National Physical Laboratory

Good Practice Guide No. 72

Characterising strength of adhesion

EXPERT

TECHNICAL LEVEL

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Abstract

The design of adhesively bonded structures requires accurate material property data. These data, which are often best obtained from bulk test specimens, can provide an indication of a material's cohesive strength, however, designers also need to consider the interfacial strength between the adhesive and substrates. Information on interface strengths is normally obtained from adhesives joint tests. The complexity of stress distributions in adhesively bonded joints, however, invariably leads to difficulties in obtaining quantitative interface strengths that are applicable to other loading configurations. As part of a BEIS (formerly DTI) funded Measurements for Materials Systems project, "Interfacial Adhesion Strength", several test methods for quantifying adhesion strength have been evaluated to assess their ability to quantify the strength of adhesion between adhesive and adherend. An experimental and Finite Element (FE) study was conducted on each of the selected methods.

As the properties of the surfaces being bonded are critical for the manufacture of strong, durable adhesive bonds, techniques for inspecting surfaces have also been assessed. There is no universal surface or adhesion test suitable for all situations – methods need to be selected based on the materials (adherend and adhesive), expected structural loading and service conditions, degree of accuracy required and budget (time and cost) for the assessment. This document provides guidance in the selection and use of surface characterisation and adhesion strength test methods.

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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.

Breaking stress: Stress at the moment of rupture of a specimen.

Brittle failure: Failure in which the fracture surface exhibits no permanent material deformation to the naked eye (e.g., stretching, elongation and necking down).

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).

Composite: Solid product consisting of two or more distinct phases, including a binding material (matrix) and a particulate or fibrous material.

Contamination-free: Absence of foreign matter, both on a treated surface (i.e., cleanliness), or which could migrate through the bulk to a bonded interface with time.

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.

Degradation: Change in the chemical structure of a polymeric material involving a deleterious change in properties.

Delamination: Separation of layers (i.e., planar defect) in a laminate.

Deterioration: Permanent change in the physical properties of a material evident by impairment of these properties.

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.

Hyperelastic: Large strain elastic behaviour modelled using a strain energy potential.

Interface: The region where two materials are in intimate contact. Interfaces can be solid-solid (cured adhesive joint), solid-liquid (uncured adhesive joint), solid-gas (exposed adherend), liquid-gas (open adhesive) or liquid-liquid (insoluble liquids).

Interphase: The region near an interface where the properties of materials are altered from their bulk properties due to the presence of the interface.

ISO: International Standards Organisation.

Laminate: Product made by bonding together two or more layers (plies) of material or materials.

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

Materials model: Constitutive equations linking stress and strain properties.

Modulus: Material property denoting the stress required to extend a specimen by unit strain. (modulus = σ/ε).

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.

Permeability: Property of a material transmitting gases and liquids by passage through one surface and out at another surface by diffusion and sorption processes.

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

Poisson's ratio: The ratio between axial and transverse strains in a uniaxial tension test as defined in Equation (3), used in the definitions of elastic moduli.

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.

Prepreg: Admixture of resins (with or without fillers), additives, and reinforcements in woven and filamentous form, ready for moulding.

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

Reinforced plastic: Polymer (plastic) with high-strength fibres embedded in the composition, resulting in some strength properties greatly superior to those of the base resin.

Roughness: Micro-roughness represents the fine structure of a surface with dimensions 0.1 µm or less. Macro-roughness, which may also have a significant influence on bond performance, suggests the coarser structure of a surface with dimensions greater than 0.1 µm.

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.

Sealant: An interlaminar layer of polymeric material applied for the purpose of filling gaps and insulating the interior of the joint from external environments. Sealants are not used to provide load bearing capacity although adhesives can also act as sealants.

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.

Soundness: Freedom from weak and loosely attached surface layers.

Stability: The stability of surface layers and oxides, towards water, organic compounds, and elevated temperatures, as a function of time following treatment.

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 (S-N) 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.

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

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

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

Uniformity: Visible or measurable consistency of the other characteristics, and of the regularity of a treated surface area.

Visco-elastic: A material whose properties combine elastic (or recoverable) and viscous (or irrecoverable) components. Visco-elasticity leads to strain rate and temperature dependent properties and is also responsible for damping.

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.

Wettability: A measure of the attractiveness of a solid surface towards a liquid, encompassing aspects such as energy and chemistry.

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 Good Practice Guide aims to provide guidance to technologists, laboratory staff and quality assurance personnel on how to characterise and quantify the adhesion of adhesives to substrates in order to design bonded structures. A general familiarity with laboratory operations and mechanical testing, but not specifically adhesives testing, is assumed. The objective of this Guide is to familiarise the operator with the options available for testing and the factors that can influence the test and design results. It is highly recommended that validation tests be performed to gain confidence in design calculations.

Finite Element Analysis (FEA) is used extensively in the evaluation and design of structures and sub-components. FEA also provides a useful tool for evaluating test methods and interpreting the results. Reliable FEA predictions require accurate materials properties data, which are often best obtained from bulk test specimens. Such data can give an indication of the cohesive strength of the materials, but designers also need to consider the strengths of the interfaces between the adhesive and substrates. Methods for obtaining such data are outlined in this guide. There is no single test that is suitable for every material and loading condition. Often there is a trade-off between the relative accuracy and cost of testing. This becomes increasingly important where a wide range of adhesives or surface treatments may need to be screened, particularly for long-term durability. The following types of test method are described:

- Overlap Test Methods (Tensile-Shear)
- Peel Test Methods
- Fracture Test Methods (Cleavage – Thick Sections)
- Tensile Test Methods
- Flexural Test Methods

Greater detail is given to the test methods investigated in the Measurements for Materials Systems project 'Interfacial Adhesion Strength' – pull-off test, butt tension and three-point bend test – but much of the guidance in this document is generally applicable to all joint tests.

