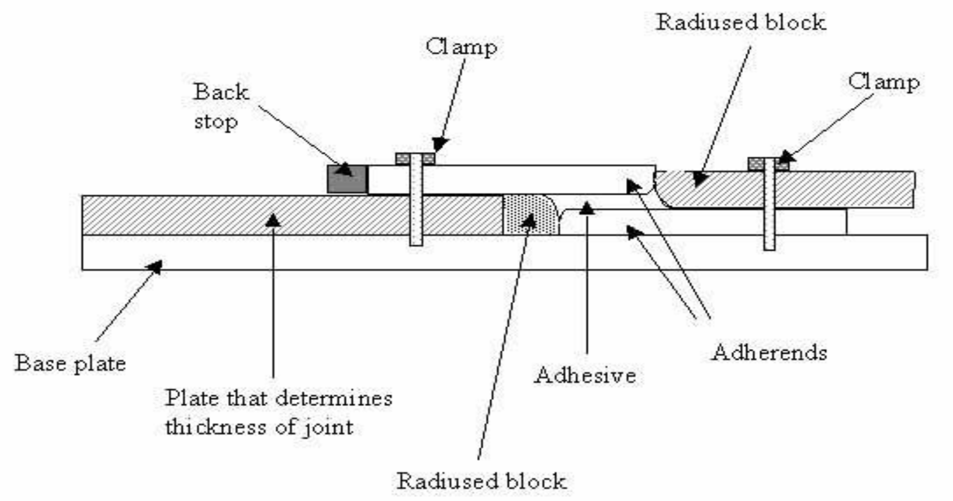

Figure 4. Fixture for controlling single-lap joint fillet

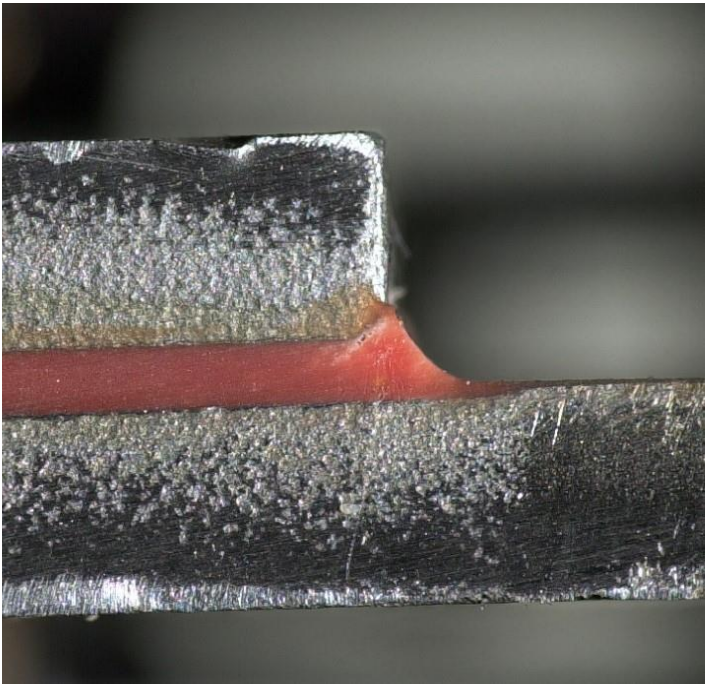

Figure 5. Single-lap joint with a concave fillet

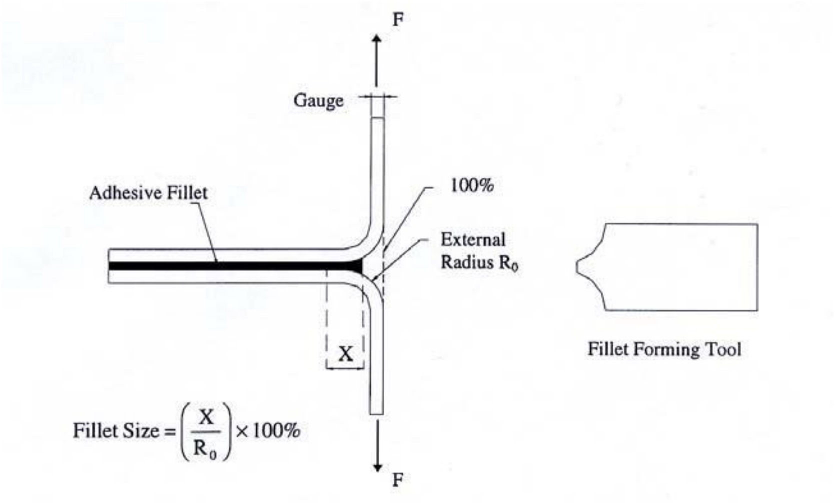

Figure 6. Definition of fillet size in T-peel joint (including fillet forming tool) [35]

Note 3: 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. It is recommended that the Tpeel joint specimen has a 50% adhesive fillet.

Several points are worth noting:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint; as shown in Figure5. The tool can be held in place using heat resistant tape. Figure 6 shows a tool that was used to produce a consistent fillet for T-peel joints bonded with a paste adhesive. The tool can be fabricated from either aluminium or stainless steel coated with release agent.
- Controlling fillet or spew geometry is not always possible as a number of adhesives undergo minimal flow during cure (e.g., flexible adhesives). The high viscosity associated with flexible adhesives prevents adhesive flow, thus making it difficult to control the fillet geometry.
- Ideally, the fillet geometry should be kept constant when producing comparative mechanical property data for different adhesive/adherend systems.
- Strengths of flexible adhesive lap joints remain fairly constant for fillet lengths in excess of 2mm, and therefore for consistency, large fillets should be allowed to form.
- Care needs to be taken to ensure no adhesive is removed from inside the bond area when removing excess adhesive from the joint prior to cure. Removing adhesive from inside the joint will result in localised debonding and poor joint performance.

- Avoid removal of adhesive spew from the ends of joints after cure, as there is the possibility of damaging the joint. It may be convenient to remove spew from the specimen sides to provide a straight edge for aligning in a test machine. This can be achieved using emery paper.

The use of tapered or bevelled external scarf and radius fillets at the bond-line ends will reduce peel and shear stresses induced by eccentricity in the loading path (see Figure 7). These additional features will add considerably to the costs of specimen manufacture.

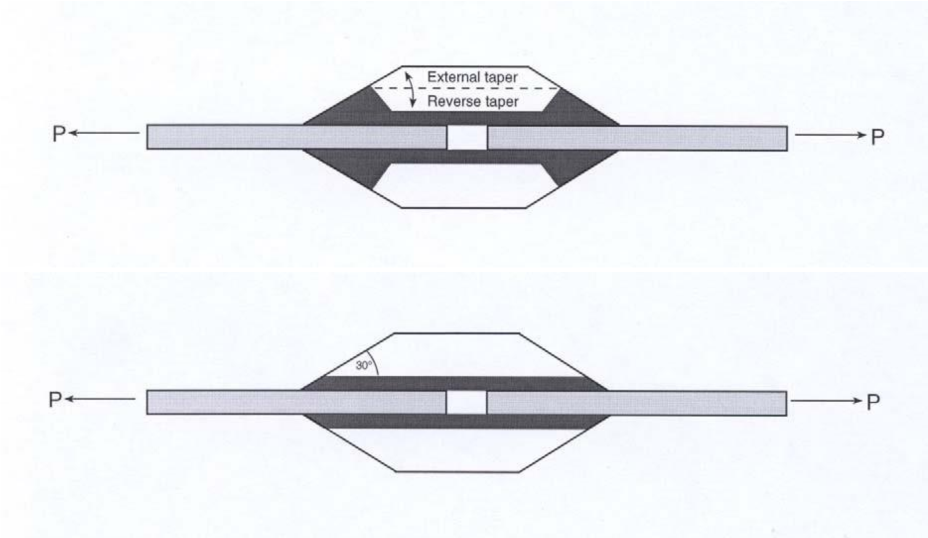

Figure 7. Tapered (bottom) and bevelled (top) strap joints

Bond-line thickness

Bond-line thickness needs to be accurately controlled (i.e., uniform adhesive layer thickness across the entire bonded area) to obtain consistent and reliable joint strength. Also, the method used to control bond-line thickness must not introduce voids or promote void formation in the adhesive otherwise the joint performance will be compromised. It should be noted that the thicker the bond-line the higher the risk of incorporating a high level of voids. In addition, stresses at the corners of the joint tend to be larger as it is difficult to maintain axial loading with a very thick bond-line. Thick adhesive layers can change the cure properties producing internal stresses, thereby reducing short and long-term performance. Conversely too thin a bond-line can result in adhesive starvation and debonding. Optimum bond thickness will depend on the type of adhesive used.

Control of bond-line thickness can be achieved by mechanical means (i.e., separation of adherends physically controlled by the bonding fixture), using thin wire spacers (e.g., stainless steel) inserted between the adherends or by ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1% by mass). Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness. It is essential that wire spacers used to control bond-line thickness are located well within the bonded area away from the specimen edges and regions of high stress concentrations (i.e., ends of joints).

When using glass beads to control bond-line thickness, the distribution of glass beads in the adhesive must be uniform and therefore glass beads should be thoroughly mixed into the adhesive. Mixing should take place before applying the adhesive to the adherend surfaces. Controlling the bond-line thickness of flexible adhesives joints is difficult due to the highly viscous nature of flexible adhesives. The preferred method is to use thin wire spacers, however work carried out at NPL showed that this method of bond-line control was not always reliable.

Curing adhesive

There are several key points that should be considered when curing adhesive joint specimens [10, 12, 14-17]:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint; as shown in Figure5. The tool can be held in place using heat resistant tape. Figure 6 shows a tool that was used to produce a consistent fillet for T-peel joints bonded with a paste adhesive. The tool can be fabricated from either aluminium or stainless steel coated with release agent.
- 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. Visual inspection should be carried out to ensure there is no air entrapment.
- The cure state of the adhesive layer in the adhesive joint should be similar to that of bulk adhesive specimens [14-15]. Failure to achieve similar thermal histories can result in significant differences in material properties. Differences between thermal histories will lead to differences in mechanical properties (See Chapter 2 [12]).
- Temperatures in the adhesive should be monitored throughout the cure cycle. It is recommended that trials be carried out on the adhesive joint using a thermocouple embedded in the adhesive in order to ensure that the temperature within the adhesive layer actually reaches the specified cure temperature.
- Due to differences in thermal mass, joint specimens may heat at different rates than bulk test specimens and therefore the final temperature of the adhesive joint at the end of the cure period can be significantly different to that of the bulk adhesive.

- For heat curing systems, the temperature of the specimen will lag behind the oven temperature, and it may therefore be necessary to elevate the oven temperature when curing joint specimens.
- Adhesives should be fully cured prior to conditioning and testing otherwise an 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, thus causing heat damage.
- Residual thermal stresses may be generated as a result of non-uniform (rapid) cooling, resin shrinkage and thermal expansion coefficient mismatch between the adhesive and adherend. As the joint is cooled down from the cure temperature, residual stresses are frozen in the material.
- As mentioned in Chapter 2, handling adhesives can be hazardous to human health, thus COSHH procedures should be followed to minimise operator exposure. Ovens and work areas should be suitably ventilated, ensuring minimal levels of hazardous vapours/gases in the work area.

It is recommended that the quality documentation should include details on the cure variables (i.e., temperature, pressure, heating and cooling rates and dwell times), and a record of equipment used for curing the adhesive joints and monitoring the temperature within the oven and adhesive joint (i.e., oven type and thermocouples).

Real-time monitoring of material property development in adhesives can be achieved using oscillatory rheometry or ultrasonic methods. Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA), can provide useful information relating to adhesive composition and final state of cure [17, 36-37] – see also NPL Measurement Good Practice Guide No. 32 [38].

Differential scanning calorimetry (DSC)

DSC is used to determine the heat flow associated with material transitions as a function of time and temperature or changes in heat capacity using minimal amounts of material. The technique provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions caused by phase changes, melting, oxidation and environmental degradation. The technique involves slowly heating a small sample (typically 5 mg to 10 mg) of material and measuring the heat absorbed or emitted by the sample as a function of temperature compared to a reference material. ISO 11357 [36] specifies methods for the thermal analysis of polymeric materials, including composite materials using DSC.