Surface preparation is recognised as a critical step in the adhesive bonding process and considerable effort is frequently expended in optimising surface treatments for bond strength and durability. Correct surface preparation is essential ensuring the long-term structural integrity of bonded structures under hostile service conditions. Unsatisfactory surface preparation will result in premature and unpredictable interfacial failure of bonded joints. An essential part of ensuring good adhesion and long-term durability is the ability to control and monitor the surface morphology and chemistry of the substrate materials during surface preparation and storage. Methods of assessing the surface of adherends, to provide a route to quality assurance of bonds, are described.

Emphasis is given to techniques for testing interfacial adhesion strength and assessing surface properties, which can be used simply, quickly, and cheaply, suitable for quality assurance testing or large-scale screening of materials or processes.

The information presented in this guide has been gathered under BEIS (formerly DTI) funded programmes of research on the Performance of Adhesive Joints (PAJ) and Measurements for Materials Systems (MMS).

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Chapter 1

Scope

Scope

The performance of a materials system, such as an adhesive joint, coating, or composite, is influenced by the properties of the bulk materials and of the interfaces within the system. The design of adhesively bonded structures requires accurate materials data and knowledge of the performance of the interfaces. Materials property data are often best obtained from bulk test specimens. Such data can give an indication of the cohesive strength of the adhesive, but designers will also need to consider de-bonding at an interface as a potential mode of failure. However, quantitative properties of the interface are seldom considered in the design process owing to the lack of validated data and the absence of design models and tools. Information on interface strengths can be obtained from adhesive joint test. The plethora of available methods, mixture of failure modes, difficulty in detecting the start of failure and the complexity of evaluating stress distributions in joints, however, creates difficulties in obtaining quantitative interface strengths that are applicable to all loading configurations.

Surface preparation is recognised as a critical step in the adhesive bonding process and considerable effort is frequently expended in optimising surface treatments for bond strength and durability. Correct surface preparation is essential ensuring the long-term structural integrity of bonded structures under hostile service conditions. Unsatisfactory surface preparation will result in premature and unpredictable interfacial failure of bonded joints. An essential part of ensuring good adhesion and long-term durability is the ability to control and monitor the surface morphology and chemistry of the substrate materials during surface preparation and storage. Methods of assessing the surface of adherends provide a route to quality assurance of bonds.

This Good Practice Guide seeks to describe methods for testing interfacial adhesion strength and assessing surface properties. Emphasis is given to techniques, which can be used simply, quickly, and cheaply, suitable for quality assurance testing or large-scale screening of materials or processes. The information presented in this guide has been gathered under BEIS funded programmes of research on the Performance of Adhesive Joints (PAJ) [1-5] and Measurements for Materials Systems. The outputs from this work are available through the Adhesives Design Toolkit website (www.adhesivestoolkit.com), a free information resource for adhesives.

Chapter 2 describes methods that can be used for assessing and quantifying surface conditions. There are many surface preparation methods available to treat the wide array of materials that may be bonded; it is not the purpose of this Guide to recommend any specific treatment.

Chapter 3 describes many of the methods that could be considered for determining the strength of adhesion between the adhesive and adherend. Adhesion mechanisms are discussed in Appendix 1. Good quality test specimens are critical for obtaining accurate and reproducible test data. Preparation of adhesive joints must be undertaken using carefully controlled procedures, since manufacturing variability contributes significantly to the degree of repeatability in the test data. General guidance on adhesive joint manufacture is given in Appendix 2 and Good Practice Guide No. 47 [3].

Interpretation of adhesive joint data, even if the results are accurate and reproducible, may not be straightforward. Chapter 4 outlines some important points to be considered when interpreting results. Methods of including adhesion strength in predictive finite element design calculations are discussed in Appendix 3.

Further information and guidance on the testing of adhesives and adhesive joints may be obtained from other NPL Good Practice Guides [1-5] and various textbooks [6-13].

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Chapter 2

Surface preparation and assessment

- Surface preparation
- Surface assessment methods
- Surface analytical techniques
- Imaging techniques
- Quantifying appearance
- Surface energy
- Mechanical properties of surfaces
- Electrical and optical properties of surfaces
- Summary of surface inspection methods

Surface preparation

Good surface preparation is crucial to ensuring optimum bond strength and reliable long-term performance of bonded joints, particularly under hostile service conditions [6-13]. Unsatisfactory surface preparation will result in premature and unpredictable bond failure at the adhesive/adherend interface. Surface preparation is recognised as the most critical step in the adhesive bonding process and considerable effort is often expended in optimising the surface treatment.

The purpose of surface treatments is to [3]:

- Remove contaminants that may interfere with bond formation;
- Remove weak surface layers;
- Produce a surface morphology that enhances the surface area available for bonding and/or allows mechanical keying;
- Chemically modify the surface to increase surface energy and chemical compatibility with the adhesive.

The selection of surface treatment is largely dependent on the substrate, the required strength and durability of the joint and economic considerations (such as costs and time involved in preparation). Surface treatment processes often consist of a series of different steps. Surface treatments can be classified as either passive or active. 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.

It is important that the process of surface preparation only affects the chemistry and morphology of a thin surface layer of the adherend(s) and does not alter the mechanical and physical properties of the underlying substrate. There are many procedures available – further information can be found in “Guide to the Structural Use of Adhesives” produced by The Institution of Structural Engineers [13] and specific treatments are defined in various standards, e.g., BS 7079 [14], BS 5350-A1 [15], ASTM D2093 [16], ASTM D2651 [17], and BS EN 13887 [18] – see also [3, 19-22]. Advice is usually 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 (Control of Substances Hazardous to Health) specifications.

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 (for examples 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 [3]

Surface assessment methods

Surface characterisation of the adherend prior to bonding is an essential part of the evaluation of surface treatments to ensure optimum bond strength and environmental resistance for specific service conditions. These techniques can provide important information on:

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

Surface and chemical characterisation techniques can provide qualitative and quantitative information relating to the strength and durability of bonded joints [6, 8, 11, 23-27]. This section examines physical and chemical techniques that can be used to characterise surfaces with particular attention given to those techniques that can be used for on-line inspection (i.e., quality control) and are accessible by small industrial concerns [25]. The practicality and limitations of these techniques are discussed, and relevant data is provided to demonstrate the effectiveness of the technique for different substrate/surface treatment systems.

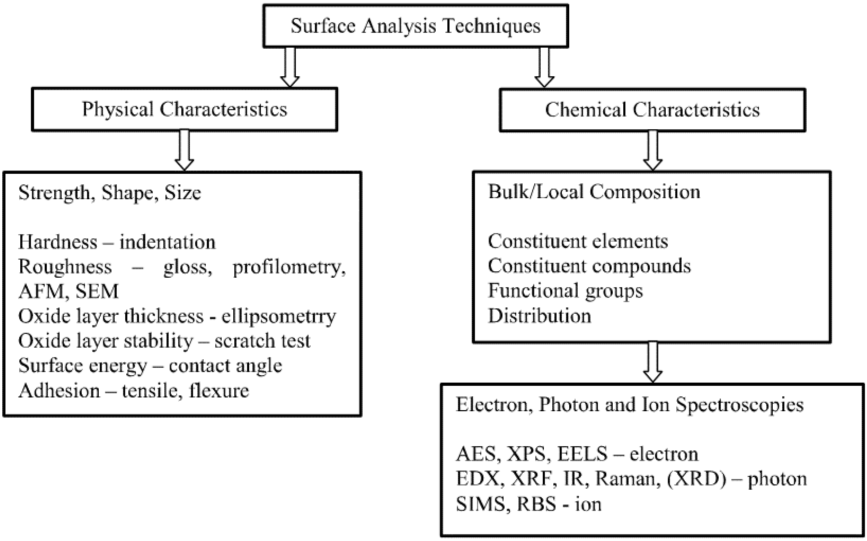

Figure 1. Surface characterisation techniques

There are many physical measurement and chemical analysis techniques that can be used for surface characterisation of metals, plastics (including fibre-reinforced plastic composites) and other substrates -.see Figure 1. The section describes techniques that can be used to evaluate surface treatments applied to ensure optimum bond strength and environmental resistance for specific service conditions. The techniques available to the researcher range from the inexpensive and simple (e.g., visual inspection) to the expensive and specialised (e.g., photoelectron spectroscopy).

Spectroscopic techniques require significant investment in both facilities and trained operators. Hence, they are generally only available at large manufacturing and research facilities. Although many research technology organisations (RTOs) and academic institutions offer spectroscopic measurement services many adhesive bonders are discouraged from regular use by the costs and need to schedule/transport samples.

Surface analytical techniques

Chemical characterisation, either elemental or functional group analysis, can be achieved using spectroscopic techniques. The various techniques can provide qualitative and quantitative information on chemical composition and varying information on molecular structure and physical-chemical characteristics, which determine the attractive forces towards adhesive molecules and, hence, bonding performance. The techniques have different levels of capabilities for scanning areas or providing depth profile information. Except for the optical techniques (IR and Raman), the measurements are performed with samples in an evacuated chamber, which adds to the cost and complexity of the technique. Many techniques require ultra high vacuum (UHV) conditions to avoid scattering or generation of secondary x-rays or electrons from gas molecules. Under UHV, there may be loss of volatile material from the surface and alterations to the microstructure of the surface being investigated.

Infrared spectroscopy (IRS)

Description	Use/Limitations
Absorption of IR photons (transmitted or reflected) occurs at specific frequencies associated with internal vibrations of groups of atoms in molecules.	IR provides: • Qualitative and quantitative chemical analysis data, particularly useful polymer chemistry. • A fingerprint of the adhesive or coating composition in any physical state that can be compared with databases of spectra to enable characterisation of molecular state. Since it is an optical (photon in/photon out) technique it is not necessary for such studies to be carried out in vacuum. IR is a commonly available laboratory technique.

Raman spectroscopy

Description	Use/Limitations
Raman is based on the inelastic scattering of monochromatic light. A laser excites the material, usually in the visible region of the spectrum. The frequency of scattered light is analysed and compared to incident values.	The technique is similar to IR in determining the nature of molecular structures and is a complementary technique to IR when characteristic frequencies are weak or for highly absorbing materials. Samples require minimal preparation, but need to be stable to high intensity light and contain no species that fluoresce when excited by visible radiation.

Energy dispersive x-ray analysis (EDX)

Description	Use/Limitations
X-rays emitted from a surface material upon exposure to a primary beam of electrons are characteristic of the atom from which they originated.	Detection and analysis of the characteristic Xray lines of various elements can be obtained using an EDX system attached to an SEM. EDX generates elemental distribution maps, enabling both qualitative (boron to uranium) and some quantitative (sodium to uranium) elemental analysis. Samples must be studied under vacuum.

X-ray photoelectron spectroscopy (XPS)

Description	Use/Limitations
Measures the energies of photoelectrons emitted from atoms of a sample when irradiated with soft (or low energy) X-rays.	XPS is surface-sensitive and used for quantitative elemental analysis, capable of detecting all elements with the exceptions of hydrogen and helium. It can discriminate between different oxidation states and different chemical environments, and be used in conjunction with inert gas ion sputtering to determine the variation in chemical composition with depth. Some polymeric material samples are sensitive to ion beam damage. Samples must be studied under vacuum.

Auger electron spectroscopy (AES)

Description	Use/Limitations
High-energy electron beam bombardment of the surface results in the emission of Auger electrons at characteristic discrete energies.	AES is a surface sensitive, non-destructive technique for identifying the elements, with the exception of hydrogen and helium, in the first few atomic layers and is able to provide quantitative data (by comparison with known standard samples). Combined with inert gas ion sputtering, AES can obtain depth composition profiles and scan the specimen surfaces with high spatial resolution (0.5µm). AES is not particularly suited to insulating materials. Samples must be studied under vacuum.