DSC can be used to measure the lowering of T_g with increasing moisture content for neat resins (including adhesives) and fibre-reinforced polymer composites. The technique has many advantages, including:

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

The disadvantages of DSC include:

- Incorrect transition may be assigned to T_g
- T_g is not detectable for all materials

Heating rates of 10 °C/minute or 20 °C/minute are typically used for T_g determination [38]. The reported T_g is generally independent of heating rate, however this is not the case for the first order transitions. A higher heating rate will broaden such transitions (e.g., curing or melting), which will reduce the accuracy of analysis.

Dynamic mechanical analysis (DMA)

DMA (or dynamic mechanical thermal analysis (DMTA)) enables the determination of transition temperatures, storage modulus and loss modulus of the sample over a wide range of temperatures (-150 °C to 800 °C), frequencies (0.01 to 200 Hz) or time, by free vibration, and resonant or non-resonant forced vibration techniques (ISO 6721 [37]). Flexural samples are popular, whereby the sample is mounted on a clamp and then subjected to sinusoidal changes in strain (or stress) while undergoing a change in temperature. DMTA is suitable for adhesives and laminated composites with stiffness values ranging from 1 kPa to 1,000 GPa.

Tension, compression, and flexure loading configurations can be employed. Three-point flexure specimens are typically 40 mm in length, 10 mm wide and 2 mm thick. Specimens can be up to 50 mm in length, 15 mm wide and 7 mm thick. DMTA can be used to measure heat distortion temperature, and thermal expansion and contraction (i.e., coefficient of thermal expansion) under dynamic or isothermal heating conditions. Thermal transition behaviour can be used as a quality assurance technique for determining the extent of resin cure.

The technique has many advantages, including:

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

The disadvantages of DMTA include:

- Poor reproducibility of modulus
- Each test is relatively time consuming

The selection of heating rate will have a significant influence on the results from DMTA [38]. The recommended heating rate is 3 °C/minute for routine testing. A lower heating rate reduces temperature variations within the specimen, while a higher heating rate allows quicker testing of specimens. It is difficult to make general recommendations for the applied strain on the specimen. The major requirement is that the strain should be in the linear elastic region for the material. Ideally, this should be determined for the material by determining the modulus at a range of strains and determining the elastic limit from the results. In practice, a strain of less than 0.5 % should be suitable for most materials.

General comments on thermal analysis techniques

The analysis of material by any 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 in a thermal analysis technique. 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. If the difference in T_g is greater than 5 °C between the first and second runs, then a third run is recommended. It is important that the material used for DSC and DMTA samples is representative of the adhesive within the bonded structure. The suggested method for preparing samples is to place the adhesive between two flat, smooth, parallel metal adherends (i.e., stainless steel), which have been coated with a thin layer of lubricant or release agent to prevent adhesion.

Quality assurance

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 (see [9]). 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 techniques are available for the non-destructive inspection of adhesive joints:

- Ultrasonic C-scan
- X-radiography
- Thermography

Note 4: There is no NDE technique that can provide a quantitative assessment of joint strength. Ultrasonic C-scan technique is particularly suited to the detection of planar type defects (e.g., debonds and delaminations) normal to the incident beam. Voids and porosity in the adhesive and adherends are also detectable. The minimum size of voids and delaminations detectable with C-scan is approximately 2 mm. The technique is not suitable for detecting surface contaminants (e.g., oils and grease).

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

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

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

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

Specimens should be stored in a desiccator (unless otherwise specified) to prevent moisture ingress, which can degrade the adhesive joint. Adhesive joints have been known to deteriorate under standard laboratory conditions (10% strength loss within 3-6 months). It is recommended that an accurate record be maintained for all batches of adhesive joints.

Documentation should include details on date of manufacture, precursor materials (i.e., adhesive and adherends), description of method of joint fabrication and assembly (including processing variables - curing time, temperature, and pressure), surface preparation and specimen dimensions (including width, bond length, adhesive layer and adherend thickness).

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Chapter 5

Preparation and assembly of adhesive joints

- Test Machine and specimen alignment
- Gripping of specimens
- Strain and displacement measurement techniques
- Mechanical testing
- Cyclic loading
- Creep
- Environmental conditioning and testing
- Conclusions

Introduction

This chapter considers the affect of test parameters (i.e., test machine alignment, load train stiffness, methods of gripping test machines, accuracy of load and displacement transducer) on the accuracy and reliability of strength and long-term performance of adhesive joints. Guidance is provided on the main factors that need to be controlled when carrying out mechanical testing. Consideration is given to various loading modes (i.e., static, cyclic fatigue and creep) and environmental conditions (i.e., elevated humidity and temperature). Appendix 1 provides a summary of commonly used adhesive joint test methods and related standards [13].

Test machine and specimen alignment

The test machine should have high lateral rigidity and accurate alignment between the upper and lower gripping faces. The load train should be as short and as stiff as possible (i.e., no universal joints included). If the grips are articulated, as in the case of universal joints, then the specimen may be subjected to large bending and twisting loads, resulting in reduce joint strength. Avoid eccentric acting forces. Small lateral (1 to 2 mm) or angular (1 to 2 degrees) offsets in the loading train can lead to additional shear and bending stresses, resulting in premature joint failure. It is worth noting that the slope of the load-displacement response can be similar for poor and well-aligned specimens.

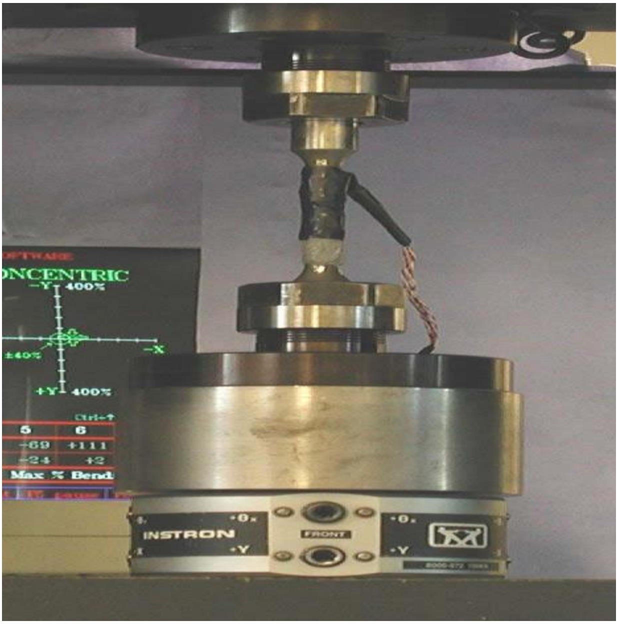

Figure 8. Alignment specimen with strain gauges

It is recommended that the alignment of the test machine and the test specimen be checked at the centre of the gauge length using a strain gauged coupon specimen [39-40]. Alignment specimens can be in the form of a rectangular or circular bar (see Figure 8). These specimens need to be accurately machined to ensure errors in parallelism are < 0.2 mm/m and in concentricity (lateral offset) of 0.03 mm [39]. Strain gauges are bonded to the surface of the alignment specimen to monitor alignment and bending strains. Bending strains should be less than 3 to 5% of the average axial strain.

Use a device to ensure that the specimens are positioned in the grips in a repeatable manner. An alignment fixture can also be included in the loading train to minimise angular and lateral offset between the upper and lower machine grips or loading rods. The alignment cell is attached to the upper or lower crosshead of the test frame; whichever is the most convenient. Commercial alignment cells are available that allow lateral movement, tilt and rotation of the machine grip or loading rod (see Figure 8).

Gripping of specimens

Grips for holding test specimens to be loaded in tension should be attached to the test frame so that the major axis of the test specimen coincides with the direction of pull through the centreline of the gripping assembly. The centre line of the specimen should be aligned with the axis of the loading fixtures to avoid bending and asymmetric loading. It is important than when loading test specimens in the grips that no lateral or angular offset is introduced to the specimen. Avoid rotating the grips during gripping operation. If one of the grips is articulated, this should be tightened first to prevent the specimen being subjected to large bending and twisting loads during tightening. Care should be taken to avoid axially stressing the specimen whilst the grips are being tightened. Any pre-stressing of the specimen should be kept to a minimum. Grips should be slowly tightened with any induced loads removed by progressively adjusting the crosshead position. The applied load on the specimen should be zero at the onset of testing. It may be necessary to use a device (i.e., metal spacer) during the test set-up to ensure good alignment and repeatable test results, as often the specimen width is less than the width of the mechanical grips.

Manual or servo-hydraulic grips can be used to hold specimens during testing. Wedge-action grips are recommended as the lateral force (i.e., pressure) applied to the test specimen in the gripping regions increases as the axial load applied to the specimen increases. Gripping pressure should be sufficient to prevent specimen slippage throughout the duration of the test, but not excessive to initiate failure of the specimen at the grips. For cyclic loading, it is essential that fretting in the gripped region be prevented to avoid the possibility of premature failure.

Strain and displacement measurement techniques

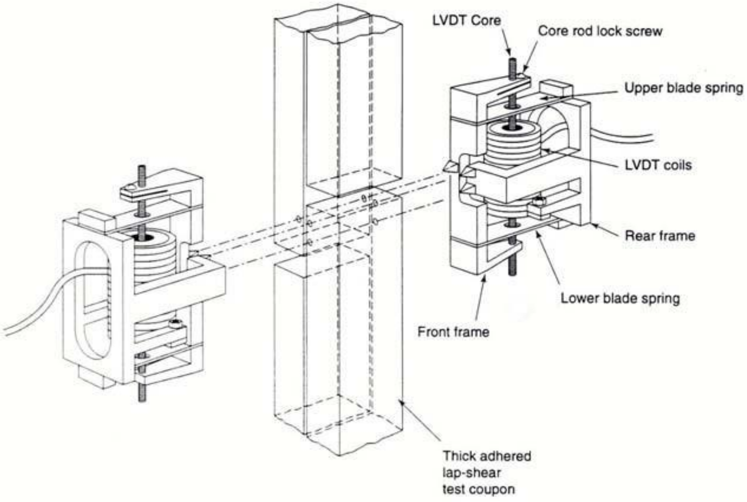

Figure 9. TAST specimen with extensometers for measuring shear deformation

Several contact and non-contact techniques are available for measuring strain and displacement. This section considers the use of contact extensometers, video extensometers, electronic speckle pattern interferometry (ESPI), strain gauges and crosshead movement for measuring strain and displacement under ambient and hostile environments, and static and cyclic fatigue loading conditions [10, 19]. Apart from strain mapping techniques (i.e., ESPI), the measured strain will be an average strain in the bond-line. Also, strain gauges only measure strain at the location of the gauge.

Contact extensometers

Contact extensometers are the preferred method for measuring strain and displacement, and hence stiffness of bonded joints. It is recommended that two extensometers, attached to opposite faces of the specimen, be used to measure displacement [10]. Any bending of the specimen will be apparent from diverging displacement readings. It is recommended that the individual transducer readings be recorded so that the quality of the test data can be checked. Errors due to minor bending are minimised by taking the average measurement of the two displacement transducers. To minimise inclusion of adherend deflection in the measurement the contact points should be as close to the bond layer as possible.

Figure 9 shows two extensometers attached to a thick adherend shear test (TAST) specimen for measuring shear deformation. The three-point contact minimises rotation of the extensometers. Knife-edged tensile extensometers, as described in reference [10], can be used if extensometer straddles the bond-line. The deformation of the adherends needs to be accounted for when analysing the data, but where the stiffness of the adherends is very much greater than that of the adhesive layer then corrections may be minimal.

Where adherends are flexible, it is advisable to support the weight of the extensometer because allowing the extensometer to hang unsupported from the specimen may cause bending and introduce contact stresses. The contact forces should be sufficient to prevent slippage between the extensometer and the specimen, but not large enough to cut or nick the specimen surface causing the specimen to fail prematurely. It may be necessary to remove extensometers attached to a specimen prior to failure to prevent the possibility of the extensometer sustaining damage during failure. Failure can be a violent event, releasing considerable energy, thereby damaging, or even destroying the extensometer.

An extensometer should be capable of measuring the change in gauge length with an accuracy of 1% of the applied displacement or better (i.e., equivalent to ± 0.5 μm for 10 % strain over a typical bond thickness of 0.5 mm). It is important that the extensometers can operate satisfactorily within the test environment (i.e., temperature and humidity), and that these devices are resistant to chemical attack when used in hostile environments. Precautions may need to be taken to insulate the leads to prevent moisture ingress.

Non-contact extensometers

Non-contact or optical extensometers (e.g., video extensometers) are available, which avoid contact damage and can be used up to failure, since there is no possibility of damage to the extensometer. Video extensometers are not particularly suited to measuring small strains (e.g., movements of a few μm), which limits their applicability to structural adhesive joints. Furthermore, measurement is normally only possible at one side of the joint so that bending cannot be evaluated. However, video extensometers have been used in tests on joints bonded with flexible adhesives where deflections are larger. Measurements of joint stress-strain curves have been in reasonably good agreement with contact extensometer results. Some systems provide capabilities for dot location measurements (facilitating strain mapping).

The technique relies on a remote camera monitoring the separation of two marks or lines inscribed on the test specimen, which define the gauge length. The change in separation of the two lines is recorded throughout the test. The gauge marks should be approximately equidistant from the mid-point, and the measured distance between the marks should be measured to an accuracy of 1%, or better. Gauge marks should not be scratched, punched, or impressed on the specimen in any way that may cause damage to the specimen. It is advisable to ensure that there is a sharp contrast in colour between the specimen surface and the gauge marks. The lines should be as narrow as possible. There are no temperature restrictions as video extensometers can be located outside the test chamber.

Strain gauges

Currently there are no standard tests using strain gauges to monitor strain in the adhesive layer. However, structural monitoring capabilities, where strain-sensing devices are embedded in materials, are the subject of research in many organisations. Strain gauges can be attached to adherends and will measure the strain in these adherends. The usefulness of such measurements may be limited except in the cases where changes in joint performance are manifested in measurable changes in the adherend strain. One such application is back-face strain gauging of thin lap-shear joints where crack growth in the adhesive layer can be monitored through strains measured by gauges bonded to the external surface of the adherends at the overlap. Strain gauges are occasionally used for monitoring strain in bonded structures (e.g., four-point bending of sandwich structures).

Strain gauges are generally limited to the measurement of strains less than 10%. Biaxial rosettes are available for measuring longitudinal and lateral strains. Large strain gauges are preferable as alignment and handling is easier, and they average out local strain variations. Local strain variations can cause premature failure of the strain gauges. Correct alignment of strain gauges is important, as significant errors can be caused by careless application of the strain gauges to the specimen. Errors of 15% can occur from a 2° misalignment [41].

The adhesive used to bond the strain gauge should be capable of withstanding the test environment for the complete duration of the test [11]. Most adhesives are sensitive to moisture (and other chemicals), which can often preclude bonding prior to specimen conditioning. Moisture attack of an adhesive and strain gauges will occur from the top, edges and in the case of polymeric materials through the substrate beneath the gauge. The situation is exacerbated at elevated temperatures. It is therefore important to ensure that the adhesive selected for bonding the strain gauge and associated electrical wiring is suitably encapsulated.

The strain gauges are usually bonded to the specimen following moisture conditioning (i.e., immersion in water or exposure to humid environments). However, bonding the strain gauge to the specimen may require heat and pressure, which will induce drying out of the conditioned specimen. To avoid drying out, room temperature curing anaerobic adhesives have been used and have proved satisfactory for bonding strain gauges to moisture conditioned specimens. For hot/wet conditions, a high temperature anaerobic adhesive can be used provided the application temperature does not result in thermal damage to the adhesive joint (i.e., adhesive and adherend). Although anaerobic adhesives have good moisture, solvent and temperature degradation resistance, these adhesives are known to attack certain plastics. Hence, precautions need to be taken when selecting these materials for use with plastics or fibre-reinforced polymer composites. Cyanoacrylates (or super glues), which are sensitive to surface moisture and low pH levels, are unsuitable for environmental testing. Strain gauge manufacturers can provide information on adhesive selection and procedures for protecting strain gauges.

For cyclic loading, it is essential that the fatigue life of the strain gauges, over the operating strain levels, should be well more than the life expectancy of the test component. Autogenous (self-generated) heating can degrade the mechanical properties of the adhesive bond between strain gauges and the specimen. This can result in small errors in strain measurement, thus requiring correction of the data to account for the temperature rise. Measurements should also be carried out to determine the magnitude of creep within the strain gauge adhesive.

Crosshead displacement

An approximate measurement of strain and hence stiffness can be obtained from measuring the crosshead displacement of the test frame [10]. The strain is the ratio of crosshead displacement and the initial grip separation. Hence, any slippage within the loading train will produce errors in the strain measurement. The strain values obtained from crosshead measurements will differ from the actual strain in the central region of the specimen.

Stiffness measurements directly obtained from the crosshead movement need to be corrected account for the stiffness of the loading train. This can be a difficult task as the specimen size and geometry, and the deformation behaviour of the specimen need to be considered [14]. Given the small adhesive layer deflections that occur even at large strains owing to the thin bond-lines, the accuracy of strains determined using crosshead displacements must be considered suspect and used only for qualitative purposes.

Electronic speckle pattern interferometry (ESPI)

Electronic speckle pattern interferometry (ESPI) is a non-contact technique capable of measuring and monitoring non-uniform strain fields at high resolution. The system can measure the deformation and thus the strain under mechanical and/or thermal loads along the three material axes (i.e., 3-D strain measurement). ESPI systems can measure local deformation with a resolution of 0.1 μm, equivalent to 200 microstrain for a 0.5 mm thick bond. The technique needs minimal specimen preparation and can inspect areas ranging from 25 mm^2 to 600 mm^2, but capital outlay for equipment is generally prohibitive for most test facilities. The technique can be used to measure strain distributions in complex geometries, and for checking finite element analysis. Further details of the technique with illustrated case studies are given in reference [42]. Interferometry techniques are not routine and are thus unlikely to be suitable for mass screening programmes. Similarly, the technique may not be suitable for cyclic testing.

Mechanical testing

The strength or stiffness measured in mechanical tests form only part of the useful data that can be obtained. The mode of failure (i.e., cohesive failure in the adhesive or interfacial failure) and degree of cohesive and adhesive failure should be recorded for each specimen. Optical microscopy or scanning electron microscopy may be required to analyse the fracture morphology, particularly if failure is close to the adhesive-adherend interface. Standards often recommend that results from specimens where failure occurs in the adherend should be discarded and replaced. However, unless the reason for adherend failure can be traced to inherent flaws in the test adherend that are unlikely to be replicated in the bonded component then these tests can still provide some valuable information.

The effects of test environment need to be considered. The operator should ensure that the equipment used to load and monitor (i.e., extensometers and load cell) the specimen is unaffected by the test environment. It may be necessary to thermally insulate load cells and use molybdenum grease to ensure moving parts in test fixtures do not seize whilst testing. It is recommended that the loading fixtures be fabricated from stainless steel to avoid environmental attack.

Number of test specimens

A minimum number of five specimens should be tested for each batch of specimens. If a greater precision of the mean value is required, then the number of specimens tested should be increased (see ISO 2602 [43]).

Specimen dimensions

Specimen dimensions and crack length need to be accurately measured, as small measurement errors can translate into large variations in strength or fracture toughness, particularly if the calculation includes squares or cubes terms of the measured parameter (see Table 3). The uncertainty in strength or fracture toughness calculation is compounded where there is more than one term (i.e., width, thickness, and crack length, etc), each with an associated uncertainty. The bond line thickness, being a very small dimension, tends to be the dimension where accuracy and precision of dimensional measurement are most critical. Measurements at different locations should be carried out to check the uniformity of bond-line thickness.

Vernier callipers or travelling microscope are recommended for measuring specimen width and bond length, and a micrometer or travelling microscope for measuring specimen thickness. A travelling microscope should be used to measure crack length.

Dimensional Error	Linear Error	Squared Error	Cubed Error
± 1	± 1	± 2	± 3
± 5	± 5	± 10	± 16
± 10	± 10	± 21	± 33

Table 3. Associated uncertainty with measurement error (percentage)

Speed of testing

Polymeric adhesives are viscoelastic materials; that is their mechanical properties (strength and stiffness) are sensitive to the rate at which they are loaded (or more accurately the strain rate). Standards relating to testing of adhesive joints infrequently specify the speed or rate of testing required, but instead specify that the test joint be loaded at a constant stress or strain rate and to ensure that failure is achieved in a prescribed period (typically 60 to 90 seconds). This introduces a degree of subjectivity into the selection of test conditions. Where specimens have different bond-line thickness then strain rates may vary between specimens leading to greater uncertainties in the results. For comparative measurements, it is recommended that all joints be tested at comparable strain rates. This can be achieved by ensuring the ratio of test speed over bond thickness is approximately the same for each test specimen. The standard requirement to fail the joint in the prescribed time is convenient for testing but may not impose strain rates relevant to the design requirement.

A series of trials to failure are recommended to ascertain the test speed required to meet the strain rate or time limit specified in the standard. It is therefore advisable that 4 or 5 additional specimens be prepared with each batch of specimens for this purpose. It should be noted that the small gauge length, due to the thin bond line, leads to relatively high rates of strain in the adhesive at moderate test speeds. This needs to be considered when comparing joint specimen tests with bulk specimen properties.

Adherend property and geometric effects

Altering the geometry of a bonded joint will invariably cause changes to occur in the stress and strain distribution within the adhesive layer. These differences can have a profound effect on the stress concentrations and consequently the load-capacity and long-term performance of the joint. Currently, there are no well-established design procedures for predicting failure behaviour or relating changes in material and geometric parameters to joint strength. Finite element analysis (FEA) enables the prediction of the effects of changing joint geometry parameters on stress/strain levels in the structure. Thus, joints can be designed to minimise stress concentrations. With accurate material properties data, relevant materials models, and reliable failure criteria the strength of any joint under any stress state could be predicted. However, the state of the art is not at this stage yet. Research on this is continuing.

Adherend Thickness/Overlap Length	Load/Width (N/mm)
CR1 Mild Rolled Steel	
1.5 mm/12.5 mm	334 ± 11
2.5 mm/12.5 mm	354 ± 10
2.5 mm/25.0 mm	428 ± 38
2.5 mm/50.0 mm	633 ± 63
5251 Aluminium Alloy	
1.6 mm/12.5 mm	191 ± 14
3.0 mm/12.5 mm	325 ± 28
6Al-4V-Titanium Alloy	
2.0 mm/12.5 mm	457 ± 52
Unidirectional T300/924 Carbon/Epoxy	
2.0 mm/12.5 mm	369 ± 41
Plain Woven Fabric (Tufnol 10G/40)	
2.5 mm/12.5 mm	275 ± 28
2.5 mm/25.0 mm	454 ± 27
2.5 mm/50.0 mm	511 ± 32
5.1 mm/12.5 mm	327 ± 27

Table 4. Failure load per unit width for AV119 epoxy single-lap joints

Bond-line Thickness (mm)	Bond Thickness (mm)		
	12.5	25.0	50
0.31	585 ± 55	1,162 ± 21	1,988 ± 215
0.52	562 ± 36	1,344 ± 49	2,199 ± 166
0.71	498 ± 24	1,050 ± 59	1,689 ± 162
1.10	455 ± 29	880 ± 105	1,707 ± 27

Table 5. Failure load (N) for mild steel/elastomer single-lap joints

The results presented in Tables 4 and 5 clearly indicate that the joint strength is dependent on the adherend material, adherend thickness, bond-line thickness and bond length. Table 4 compares the failure load per unit width (N/mm) for various single-lap joint configurations and materials bonded with an epoxy adhesive [44-46]. Table 5 demonstrates the sensitivity of single-lap strength on bond-line thickness and bond length. The joints consisted of mild steel sections bonded with an elastomer adhesive. Small variations in bond-line thickness can result in significant changes in bond strength. For comparative studies, careful consideration should therefore be given to ensuring that the stress and strain distributions (i.e., maximum peel and shear stresses at the ends of the joint) for different systems are at least similar.