X-ray fluorescence spectroscopy (XRF)

Description	Use/Limitations
Irradiation with X-rays results in the release of photons with element specific characteristic energies.	XRF provides qualitative and semi-quantitative elemental analysis for elements above aluminium in the periodic table. It requires very flat samples. Samples must be studied under vacuum.

Secondary ion mass spectrometry (SIMS)

Description	Use/Limitations
The bombardment of a surface with a beam of high-energy ions results in the ejection of molecular fragments, atoms and ions from the surface that are subsequently analysed.	SIMS provides surface elemental analysis and depth concentration profiles on areas from several mm to sub micron. It can detect all elements and isotopes including hydrogen and hydrogenated compounds with very high sensitivity (parts per billion). Quantitative analysis is complex and requires reference standards. Samples must be studied under vacuum.

Rutherford backscattering spectroscopy (RBS)

Description	Use/Limitations
A beam of positive ions is directed at the target surface and produces ions, which are scattered by the sample nuclei, to be measured and analysed.	RBS is used for the quantitative, non-destructive compositional depth profiling and thickness measurements on thin films. The depth resolution is 10 to 20 nm and RBS can probe several thousand atomic layers and is ideal for surface analysis up to 2 µm depth. The erosion and the radiation degradation of the sample material by the particle impact are negligible. Samples must be studied under vacuum.

Imaging techniques

Often the condition of a surface is checked by visual inspection to detect signs of any problems, such as damage (e.g., scratches) and contamination (e.g., grease, marks) that might compromise bonding. The surface appearance and morphology will be assessed to determine whether surface treatments have been applied uniformly (e.g., the roughening caused by grit blasting a smooth surface). Many surface treatments affect the surface morphology at scales too fine to be resolved by the human eye and there are techniques available (summarised below) to view surfaces at higher resolution. These techniques produce images, which give qualitative information of the surface. Often these images are compared with reference images to aid interpretation. Techniques that can provide quantitative information on surface appearance are described in the next section.

Optical microscopy

Description	Use/Limitations
Observation of the surface through magnifying lenses.	Optical microscopes are commonly available laboratory equipment. Resolution is limited by the wavelength of light to around 1 µm, which is not sufficient to view many surface features. Illumination conditions can be altered (e.g., to generate fluorescence) and light signals filtered (e.g., polarising filters) to enhance images.

Scanning electron microscopy

Description	Use/Limitations
A highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated and formed into an image, the intensity of which is governed by surface topography.	Qualitative three-dimensional (3-D) imaging of surface features, Resolution of SEM is approximately 100 times greater than for optical microscopy and features can be seen to approximately 5 nm. SEM is suitable for all materials, but non-conducting materials must be given a thin conductive coating (e.g., gold sputtered), which can mask the true surface morphology. To avoid degradation of the signal by scattering of the secondary electrons SEM measurements are normally performed under vacuum.

Transmission electron microscopy (TEM)

Description	Use/Limitations
TEM focuses a beam of electrons on the specimen. The transmitted signal is projected onto a phosphor screen and an image is created.	Extremely high resolution (0.2 nm) is possible. However, electrons cannot penetrate a specimen very deeply so sample thickness is a major problem. UHV is needed.

Atomic force microscopy and scanning tunnelling microscopy

Description	Use/Limitations
Atomic Force Microscopy (AFM)/Scanning Tunnelling Microscopy (STM) measures the morphology and topography of surfaces on the atomic scale. A sharp tip, mounted on a cantilever, is scanned across the surface (in contact or non-contact modes) using interaction forces. The vertical movements of the cantilever are analysed.	Atomic force microscopy is a popular method used by many industrial R & D sectors to measure surface topography and materials properties with spatial resolutions of 5 - 20 nm and height dimensions in the range from 100 nm down to 5 nm and exceptionally to 0.2 nm. STM can produce a 3-D image for visual inspection of conductive surfaces and also measure relative surface stiffness giving local hardness and modulus information. However, investigated areas are small and scans take significant time to perform. No special atmosphere is required for AFM but the technique is sensitive to vibrations.

Quantifying appearance

Gloss measurement

Reflectance measurements can be made using a gonio-photometer. A beam of light (often a laser) is projected onto the target surface at a specific angle and the amount of light reflected is measured. For a perfectly smooth, mirror surface all the light is at the same angle, the specular angle. Surface roughness causes incident light to be scattered at angles other than that of specular reflection, such that the scatter increases with roughness. Analysis of the angular distribution and intensity of the reflected and scattered light allows parameters related to the surface roughness to be defined. These parameters include the total integrated scatter (ratio of total scattered power to total reflected power, which is related to RMS roughness R_q) and the bi-directional reflectance distribution function (scatter density normalised to incident power which contains valuable information on the distribution and size of surface features). In order to achieve this an array of detectors or a detector moving in an arc relative to the surface are used. Such an arrangement is cumbersome and lacks portability.

Simpler hand-held instruments, gloss meters, are available to quantify the degree of specular reflectance from a surface, (see Figure 2). These instruments follow standards specifying the angle of incident beam and the spot size (ASTM D523 and ISO 2813 [28-29]). A standard black glass is used as a reference for comparisons. Gloss meters have been successfully used with flat and curved surfaces for determining the surface finish of polymer products and painted surfaces for quality control purposes [30]. These instruments provide quantitative data on the uniformity and quality of surface treatments. The technique can be used to differentiate between surface treatments and the quality of the surface treatment. Commercial handheld instruments measure reflectivity at fixed angles to the substrate (20°, 60° or 85°). The technique can be automated for on-line inspection. Whilst gloss measurement shows sensitivity to gross changes in the surfaces, for example easily distinguishing grit blasted regions from untreated, it lacks the sensitivity to discriminate between different levels of surface treatment [25].