As a first approximation for single-lap joints, adherends with similar products of stiffness and thickness should form joints with similar strengths if failure is cohesive in the adhesive layer, i.e.:

$$E_1 t_1 = E_2 t_2$$

w here E is the longitudinal stiffness of the adherend, t is the adherend thickness, and subscripts 1 and 2 refer to materials 1 and 2.

For the measurement of the adhesive properties, steel adherends are recommended because of the material's high stiffness. For ambient tests, suitable steels are XC18 and 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.

Cyclic loading

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.

Creep

Creep tests are performed to assess the extension of joints under load to predict long-term behaviour or to assess the long-term strength of joints under load. The first requirement needs high precision extensometry to monitor joint extension and the tests must be performed under stable environmental conditions (temperature and humidity) to avoid artefacts in the measurement. These tests could, in theory, be performed using any of the loading options outlined below but for highest accuracy options 1 or 2 would be preferred. This section addresses the requirements of creep failure (or creep rupture tests).

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 (Figure 10); and
4. Self-stressing fixture where specimens are placed in either a tube equipped with a calibrated spring system (Figure 10) for loading specimens or a circular ring.

Figure 10. 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 10), 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. 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 10 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 tested. Typically, these are between 10 and 50% of the short-term strength of the joint. It is generally 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).

Environmental conditioning and testing

To determine the effectiveness of different adhesive systems, processing variables and surface treatments, it may be necessary to expose adhesively bonded joints to various environmental and loading conditions that simulate actual service conditions. The resistance of the bonded structure to degradation agents often becomes apparent within a short period. In some circumstances only a few hours of exposure may lead to catastrophic failure or seriously compromise the structural integrity of the joint (see also Measurement Good Practice Guide Number 28 [18]).

The two predominant factors in climatic exposure are humidity and temperature. The severity of these two factors will depend on geographical location and need to be considered in assessing allowable strengths. A list of possible service environments is listed below:

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

Engineering structures are exposed to various combinations of these environments in service, often resulting in complex synergistic degradation of the material. It is recommended that when comparing the effects of surface treatments, adhesive systems, and processing variables on the durability of joints in hostile environments, that all specimens are prepared and conditioned together to account for any variability in the conditioning environment. Also, control specimens are recommended to check improvements in joint performance.

A listing of fatigue, creep and environmental standards are presented in NPL Report CMMT(A) 61 [13] along with a summary of environmental conditioning procedures, covering the scope of the standard, relevant materials, environmental conditions, and specimen requirements. Several NPL reports [46-50] cover the use of statistical analysis for analysing durability data.

Degradation agent

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

Moisture (water) degradation is probably the major cause of in-service failure in bonded structures. The ubiquitous nature of water combined with the ability to penetrate the adhesive structure poses considerable problems. With good design it is possible to significantly reduce the rate of moisture diffusing to the adhesive/adherend interfaces. This problem is further exacerbated at elevated temperatures and mechanical stress. Hot and humid environments can often cause rapid loss of strength in metal-epoxy joints within a short duration (i.e., 2 years) with catastrophic consequences. Failure invariably occurs at the adhesive/adherend interface.

The major cause of strength loss in adhesively bonded metal joints is associated with interfacial degradation through water-substrate interaction rather than weakening of the adhesive. Water can degrade the strength of adhesive joints through hydration of metal oxide layers. Corrosion products, such as rust, at the interface are considered a post-failure phenomenon. The presence of water in epoxy adhesives results in plasticisation (essentially softening) of the polymeric material. At temperatures below the glass transition temperature T_g, polymer property reduction is reversible upon dehydration, whereas above T_g, matrix properties are permanently degraded. Surface treatments, such as phosphoric acid anodisation and organosilane primer coatings will bestow joints with improved moisture resistance.

Accelerated conditioning

The degree of degradation that occurs in adhesive structures when in service is linked directly with the amount of moisture absorbed. The moisture absorption kinetics of polymer systems will differ widely and change with physical ageing. The approach often adopted, is to define a constant relative humidity environment that will produce a moisture level that is representative of a bonded structure that has been exposed to a real-life environment at a particular geographical location. The variability of a natural environment, that is daily, monthly, or seasonal changes in temperature and humidity are known to be a major factor in determining both the final moisture equilibrium level in the material and the distribution of moisture in the outer surface layers. The military consider the worst worldwide environment to be represented by a relative humidity of 85% and a temperature of 70°C.

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

The usual approach used to accelerate moisture uptake is to increase the diffusivity of the adhesive by elevating the temperature of the conditioning environment. However, there is a temperature limit to which most polymer resins can be raised without affecting a change in the mechanism by which moisture is absorbed. Above this critical temperature, there is a strong possibility that the degradation mechanism is altered.

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

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

Note 5: Although accelerated ageing is widely used, a full understanding of the effects is not yet available and there is no agreement within industry as to a satisfactory accelerated test.

Chapter 6

Conclusions

Conclusions

The two most important criteria when selecting a test method are the availability of a standard test method and the ability of the test method to produce consistent and reliable engineering data for a range of service conditions. When planning a test strategy the following should be considered:

- Selection of appropriate test for ranking and screening, design data and QA/QC (i.e., conformance to design standards/codes) purposes.
- Knowledge of existing standards/legislative requirements.
- An understanding of the material (i.e., adherend and adhesive) characteristics, test method and the test data generated. An awareness of the mechanical and physical properties of the adherend, adhesive and the joined system are invaluable in understanding the test results and for trouble-shooting.
- Cost of fabrication and testing/test facility requirements.
- Knowledge of the factors that affect reliability of the test data.

Note 6: Many of the test methods/standards are limited to providing comparative data on adhesive systems and surface treatments, and are unsuitable for generating design data.

Independent of the test method selected, correct surface preparation is essential for ensuring both initial adhesion and long-term joint durability. Although the guide provides advice on the surface preparation of the commonly used structural materials, it only covers the main steps in each process. **It is essential that expert advice from the adhesive manufacturer is obtained and that the detailed requirements specified by the manufacturer are completely satisfied**. Special care is needed to ensure that bond-line thickness is uniform and free of voids, and that the method used to control the bond-line thickness does not compromise the strength or durability of the joint. Assume the degradation process is irreversible and commences on completion of the cure cycle.

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Appendix 1

Test methods

List of test Methods

Material Property	Standard/Test Method
Bulk Plastic/Adhesive Tests	
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
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	ISO 4587, ASTM D3528, BS 5350-C15
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 D3167
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

Adhesive joint testing

The four main loading modes of bonded joints are (see Figure A1.1):

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

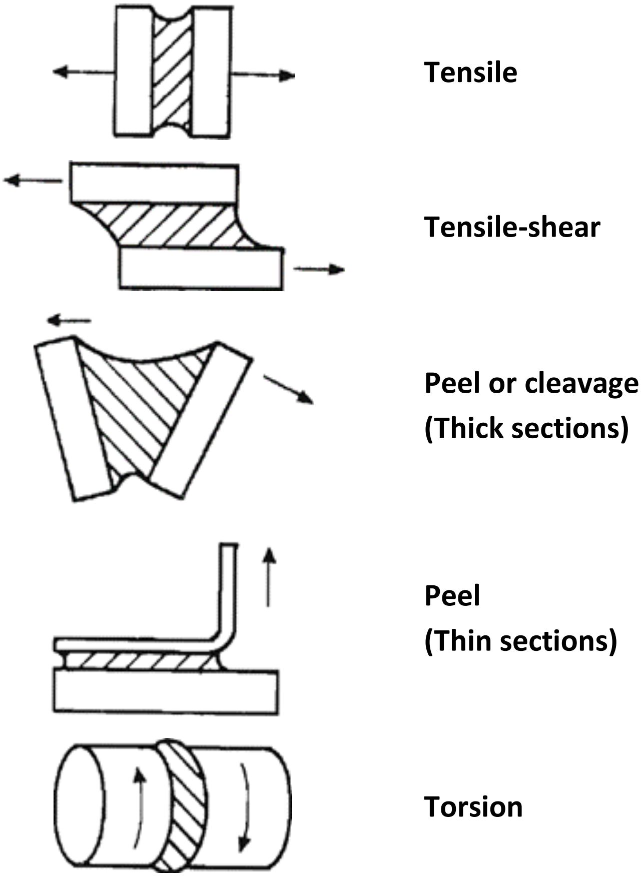

Figure A1.1. Basic loading modes experienced by adhesive joints

Tables A1.2 to A1.19 in Appendix 1 provide detail information on adhesive joint test methods.

Tension test methods

Advantages	Disadvantages
Yields tension, torsion, and compression Stiffness/strength Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for environmental and cyclic testing (QA only)	Qualitative adhesive property data only Strain measurements difficult High shear/peel stresses at bond-line edges Strength sensitive to spew fillet Sensitive to specimen misalignment Limited to thick and rigid adherends Failure attributed to peel stresses Special bonding fixture required

Table A1.2. Tensile butt joint test method (see Figure 3)

Peel test methods

Advantages	Disadvantages
Yields peel strength Straightforward/economic • Specimen fabrication • Testing • Data reduction Discriminates between surface treatments and adhesives Suitable for In-situ environmental (QA only) and cyclic testing	Limited to thin flexible adherends Not suitable for generating design data Large uncertainties in measurements No allowance for large adherend deformation Sensitive to fillet dimensions

Table A1.3. T-Peel test method (see Figures 6 and A1.2)

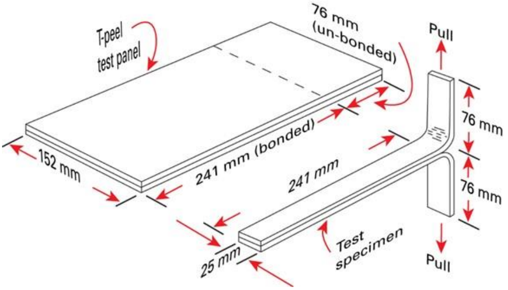

Figure A1.2. T-peel specimen

Advantages	Disadvantages
Yields • Peel torque • Skin (facing) stiffness of honeycomb sandwiches Laminated or honeycomb sandwich structures Straightforward/economic • Testing • Data reduction	Special test fixture required Limited to: • Laminated or honeycomb sandwich structures • Low stiffness skins (facings) Large and expensive specimens Comparative data/service correlation unknown Unsuitable for fatigue testing Unsuitable for in-situ environmental testing

Table A1.4. Climbing drum peel test method (see Figure A1.3)

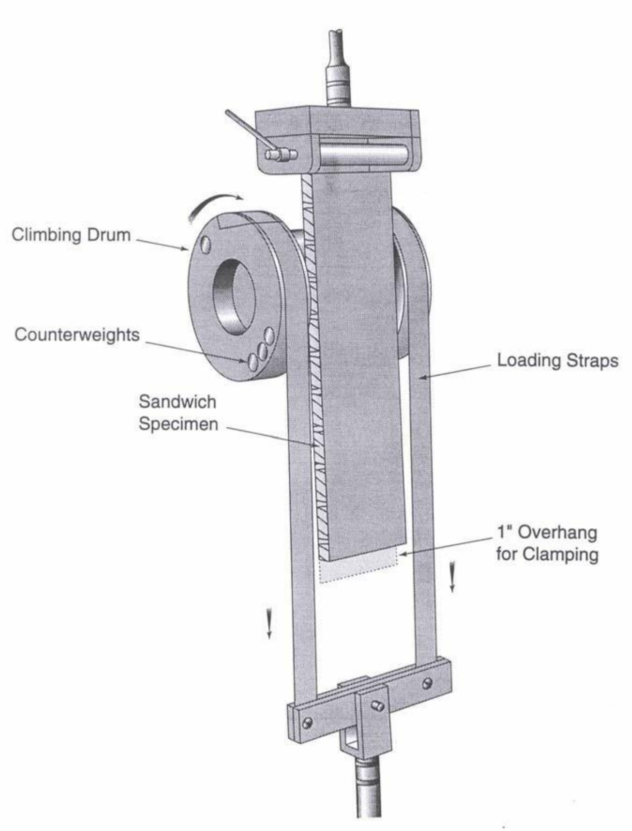

Figure A1.3. Climbing drum test of sandwich structure

Advantages	Disadvantages
Yields peel strength Compatible with metals and PMCs Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction	Special test fixture required Limited to rigid-to-flexible adherends No allowance for large adherend deformation Comparative data/service correlation unknown Unsuitable for fatigue testing Unsuitable for in-situ environmental testing