Colour measurement by reflectance spectroscopy

Colorimetry can be used to detect differences between surface treatments and the degree or intensity of the surface treatment (e.g., exposure time). Colour is sensitive to surface morphology and chemical composition. When light falls on a surface with an oxide film, several phenomena occur at the interface. Some of the light is reflected back at the film-air interface and does not enter the oxide layer, whilst the transmitted component enters the oxide layer. A portion of the transmitted light is reflected from the metal-oxide interface. Interference of the two reflections will occur, affecting the perceived colour of the surface. The equipment is basically a spectrophotometer that measures the intensity of wavelengths in the reflectance spectra (see Figure 2).

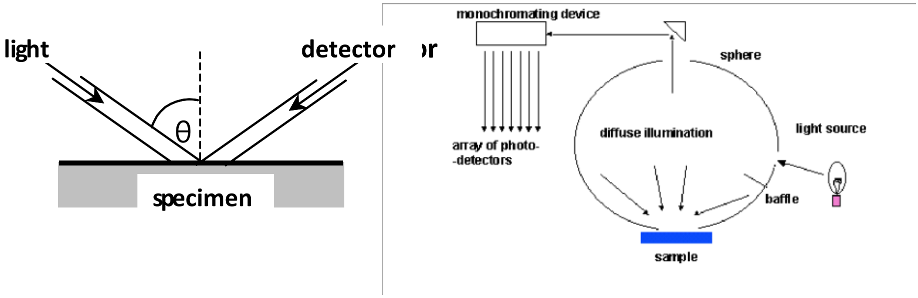

Figure 2. Schematics of reflectance measurement instruments: gloss meter (left) and colorimeter (right)

Colour measurement [31] requires a spectrophotometer to illuminate the sample with white light and to calculate the amount of light that is reflected by the sample at each wavelength interval by passing the reflected light though a monochromatic device that splits the light up into separate wavelength intervals. The instrument is calibrated using a white tile whose reflectance at each wavelength is known compared to a perfect diffuse reflecting surface. The reflectance of a sample is expressed between 0 and 100 (as a percentage). Reflectance values obtained are relative values and, for non-fluorescent samples, are independent of the quality and quantity of the light used to illuminate the sample. The spectrophotometer exposes an area on the surface to a light source with a daylight colour temperature and compares the percentage reflectance within the visible spectrum (360 – 750 nm wavelength, in 1 nm steps) to that of reference white and black colour tiles.

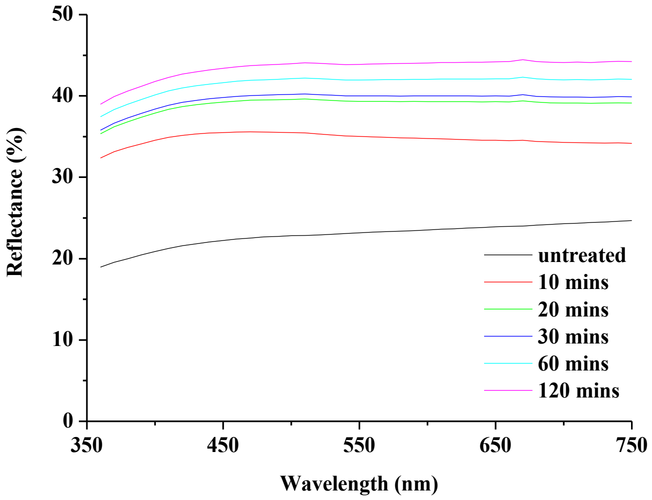

Figure 3. Reflectance spectra for untreated and CAE aluminium

Colorimetry results have shown that surface reflectance measurements have some promise and can detect differences between some surface treatments [25]. Figure 3 shows colour reflectance spectra measurements made for an aluminium sample subjected to different levels of chromic acid etch (CAE) treatment [32]. The reflectance increases with increasing treatment time, corresponding to increasing oxide film thickness. As the oxide layer grows, the reflection becomes brighter. There is a large change between the untreated surface and the treated surface but the differences between different levels of treatment are less significant.

A series of colour measurements, shown in Figure 4, were made [31] on a set of six samples of aluminium with different surface treatments (described in Table 3). Each point on the curve is an average of measurements on five different samples. For clarity, error bars are only shown for Treatment 1 (untreated), the degree of variability is typical of those found for the other curves. The results indicate that the colour measurements can distinguish significant differences between the untreated surface and the treated surface. However, given the degree of uncertainty, it is probably not possible to state with confidence that there are any significant differences between the other surface treatments apart from treatment 6 where the spectral response is significantly flatter than for the other treatments.

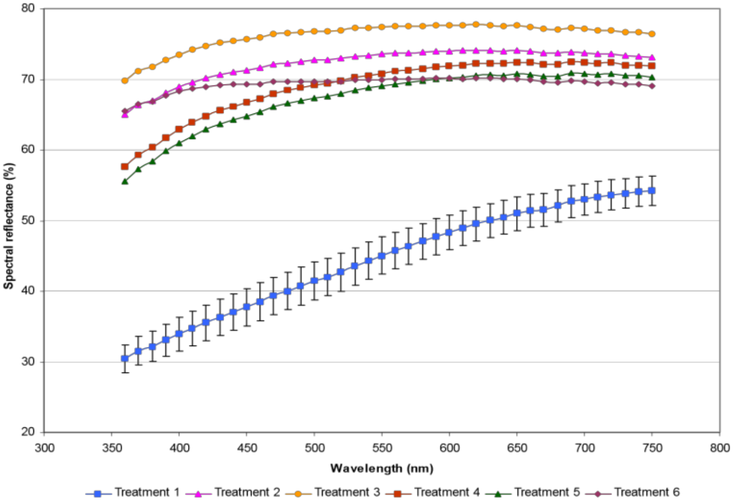

Figure 4. Colorimetry measurements for different aluminium surface treatments

These samples were exposed to hot/wet conditioning (70 °C/85% relative humidity) to degrade the surfaces. Figure 5 shows the effect of 0, 18 and 36 days conditioning on the colour spectra of samples with Treatments 1 (untreated, mill finish) and 2 (light clean). There is no significant change on conditioning for Treatment 1. For Treatment 2 there is a change between fresh and conditioned samples but no significant differences between the two conditioning durations (this was also the case for Treatments 3-6).

Treatment	Finish	Code	Description
1	Mill finish	MF	Untreated aluminium
2	Light clean	PC	Lightly degreased to remove oils - most of the surface structure and oxides still remaining.
3	Full clean	FC	An extensive electrolytic acid etch - equivalent of the optimized system without an anodised layer.
4	Full clean/anodised	FC + 25 nm	Full clean + 25 nm barrier film ("optimised" system with a full etch and a 25 nm barrier anodised film).
5	Light clean/anodised	PC + 25 nm	Light clean + 25 nm barrier film (a variant with a lower level of etch).
6	Over anodised	FC + 25 nm + 75 nm	Full clean + over-anodised - braid (a variant with extended anodisation period to produce a 25 nm barrier + 75 nm porous anodised film).

Table 3. Aluminium surface finishes

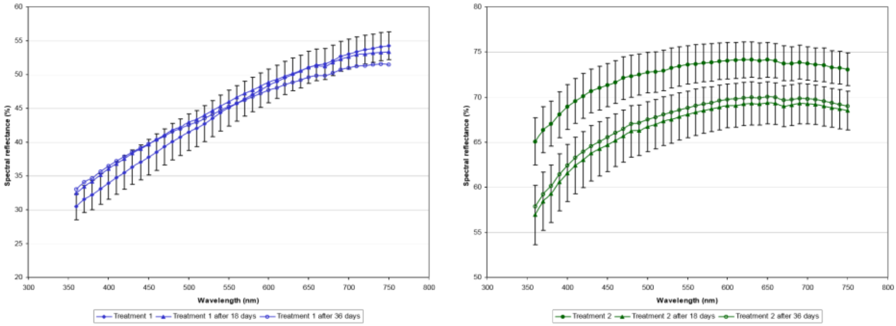

Figure 5. Effect of conditioning on colour of aluminium surfaces
mill finish (left) and light clean (right)

Colour reflectance measurements were also investigated as a method for establishing the thickness of an oil coating on a steel surface. The results shown in Figure 6 indicate that reflectance shows a good discrimination between clean and oiled surfaces. These are no significant differences evident between the different levels of oil. This situation persists during conditioning. The spectral response of the clean steel surface shows changes with conditioning but there are few significant changes for the oiled surfaces. This could be a consequence of the protective nature of the oil film.

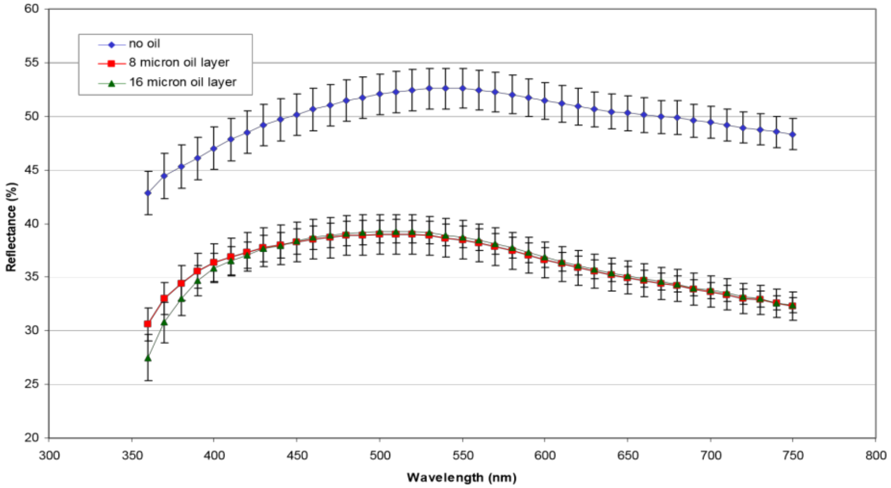

Figure 6. Effect of oil film thickness on spectral response

Surface roughness

Surface treatments are often undertaken to roughen the substrate to increase the surface area available for bonding and to provide sites for mechanical interlocking. Techniques suitable for evaluating surface topography (roughness) include:

- Contact (stylus) profilometry: hand held roughness meters typically have a measurement range of 100 nm to 4,000 nm.
- Optical profilometry; phase shift white light interferometers have typical measurement ranges of 0.2 nm to 100 µm
- Atomic force microscopy (AFM); typical measurement range 10 nm to 7000 nm.

The reliability of any measurement technique will decrease as the lower limit of resolution is approached (i.e., where the surface is smooth according to the depth scale of the instrument).

Surface topography [33] is defined as that which distinguishes a real surface from a perfectly flat, featureless one (i.e., polished). Filters are used to separate the long-wave (waviness) components of the measurement from the short-wave (roughness) components of the surface profile. Waviness is defined as the more widely spaced component of the surface texture. Roughness features are <10 µm in size and are superimposed on the waviness. Data at the beginning and end of each profile is usually discarded to remove any potential edge errors. Statistical parameters are then obtained from these profiles, which help to characterise the surface. The most common parameters used are R_a and R_q, the average roughness deviation and root mean square (RMS) roughness deviation, respectively.

Roughness parameters are calculated relative to a reference line established as the position at which half the trace lies above and half below.

$$R_a = {}^{1}/_{L} \int_0^L |y^2(x)|\, dx \tag{1}$$

$$R_q = \sqrt{{}^{1}/_{L} \int_0^L |y^2(x)|\, dx} \tag{2}$$

where *L* is the sampled length, *x* is the position along the sampled length and *y*(*x*) are the roughness profile values (see Figure 7).