Table A1.5. Floating roller method (see Figure A1.4)

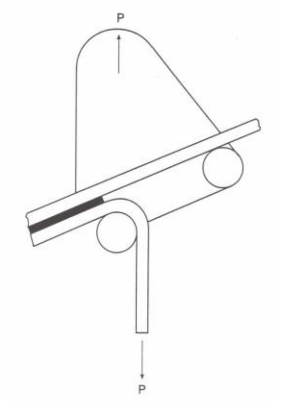

Figure A1.4. Floating roller method

Cleavage Test Methods

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

Table A1.6. Wedge cleavage or Boeing wedge test (see Figure A1.5)

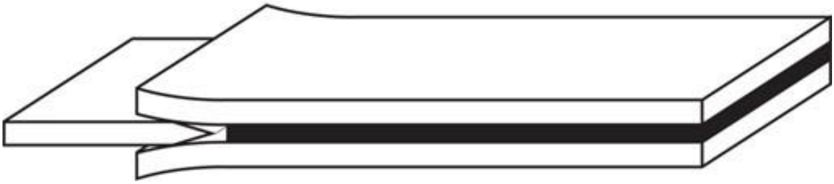

Figure A1.5. Wedge cleavage test

Advantages	Disadvantages
Yields fracture strength Compatible with metals and PMCs Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction Suitable for environmental/fatigue testing (QA only)	Special test fixture required (moderate cost) Special bonding fixture required Limited to rigid adherends Not suitable for generating design data

Table A1.7. Compact tension test (see Figure A1.6)

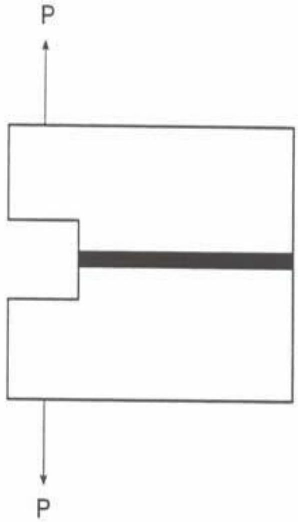

Figure A1.6. Compact tension test

Shear test methods

Advantages	Disadvantages
Yields "apparent" shear strength	Geometry dependent
Compatible with metals, plastics, and PMCs	Limited to rigid adherends
Straightforward/economic	Not suitable for generating design data
• Specimen fabrication	High shear/peel stresses at bond-line ends
• Testing	Moderate to high bending moments
• Data reduction	Failure attributed to peel stresses
Suitable for environmental and cyclic testing (QA only)	Special bonding fixture required
	Large uncertainties in measurements

Table A1.8. Single/double-lap joint tests (see Figures 2 and A1.7)

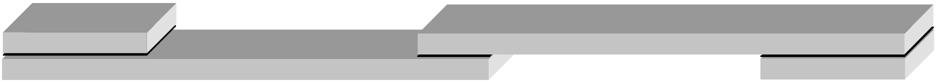

Figure A1.7. Single-lap test specimen

Advantages	Disadvantages
Yields "apparent" shear strength	Geometry dependent
Compatible with metals, plastics, and PMCs	Limited to rigid adherends
Straightforward/economic	Not suitable for generating design data
• Specimen fabrication	Elevated shear and peel stresses at bond-line ends
• Testing	Moderate to high bending moments
• Data reduction	Failure attributed to peel stresses
Suitable for environmental and cyclic testing (QA only)	Special bonding fixture required
	Large uncertainties in measurements

Table A1.9. V-notched beam shear tests (see Figure A1.8)

Figure A1.8. V-notched beam shear test

Advantages	Disadvantages
Bulk Resin	**Bulk Resin**
Shear modulus attainable for all polymeric materials	Shear modulus attainable for all polymeric materials
Shear strength attainable for thermoplastics	Shear strength attainable for thermoplastics
Adhesive Joints	**Adhesive Joints**
Yields shear modulus and strength	Yields shear modulus and strength
Suitable for compliant adhesives	Suitable for compliant adhesives
Additional Points	**Additional Points**
Low material requirements	Low material requirements
Data reduction is straightforward	Data reduction is straightforward
Suitable for use under environmental conditions Suitable for creep testing	Suitable for use under environmental conditions Suitable for creep testing

Table A1.10. Arcan shear tests (see Figure A1.9)

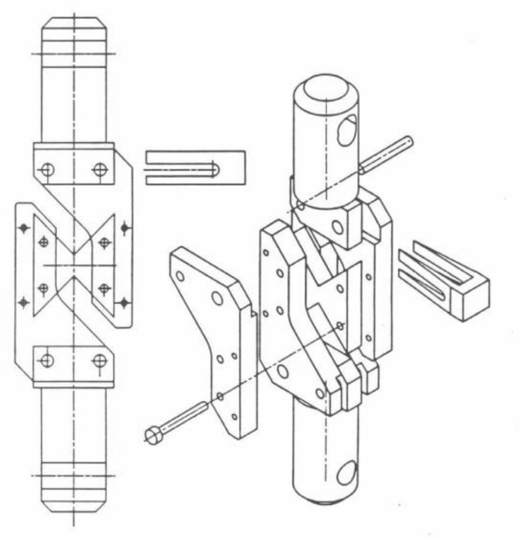

Figure A1.9. Arcan shear test

Advantages	Disadvantages
Bulk Resin Shear modulus attainable Shear strength attainable for thermoplastics **Adhesive Joints** Yields shear strength and shear modulus Compatible with metals and composites **Additional Points** Suitable for use under environmental conditions Suitable for cyclic/creep testing	**Bulk Resin** Strain gauges/extensometers required Brittle polymers - tensile failure occurs Axial strains need to be monitored **Adhesive Joints** Special bonding fixture required Non-uniform shear stress state Small strains - difficult to measure accurately **Additional Points** Accurate specimen machining required Torsion facility required Stress concentrations present at end grips Complex data reduction (non-linear stress/strain) No existing national or international standards

Table A1.11. Torsion butt tests (see Figure 3)

Advantages	Disadvantages
Yields shear strength and shear modulus Compatible with all metals, plastics, and PMCs Stress state relatively uniform over bond-line Straightforward/economic • Specimen fabrication (moderate cost) • Testing • Data reduction Suitable for environmental testing	Complex/expensive extensometers (2 off) required Special bonding fixture required Accurate specimen machining required Stress concentrations present at bond-line ends Stress analysis difficult Limited fatigue capability

Table A1.12. Thick adherend shear tests (see Figure 3)

Mode I Fracture Toughness Test Methods

Advantages	Disadvantages
Yields mode I fracture toughness Compatible with metals and PMCs Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for environmental and cyclic testing (QA only)	Complex/expensive extensometers (2 off) required Special bonding fixture required Accurate specimen machining required Stress concentrations present at bond-line ends Stress analysis difficult Limited fatigue capability

Table A1.13. Double cantilever beam (DCB) tests (see Figure A1.10)

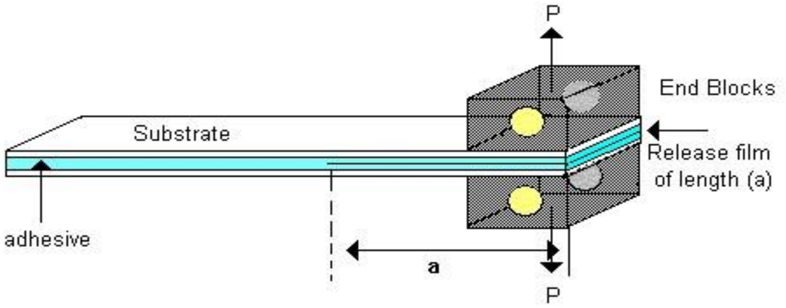

Figure A1.10. Double cantilever beam (DCB) specimen

Advantages	Disadvantages
Yields Mode I fracture toughness Compatible with metals Adaptable to thin adherends Constant compliance Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for cyclic/environmental testing (QA only)	Limited to rigid adherends/brittle adhesives Flexural test fixture required Unsuitable for generating design data Analysis required to account for • Shear deformation • Large beam deflection Non-linear load-displacement Pre-cracking difficult Crack extension measurements difficult Moderate-large measurement uncertainties No existing national/international standard

Table A1.14. Tapered double cantilever beam (TDCB) tests (see Figure A1.10)

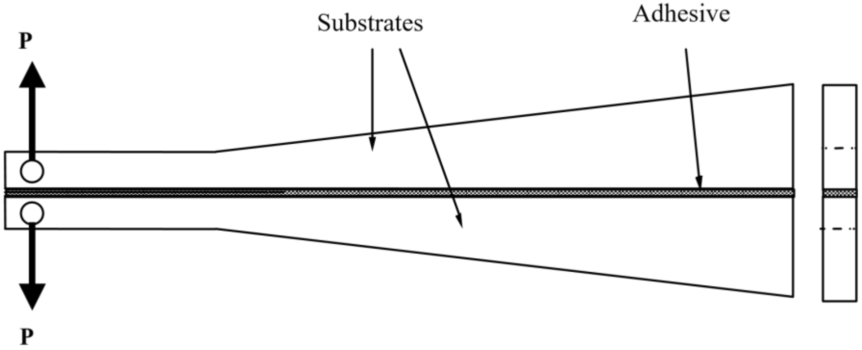

Figure A1.11. Double cantilever beam (DCB) specimen

Mode II fracture toughness test methods

Advantages	Disadvantages
Yields mode II fracture toughness Compatible with metals and PMCs Straightforward/economic • Specimen fabrication • Testing • Data reduction Suitable for cyclic/environmental testing (QA only)	Limited to rigid adherends Large specimens required Not suitable for generating design data Special test fixture required Special bonding fixture required Moderate to large uncertainties in measurements

Table A1.15. End loaded split (ELS) and end notched flexure (ENF) Tests (see Figures A1.12 and A1.13)

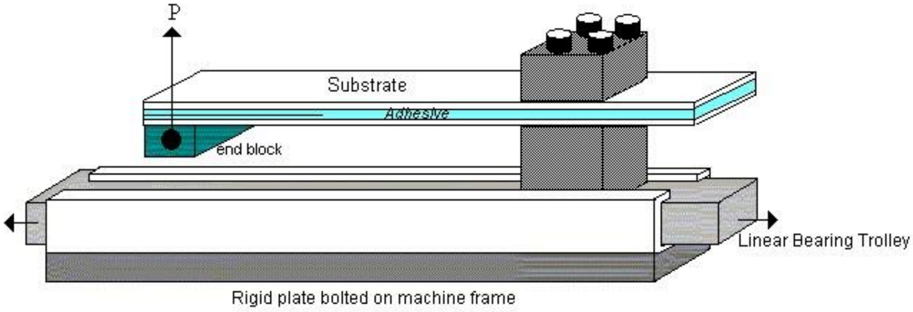

Figure A1.12. End loaded split (ELS) test

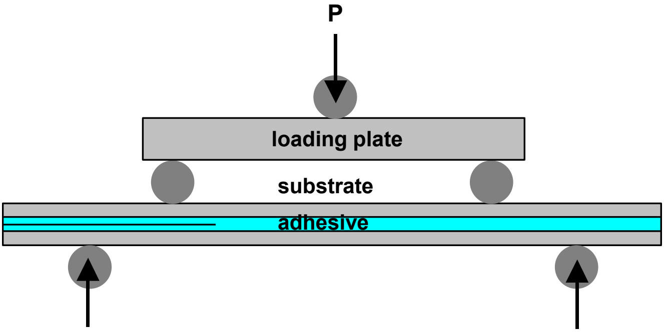

Figure A1.13. End notched flexure (ENF) test

Test Method	Tensile Butt Joint	T-Peel	Climbing Drum	Floating Roller Method
Mechanical Properties Obtained	Tensile strength/modulus	Peel strength	Peel strength/skin stiffness	Peel strength
Material Quantity Requirements per specimen	Low	Low	High	Low
Typical Specimen Dimensions (mm)	Diameter 15-25 mm Adherend thickness 12-15	Bond length 150 Width 25 Adherend thickness 0.5-1.0 Arm length 50	Long adherend 300 Short adherend 240 Width 25 Adherend thickness 0.5-5.0	Flexible adherend 250 Rigid adherend 200 Width 25 Adherend thickness 0.5-1.6
Materials Suitable for Testing	1 - 6	1, 4 and 6 Flexible-flexible adherend	Flexible-rigid adherend 1 - 6 and sandwich structures	Flexible-rigid adherend 1 - 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	High	Low - moderate
Cost of Testing/Specimen	Low	Low	Low - moderate	Low - moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Extensometer	None	None	Extensometer (2 off)
Test Equipment and Fixture Requirements	Universal test machine and end grips	Universal test machine and end grips	Special test fixture Universal test machine and end grips	Special test fixture Universal test machine and end grips
Fatigue performance	Limited	unsuitable	Unsuitable	Unsuitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Unsuitable	Unsuitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	Large uncertainty (> 30%)	To be determined	To be determined
Standards	ASTM D897 ASTM D2095 BS EN 15870	ISO 11339 ISO 8510-2 ASTM D1876	ASTM D1781 BS 5350-C13	ISO 4578 ASTM D3167

Table A1.16. Tensile and peel test methods

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

Test Method	Wedge Cleavage	Compact Tension	DCB	TDCB
Mechanical Properties Obtained	Fracture energy	Cleavage strength	Mode I fracture toughness	Mode I fracture toughness
Material Quantity Requirements/Specimen	Low	high	Low	High
Typical Specimen Dimensions (mm)	Length 200 Width 25 Adherend thickness 2	Length 25 Width 25 Adherend thickness 12	Length 356 Width 25 Adherend thickness 6.35	Length 240 Width 25 Adherend thickness 12.8
Materials Suitable for Testing	1	1 - 6	1 - 6	1 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Low	Moderate - High
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding and bonding jig	Surface preparation	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Travelling microscope or video camera	Extensometer for crack opening displacement	Travelling microscope or video camera	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Self-stressed	Universal test machine and loading fixture	Universal test machine and loading fixture	Universal test machine and loading fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	Large uncertainty	Moderate
Standards	ISO 10354 ASTM D3433	ASTM D1062 BS 5350-C1	ISO 25217	ISO 25217

Table A1.17. Cleavage and Mode I fracture toughness test methods

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

Test Method	Single-Lap	Double-Lap	V-Notched Beam	Arcan
Mechanical Properties Obtained	Shear strength	Shear strength	Shear strength/modulus	Shear strength/modulus
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 100 Width 25 Adherend thickness 2 Overlap length 25	Length 100 Width 25 Adherend thickness 12 Overlap length 25	Length 76 Width 20 Notch width 12 Adherend thickness 5	Length 52 Width 40 Notch width 12 Adherend thickness 6
Materials Suitable for Testing	1, 3 and 6	1, 3 and 6	1, 3 and 6	1, 3 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Moderate	Moderate
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	None	None	Shear extensometer Strain gauges	Shear extensometer Strain gauges
Test Equipment and Fixture Requirements	Universal test machine and loading grips	Universal test machine and loading grips	Universal test machine and loading fixture	Universal test machine and loading fixture
Fatigue performance	Limited	Suitable	Unsuitable	Suitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	Moderate	Low (30%)	Low - moderate (10-20%)	Low - moderate (10-20%)
Standards	ISO 4587 ASTM D1002 BS EN 1465 BS 5350-C3	ISO 4587 ASTM D3528 BS 5350-C13	ASTM D5379 (bulk resin)	None

Table A1.18. Shear test methods

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

Test Method	TAST	Torsion Butt Joint	Napkin Ring	ENF
Mechanical Properties Obtained	Shear strength/modulus	Shear strength/modulus	Shear strength	Mode II fracture energies
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 110 Width 5 Adherend thickness 26 Overlap length 5	Diameter 15-25 Adherend thickness 12-15		Length 356 Width 25 Adherend thickness 6
Materials Suitable for Testing	1- 6	1 - 6	1 - 6	1 and 6
Cost of Specimen Fabrication/Preparation	Low-moderate	Low-moderate	Moderate-high	Low
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig	Surface preparation Bonding and bonding jig
Specimen Instrumentation Requirements	Extensometers (2 off)	Rotary extensometer	Rotary extensometer	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Universal test machine	Universal test machine and loading fixture	Torsion test machine and loading fixture	Universal test machine and flexure fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	To be determined	Large uncertainty (30%)
Standards	ISO 11003-2 ASTM D5656	None	ASTM E229	None

Table A1.19. Shear and Mode II fracture toughness test methods

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

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Appendix 2

Round-robin adhesive joint evaluation exercise

Overview

A major concern with testing adhesive joints is the number of factors, not included in written standards, which can significantly affect the reliability (i.e., repeatability and reproducibility) of test data. Factors, including specimen and machine alignment, processing variables, surface treatment, adhesive storage and joint ageing need to be considered in addition to test and specimen parameters (e.g., displacement rate and dimensions) that are covered in national and international standards. It was evident from a statistical analysis of R-R data carried out in Performance of Adhesive Joints project PAJ3 on data from the ADH programme that the uncertainty associated with specimen preparation and testing has a substantial effect on the test results. The large scatter and disparity in results between test houses indicates that several additional factors need to be controlled in order to ensure reliable data. The situation is further exacerbated when tests are conducted in hostile environments.

One of the objectives of PAJex3 was to address these issues in a systematic manner in order to identify key variables. The approach was to run hands-on training sessions at the work place of participating organisations in conjunction with a R-R exercise, involving nine (9) participating organisations (including NPL). The industrial organisations, which participated in the R-R evaluation exercise, are listed below:

- Caswell Adhesives
- Corus
- Ford (UK)
- Honeywell Normalair-Garrett
- MIRA
- Permabond
- Stranger Science and Environment
- 3M (UK)

The R-R exercise consisted of three stages:

Stage 1: Six single-lap joint specimens manufactured at NPL were supplied to each organisation. The participants were requested to test the specimens in accordance with BS EN 1465 (Adhesives – Determination of Tensile Lap Shear Strength of Rigid-to-Rigid Bonded Assemblies) [33]. Permabaond supplied both the adhesive (two part epoxy E32) and bright mild steel test adherends (1.5 mm thick). Adhesive storage, handling and cure, and surface preparation were carried out in accordance with the procedures specified by Permabond (see Technical Requirements). Testing was carried out unsupervised by the participating organisations.

Stage 2: A second batch of specimens (6 off) was supplied to each participating organisation. Again, specimens were to be prepared and tested to the procedures used in Stage 1. The difference this time was that NPL personnel were present to witness the tests (i.e., observer status only) and to ask questions on in-house practices relating to the preparation and testing of adhesive joints. NPL provided a 1-hour presentation on “Good Practice for Preparation and Testing of Adhesive Joints”, which was followed by a discussion.

Stage 3: Adherend and adhesive materials were supplied to each participating organisation. Members were requested to prepare and test specimens, unsupervised, to the procedures used in Stages 1 and 2.

After each stage, data and specimens were returned to NPL for statistical analysis. The results of the R-R evaluation exercise along with observations and comments from participants are presented below.

The authors would like to acknowledge the contributions of Caswell Adhesives, Corus, Ford (UK), Honeywell Normalair-Garrett, MIRA (Motor Industry Research Agency), Permabond, Stranger Science and Environment, and 3M (UK) for their contribution to the R-R exercise and to this Measurement Good Practice Guide.

Technical requirements

Each participant received a technical information sheet for Permabond E32 (two part, room temperature curing epoxy adhesive) and copies of BS EN 1465 and Permabond Test Method 9 “Determination of Shear Strength of Adhesives Using Single Overlap Joints”. The technical information sheet provided with the adhesive includes a description of the two part adhesive, details on the physical properties of the adhesive (i.e., colour, viscosity, specific gravity and mix ratio of resin to hardener), recommended service temperature range, cure speed, typical lap shear strength, storage and handling instructions, and directions for use. Permabond Test Method 9 includes procedures for adherend surface preparation; test joint assembly and testing, and expression of results. In Stage 3, participants were requested to complete a form detailing surface preparation, bond-line thickness control and cure temperature (see below).

In addition, participants were requested to complete a test report for each stage of the R-R exercise. The report form identifies the test site, operator, test machine, load cell and calibration details, date of test, and temperature and humidity of the test laboratory. Operators were required to include specimen identify and details on specimen dimensions (i.e., width, overlap length and bond thickness), loading rate, maximum load, strength, mode of failure and difficulties encountered for each specimen.

Permabond test method 9

Introduction

The procedure (Determination of Shear Strength of Adhesives Using Single Overlap Joints (Issued: 30/10/00)) described below conforms to the basic requirements of BS5350: Part C5 - Single Overlap Technique. Its purpose is to ensure that adhesives conform to the specified strength requirements for their intended applications.

Apparatus

- Calibrated tensometer of suitable load capability.
- Controlled temperature environment as specified.
- Degreasing solvent (acetone, MEK, IPA) & paper towel.
- Grit blasting cabinet with 60/80 standard alumina (ex Vixen Surface Treatments) or rotary abrading table with both 100 and 600 grade 'wet and dry' discs.
- Bright mild steel test adherends of dimension 100x25x1.5 mm unless specified otherwise. (10 test pieces are required for F/V products and 6 for epoxy products).
- Suitable quantity of clean 1¼-inch fold-back clips.

Adherend surface preparation

- Examine the test adherends prior to preparation and dispose of any grossly distorted sections.
- Remove excess dirt and oil by wiping with a dry paper towel. Remove final film of oil with the aid of a solvent soaked towel.
- The end square inch of the adherends should be treated by one of the following methods:

  a) Grit blast with the air pressure set to 20 psi blast the adherends until a uniform finish is achieved.
  b) Abrade on a rotary table using firstly 100 grade 'wet and dry' followed by 600 grade to obtain a uniform, polished surface. Care must be taken to ensure the edges of the adherends are not bevelled or corners rounded during abrasion, as this will affect the ultimate performance of the adhesive under test. Immediately wipe the abraded surface dry to prevent corrosion.

- Degrease the prepared adherends with solvent and paper towel. Repeat this process if necessary to ensure all oil and contaminants are removed.

Test piece assembly

- To regulate the 12.75 ± 0.25 mm overlap, carefully mark the test-piece at the correct distance avoiding contamination of the bond area, then clearly mark the test-pieces with a suitable reference.
- Where the bond line regulation is specified, apply two pieces of wire to the overlap areas of the adherends and secure with the adhesive tape on the reverse side of each adherend.
- Apply the sufficient adhesive components in the specified manner to the surface of the adherends.
- Standing the adherends on their longest edges on top of a sheet of polythene placed on a flat surface, bring the adherends together with light finger pressure, to form the specified overlap.
- Without lifting from the surface or moving the assembly excessively, apply a 'foldback' clip centrally to the overlap area to secure it during the early stages of cure. A second 'fold-back' clip may be applied should it be necessary to move the test assembly to the specified curing environment. It is important that the bonded adherends are prevented from excessive relative movement during this transfer stage and the early stages of curing.
- After the bonded adherends have cured for the specified period, place the test-pieces in the environment where they are to be tested. Allow time for the temperature equilibration.

Testing

- Inspect all test assemblies carefully and discard those that are excessively misaligned or those, which do not have their bond area completely, filled.
- Set up a tensometer with wedge grip jaws and test each assembly with a jaw separation rate of 6 mm/minute. Record the maximum breaking load.

Expression of results

- Examine the faces of the separated adherends for irregularities and check overlap length. Further test assemblies will need to be constructed to replace those rejected.
- The value for shear strength should be calculated as follows:

  a) Calculate the mean of the breaking force in kilo Newtons (kN) to three significant figures.

  b) Express this mean in Mega Pascals (MPa) by dividing it by the overlap in square millimetres, i.e.:

$$\frac{kN \times 1000}{Area\ in\ mm^2} = MPa\ (expressed\ to\ 1\ decimal\ place)$$

For the standard overlap, multiply the value in kN by 3.137 to convert to MPa

c) Measure the spread of results by calculating the coefficient of variance (CoV). The CoV is simply the standard deviation divided by the mean.