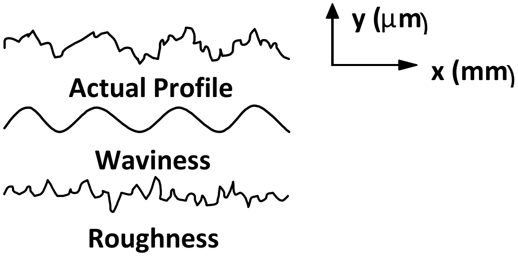

Figure 7. Surface topography definitions

Another useful measure that is occasionally used is peak count, given in peaks per unit length where a peak is any maximum to minimum which extends through a given region around the reference line.

Contact (stylus) profilometer instruments move a loaded probe (usually a diamond) across the target surface and record the vertical movement caused by the irregularities [33-37]. These measurements are analysed automatically using Equations 3 and 4 to produce a roughness value. A typical instrument is shown in Figure 8. This technique is most suitable for hard surfaces, since soft materials can be damaged, and reducing the load may result in some surface features not being registered. Lateral and vertical resolution is affected by stylus shape (usually a cone with a spherical tip (Figure 8) and angle of 60° or 90°), stylus tip radius (typically 5 µm - sharper tips increase damage) and surface profile. Instruments to measure surface roughness are limited by spatial frequency, some features being too wide and others too narrow to be detected, as well as the limits on minimum height of surface features that is detectible. Peaks narrower than the stylus are recorded as broader and the base of narrow troughs may not be reached. This technique can also have difficulty measuring highly curved or convoluted surfaces with steep slopes.

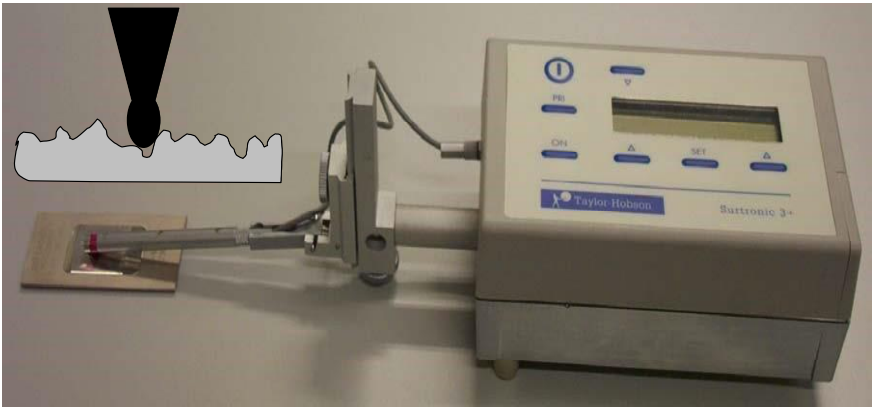

Figure 8. Stylus profilometer

Recent developments have seen the emergence of non-contact (or optical) profilometers, such as laser triangulation systems and optical interferometers for measuring 3-D surface topography. These instruments are being used to measure roughness, finish and texture of surfaces ranging from polished optics to rough surfaces, such as rolled steel and aluminium, plastics, and ceramics. The cost of the instrument increases with resolution and high-resolution equipment is relatively expensive Currently there are no supporting standards for optical systems, although there is a plethora of instruments on the market. It has been shown that stylus measurements provide comparable roughness values to optical profilometry techniques (e.g., 3-D interferometric optical phase shift white light interferometer).

Figure 9 shows an example of the use of roughness measurements. Roughness measurements were made to study differences between six samples of aluminium with different surface treatments (Table 3). The measurements on fresh, unconditioned samples indicate that this simple test indicates significant differences between the untreated sample (Treatment 1) and the other treatments. There are less significant but likely genuine differences between the anodised surfaces (Treatments 4-6) and the cleaned, unanodised surfaces (Treatments 2 and 3). Treatment 4 is nominally the optimum treatment for bonding and has the highest roughness. The effect of hot/wet conditioning (70 °C, 85% RH) is to reduce the roughness of the treated samples towards that of the untreated material (which is not significantly changed by the treatment). After 36 days the roughness of the surfaces is indistinguishable [32].

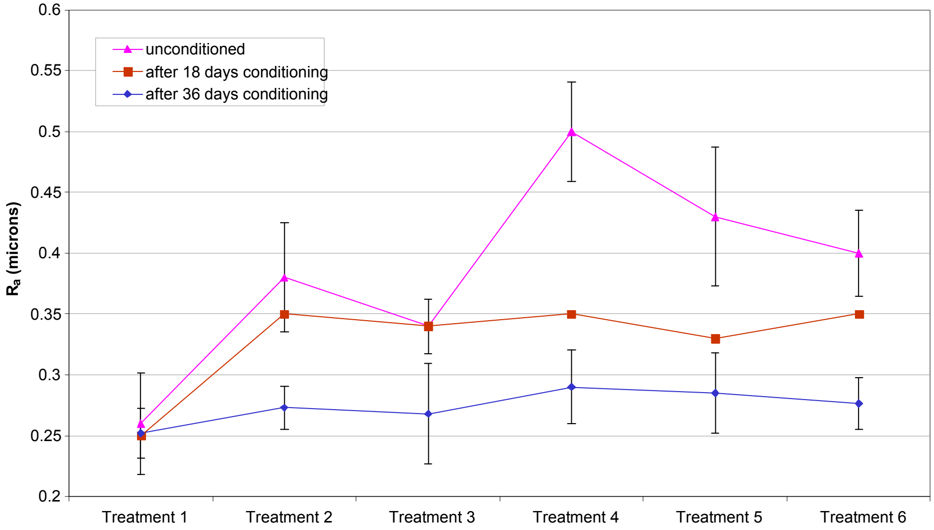

Figure 9. Surface roughness measurements

Surface energy

Wetting is the spreading over and intimate contact of a liquid (adhesive) over a solid surface (substrate). If sufficiently intimate contact is achieved between the two phases, a physical attraction develops causing the liquid to conform to the surface on a macro and micro scale, displacing air and thus minimising interfacial flaws. Good wettability of a surface is a prerequisite for ensuring good adhesive bonding, and hence a high surface energy provides a good bond. Considerable effort has been expended in developing simple wettability tests to assess surface energy/tension prior to bonding [6, 23, 38, 39].