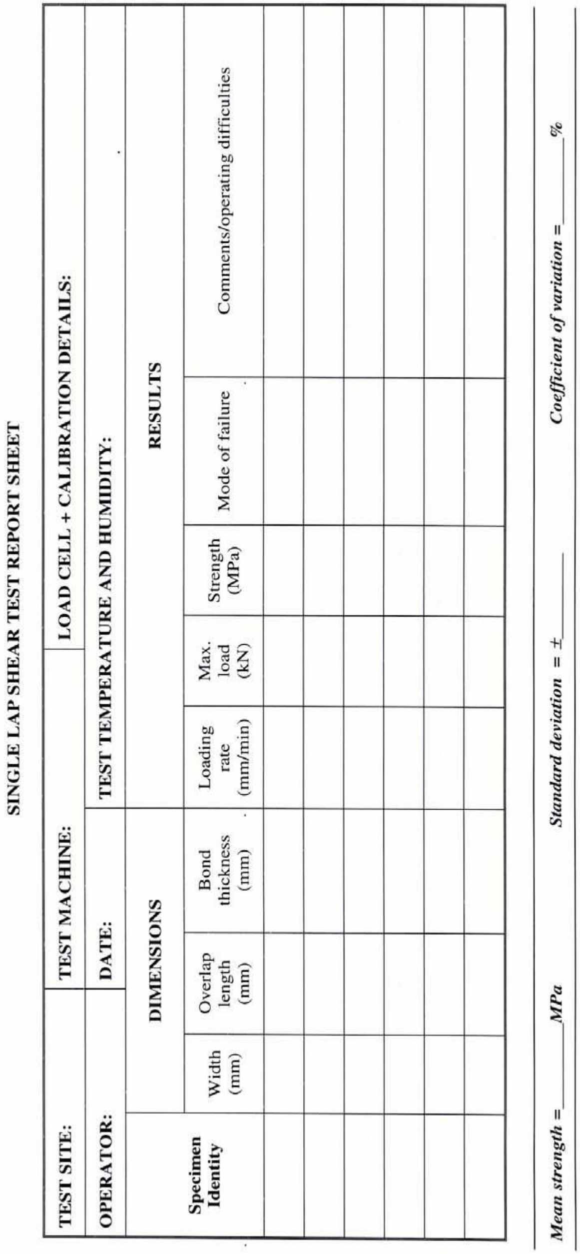
Stage 3: Specimen preparation details

In Stage 3, participants were requested to provide details on the following

Method of adherend/end tab abrasion	
Type of solvent used for degreasing	
Method of specimen alignment during cure	
Cure temperature	
Weight of ballotini balls used	

Single lap shear test report

The following data sheet was completed for each stage of the R-R evaluation exercise.



SINGLE LAP SHEAR TEST REPORT SHEET

TEST SITE:

TEST MACHINE:

LOAD CELL + CALIBRATION DETAILS:

OPERATOR:

DATE:

TEST TEMPERATURE AND HUMIDITY:

Specimen Identity	DIMENSIONS			RESULTS				
	Width (mm)	Overlap length (mm)	Bond thickness (mm)	Loading rate (mm/min)	Max. load (kN)	Strength (MPa)	Mode of failure	Comments/operating difficulties

Mean strength = ______ *MPa*

Standard deviation = ± ______

Coefficient of variation = ______ %

Test results and discussion

The results of the R-R programme on testing of adhesive joints, which were analysed according to ISO 5725-1 [51], clearly showed a need for formalised guidance on manufacturing and testing of adhesive joints. A summary of the results for Stages 1, 2 and 3 is presented in Tables A2.1 and A2.2, and Figure A2.1 with the individual results from each participant given in Tables A2.3 to A2.5 (end of Appendix 2). The R-R exercise has shown that the most crucial steps in the manufacturing process were:

- Surface treatment of the adherends.
- Adhesive preparation (i.e., mixing and adding ballotini glass balls).
- Joint assembly (including clamping).
- Cure conditions.

Note A2.1: The level of training, experience and motivation of technical staff can play a significant role in the quality of test results.

Stage	Repeatability Conditions		Reproducibility Conditions		Mean Results
	S_r	r	S_R	R	
1	7.2	20.2	7.5	21.1	6542
2	5.5	15.4	7.5	21.1	6652
3	11.5	32.2	34.2	95.6	5160

Table A2.1. Repeatability, reproducibility and mean values for stages 1 to 3

Definitions of Sr, r, S_R and R taken directly from reference [51].

Repeatability value *r*: The value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie within a probability of 95%. Repeatability refers to variance of test results within a batch of specimens tested at a single site.

Reproducibility value *R*: The value below which the absolute difference between two single test results obtained under reproducibility conditions may be expected to lie within a probability of 95%. Reproducibility refers to variance between test results from different sites where the specimens are prepared from the same material and tested to the same procedure.

Repeatability standard deviation S_r: The standard deviation of test results obtained under repeatability conditions.

Reproducibility standard deviation S_R: The standard deviation of test results obtained under repeatability conditions.

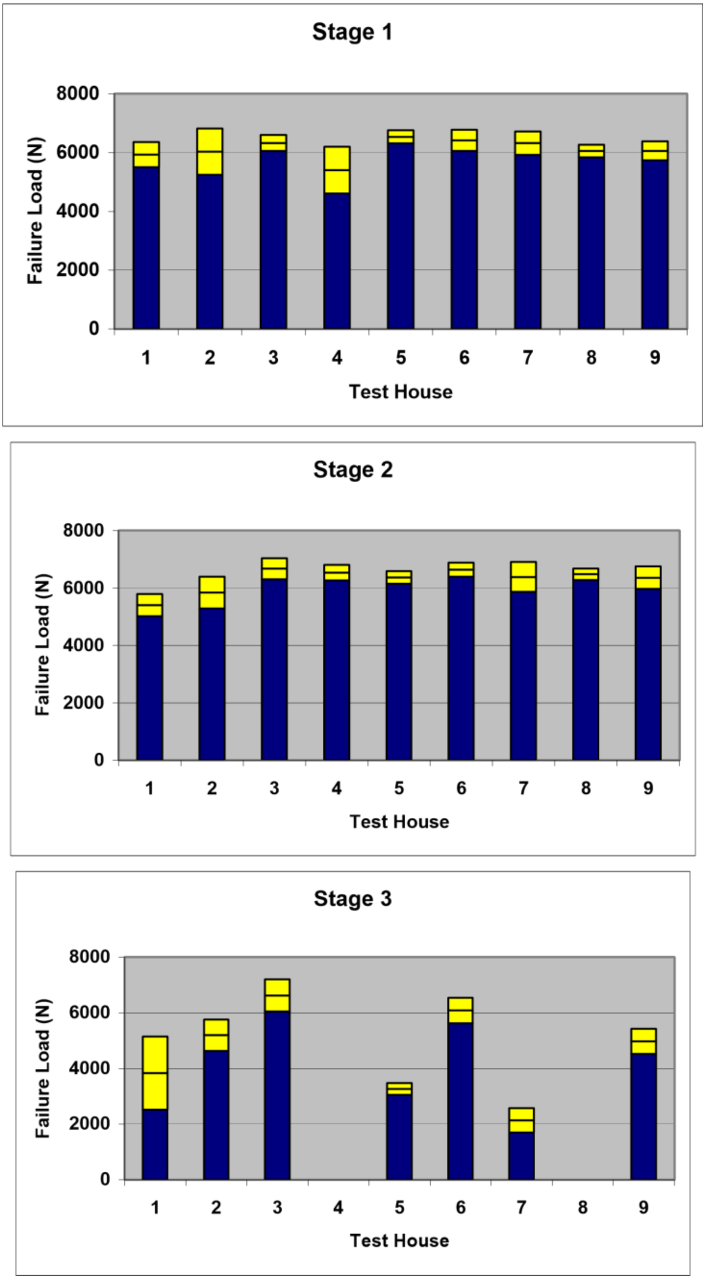


Figure A2.1 Test results (mean ± 1 standard deviation)

The results shown in Table A2.1 show good repeatability within laboratories and good reproducibility between laboratories for Stages 1 and 2. Both repeatability and reproducibility were markedly worse for Stage 3, as to be expected. Tables A2.2 provides details on manufacturing and test parameters used by the various laboratories/test houses in Stage 3.

Parameter	comments
Method of adherend/end tab abrasion	SiC (wet/dry, 60/80 and 180 emery paper) Grit-blasted (240 grit)
Type of solvent used for degreasing	Acetone (commonly used), MEK, IMS and n-heptane
Method of specimen alignment clamping during cure	Support fixture, static weight, and bulldog clips
Cure Conditions	
Cure temperature (°C)	60
Cure time (hrs)	1
Weight of ballotini balls used	1 wt. % (mixed or sprinkled)
Bond-line thickness (mm)	Ranging from 0.20 to 1.00 (typically 0.25 mm)
Test speed (mm/min)	Ranging from 1 to 6
Test Conditions	
Temperature (°C)	Ranging from 22 to 23
Relative humidity	Ranging from 45 to 53 (or not stated)
Failure mode	Cohesive/adhesive (typically 70%/30%)
Additional Observations • Test house 1 observed adhesive not fully cured (i.e., pliable/soft). Two untested specimens were post-cured for 1 hr at 60 °C. Large scatter in strength values for first batch of specimens tested prior to post-curing. • Test house 5 observed lumpy adhesive during joint preparation, which may explain the low joint strengths that were obtained. • Test house 7 observed that the adhesive was gritty (and possibly crystallised), and hence this may have contributed to the large variation in bond-line thickness between specimens (0.25 to 1 mm) and low joint strengths. • Test house 9 removed spew from several specimens after curing the joints, which may have contributed to lower strength values observed.	

Table 2.2. manufacturing and test parameters used by participants in stage 3

The R-R exercise highlighted the need for revision of standard test methods to accommodate non-standard geometries and to remove existing ambiguities. The standard method used in the exercise was open to interpretation and invariably trial specimens were required to obtain the correct machine settings.

A number of concerns raised in relation to the preparation and testing of the single-lap joint are presented below:

- Two-part adhesive needs to be carefully mixed, ensuring correct ratios of hardener to adhesive and that the mixture is uniform. When using glass ballotini balls for controlling bond-line thickness ensure the balls are unformly distributed throughout the adhesive (i.e., do not sprinkle).
- Fillet geometry is known have a major effect on the joint strength, however there is no specification for fillet geometry in BS EN 1465. The fillet should be shaped before cure and/or during cure. The spew should not be removed after cure.
- There is no guidance in BS EN 1465 (or most standards) on methods for controlling bond-line thickness or removing adhesive from the specimen edges in BS EN 1465. The lack of comparative data in the published literature also makes it difficult to select a suitable method for controlling bond-line thickness. Failure to control bond-line thickness can result in low bond strength (see Table A2.5). Temperature of adhesive layer should be monitored to ensure that cure temperature is reached. Do not assume that the oven temperature and the temperature of the adhesive in the joint are the same (see Chapter 4).
- End tabs are introduced to single-lap joints 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 (see Figure 2). BS EN 1465 does not specify the use of end tabs. The introduction of end tabs, however will add to the costs of this quality assurance test.
- Test rate is not specifically stated, but instead the standard specifies that the test joint be loaded at a constant stress or strain rate and that failure be achieved in specified period of time (65 ± 20 s). Hence, there is a need for trials to determine the rate necessary to complete tests within the specified time. At least 2-3 specimens may need to be tested in order to determine a suitable test rate. Test rates varied from 1 to 10 mm/min in Stages 1 and 2 and between 1 and 6 mm/min for Stage 3.
- Standard software format for reporting test data for different test machines could be a step forward in reducing test costs and time.
- BS EN 1465 fails to account for specimens manufactured from materials other than steel or using non-standard dimensions (adherend thickness and bond length, etc.). The standard could be more versatile by providing guidance in these cases.

Site	**Failure Load (N)**		
	Individual	**Mean**	
1	5638 6638 6683 6698 6060 6435	6359	427 (6.7)
2	5290 7179 7561 6938 7098 6839	6817	789 (11.6)
3	6514 6906 6213 6695 6401 6860	6598	271 (4.1)
4	6730 5000 5370 6700 6760 6650	6202	797 (12.9)
5	7010 6390 6800 6940 6740 6660	6757	221 (3.6)
6	7129 6821 6585 6417 7262 6430	6774	360 (5.3)
7	6810 6440 6900 7146 6957 6059	6719	398 (5.9)
8	6442 6117 6377 5933 6493 6243	6268	214 (3.4)
9	6200 6020 6340 6980 6400 6360	6383	323 (5.1)

Table A2.3. As-received stage 1 sesults

Site	**Failure Load (N)**		
	Individual	**Mean**	
1	5603 5760 5910 5145 6143 6203	5794	390 (6.7)
2	6413 6750 5820 5873 7103 -	6392	555 (8.7)
3	6450 7369 7329 7285 6783 6991	7035	366 (5.2)
4	7220 6660 6800 6810 6400 6910	6800	271 (4.0)
5	6650 6920 6440 6320 6450 6710	6582	220 (3.3)
6	6854 7162 6864 6476 6873 7117	6886	245 (3.6)
7	7589 7211 7079 7013 6255 6313	6910	525 (7.6))
8	6601 6854 6389 6541 6770 6911	6678	201 (3.0)
9	6950 7400 6390 6640 6340 6780	6750	393 (5.8)

Table A2.4. As-received stage 2 results

Site	Failure Load (N)		
	Individual	Mean	
1	3393 6203 1153* 6105 4875	5144*	427 (6.7)
2	5700 5438 6038 5888 4915 6570	5758	560 (9.7)
3	7130 6839 6321 7822 7306 7817	7206	581 (8.1)
4	-	-	-
5	3710 3110 3420 3620 3450 3520	3472	207 (6.0)
6	6193 5955 7153 6960 6385 6587	6539	456 (7.0)
7	2070 2790 2530 2800 3160 2070	2570	436 (16.7)
8	-	-	-
9	5520 5780 5320 4900 6040 4980	5423	447 (8.2)

Table A2.5. As-received stage 3 results

** Outlier value removed for analysis purposes*

Appendix 3

Surface preparation of adherends

Introduction

Appendix 3 provides a brief description of generic procedures for preparing different material surfaces [1-9, 21-29].

Steel

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

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

Zinc coated steel

Galvanised steel surfaces are less prone to rusting in most environments than untreated steels and when treated with a suitable surface preparation the adhesive-zinc interface is stronger than the steel-zinc interface. This section provides details on the method to be used for preparing galvanised steel substrates for bonding: Degrease with a suitable solvent (e.g., acetone or methyl ethyl ketone (MEK)).

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

Stainless steel

The recommended steps to be taken in preparation of stainless steel surfaces are:

- Degrease to remove oils and lubricants present on the substrate surface.
- Grit blast the surface ensuring the protective 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:

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

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

Titanium alloys

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

- Degrease surface with a suitable solvent (e.g., acetone or MEK).
- Grit blast
- Remove dust/debris by either brushing, by oil-free air blast or by vacuum cleaner.
- Acid etch the surfaces.
- Rinse and dry.
- Chemically stabilise oxide layer.

Fibre-reinforced polymer composites

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 fibre-reinforced polymer composites is as follows:

- Grit blast
- Remove dust/debris by either brushing, by oil-free air blast or by vacuum cleaner.
- Acid etch the surfaces.
- Rinse and dry.
- Chemically stabilise oxide layer.

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 [28]. 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 affects the physical and mechanical properties of the outer layer of the cured laminate. If the peel-ply contains a release agent, the adherend should be cleaned with a solvent and dried in a stream of dry nitrogen.

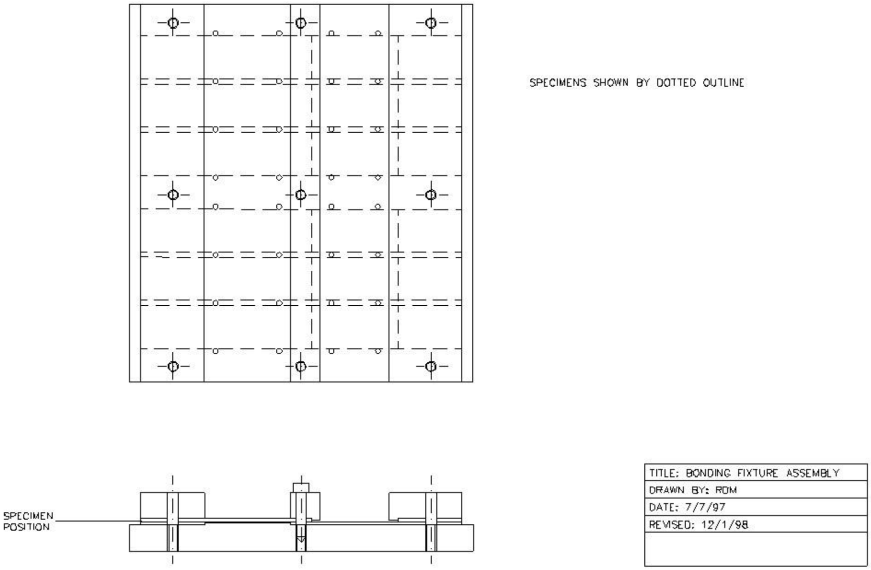
Note A3.1: All fibre-reinforced plastic composites absorb small amounts of moisture from the atmosphere when exposed to ambient levels of humidity (i.e., standard laboratory conditions). For ambient temperature curing adhesive systems, the presence of moisture in the composite adherend may pose no problems, however it is advisable, to dry the adherends prior to bonding to prevent reductions in glass transition temperature T_g and fracture toughness G_c.

Appendix 4

Bonding fixture for single-lap joints

Introduction

Appendix 4 provides technical diagrams to construct a bonding fixture for producing single-lap joint specimens (see Figure 2). This fixture was used to produce the specimens for Stages 1 and 2 of the R-R exercise reported in Appendix 2.



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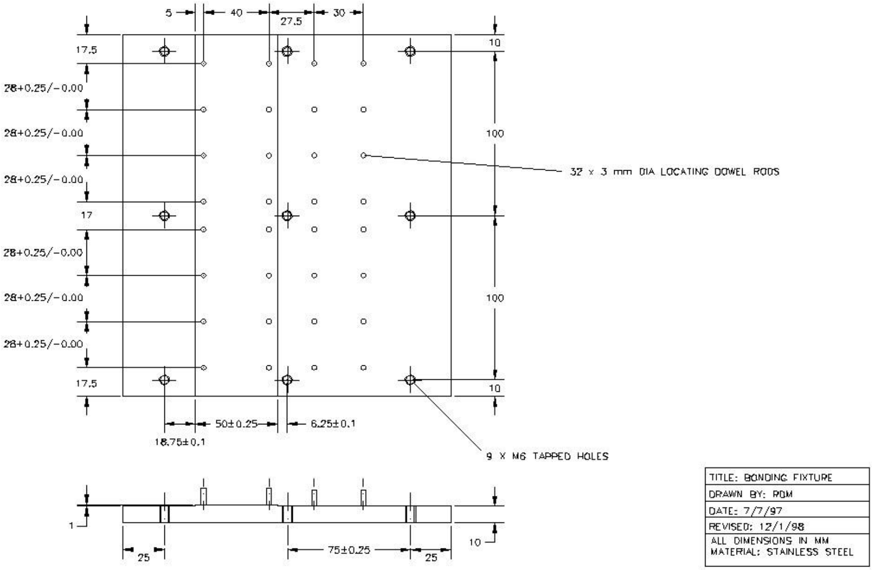
REVISED: 12/1/98



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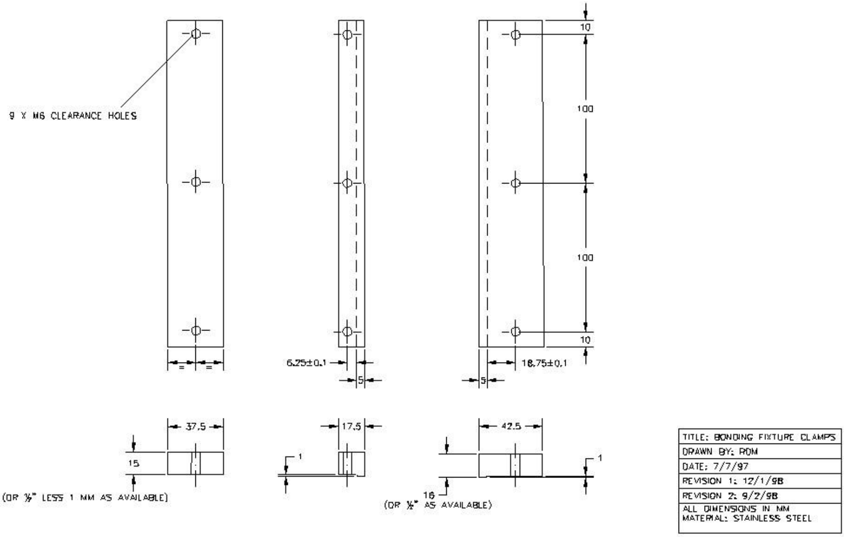
DATE: 7/7/97
REVISED: 12/1/98
ALL DIMENSIONS IN MM
MATERIAL: STAINLESS STEEL



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DATE: 7/7/97
REVISION 1: 12/1/98
REVISION 2: 9/2/98
ALL DIMENSIONS IN MM
MATERIAL: STAINLESS STEEL

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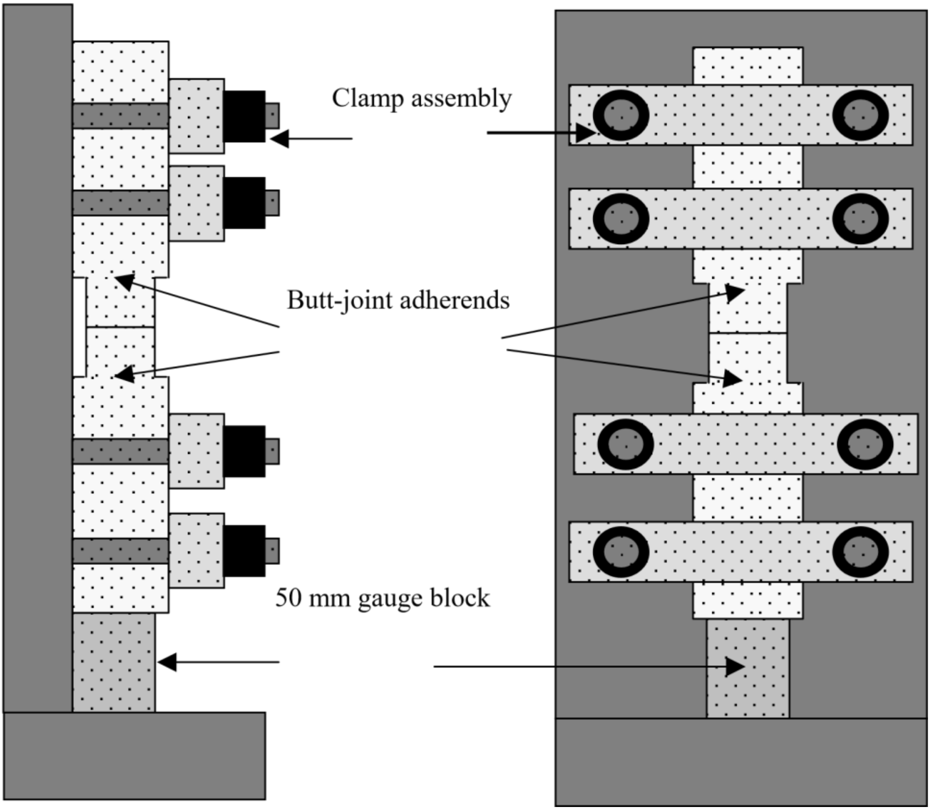
Appendix 5

Alignment fixture for bonding tensile butt joints

Alignment fixture for bonding tensile butt joints

The diagram shown below is a schematic of the alignment fixture used at NPL for bonding tensile butt joint specimens (see also Figure 3).



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