Contact angles are closely related to wettability and adhesion, providing information on the heterogeneity and roughness of the adherend surface and the effect of surface treatments. A liquid (adhesive) will wet a solid (adherend) when its surface energy is lower than the solid surface energy. The equilibrium force balance at the solid-liquid boundary is given by Young equation for contact angles greater than zero (see Figure 10). The lower the contact angle, the greater the tendency for the liquid to wet the solid surface, until complete wetting occurs at a contact angle of zero. Large contact angles are associated with poor wettability and low adhesion forces.

The Young equation relates the contact angle θ, and the surface free energies of the liquid-vapour, solid-vapour, and solid-liquid interfaces (γ_{lv}, γ_{sv} and γ_{sl}, respectively).

$$\gamma_{lv} \cos\theta = \gamma_{sv} - \gamma_{sl} \tag{3}$$

The solid-vapour and liquid-vapour energies are defined with respect to air (the vapour pressure in air is material dependent and can be extremely low). The lower the contact angle, the greater the tendency for the liquid to wet the solid, until complete wetting occurs at an angle θ= 0 (cosθ = 1). The surface tension of the liquid is then equal to the critical surface tension of the substrate. The surface energy of a solid can be determined by using the Young equation and probe liquids with different surface tensions to produce a Zisman plot [6]. However, in most cases the contact angle is used as a relative measure of the surface energy.

Surface energy is sensitive to the chemistry of the surface, the morphology, and the presence of adsorbed materials. The adsorption of chemicals on a surface lowers the surface free energy (wettability). Surfaces with high surface energies will have a strong tendency to adsorb materials (e.g., moisture or dust particles) from the atmosphere, reducing wettability. Therefore, contact angle measurements offer a means of studying the ageing of surfaces.

Simple wetting tests

The water break test [40] is a qualitative (go/no-go) test, involving the specimen (in the form of a flat plate) being either immersed in water or water brushed or sprayed onto the surface. The plate is then checked to determine the distribution of water on the surface (i.e., remains as a continuous film indicating good wettability or forms distinct droplets indicating poor wettability).

The wetting or Dyne pen test is a semi-quantitative test [41, 42] that involves marking the surface with a pen containing ink of a known surface tension and observing whether or not the initial continuous line breaks into distinct droplets (indicating that the surface energy is lower than the surface tension of the ink). ‘Dyne solutions’ are available in sets of different surface tensions and systematic use can quickly provide an estimate of surface energy. Despite the widespread use of these solutions, the method has notable critics [43]. The shelf life of pens can be limited, particularly if in regular use, as transfer of contamination from surfaces to the ink may occur.

Contact angle determination by sessile drop

Contact angles between liquid drops and surfaces can be measured directly from the angle formed at the contact between the liquid and the flat surface [44-47]. Measurements can be made using a goniometer, an inexpensive instrument. The drop is illuminated from behind and viewed through a lens focussed on the silhouette of the drop. A reference line is manually positioned to read the contact angle. The drop may also be projected onto a screen to view the contact angle. Manual measurements can be prone to operator subjectivity.

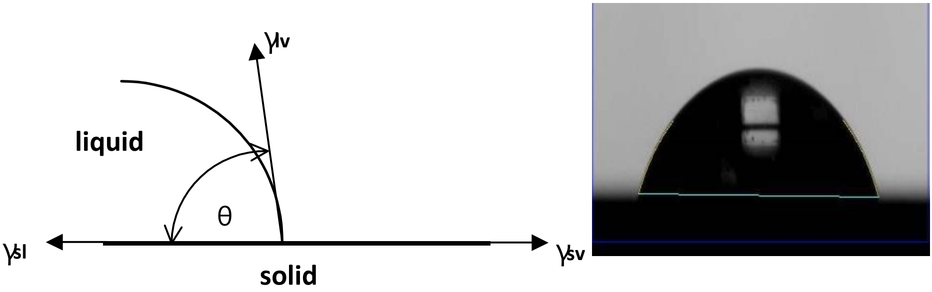

Figure 10. Contact angle measurement

Operator subjectivity can be removed from the process by image analysis of the drop shape and contact using a computer programme. There are many commercially available automated contact angle measurement instruments where the image of the drop is viewed using a video camera and captured on computer. An image analysis programme detects the edge of the drop and the surface. Numeric algorithms are run to establish the 'shape' of the drop and the slope of the edge at contact with the surface. The software will also analyse the shape of hanging pendant drops to measure surface tension of liquids. Axisymmetric Drop Shape Analysis (ADSA) is one example of a computer program that uses digital image analysis to detect the edge of a static drop and accurately determine the interfacial tension and contact angle [48]. ADSA is capable, under ideal conditions, of determining contact angles to very high accuracy with uncertainties of less than 0.1°. Through careful control of the position of the syringe plunger it is possible to make the drop advance and recede on the surface. Images of the drop can be captured in real time allowing measurement of contact angle hysteresis through the advancing and receding contact angles.

With automated contact angle measurements, the determination of contact angle should be straightforward provided that:

- The background illumination is of the appropriate intensity to achieve a good contrast between the drop and the background;
- The background illumination level is uniform;
- The camera achieves a sharp focus on the drop and base;
- The surface is flat and level (e.g., checked using spirit levels in two orthogonal directions);
- The plane of the surface is normal to the plane of the camera, i.e., the surface should not slope from left to right or from back to front in the image;
- The surface roughness is not significant (local topography at the junction of the drop and surface can lead to interpretation errors).

During contact angle measurements drops tend to spread on the surfaces with time and the volume of the drop may change due to evaporation or absorbance of moisture. Figure 11 shows a measured contact angle decreasing with time. The procedure for taking measurements needs to specify the time after dispensing the drop at which the measurement should be made, to reduce the measurement variability. A duration of 30s after dispensing the drop is suggested.

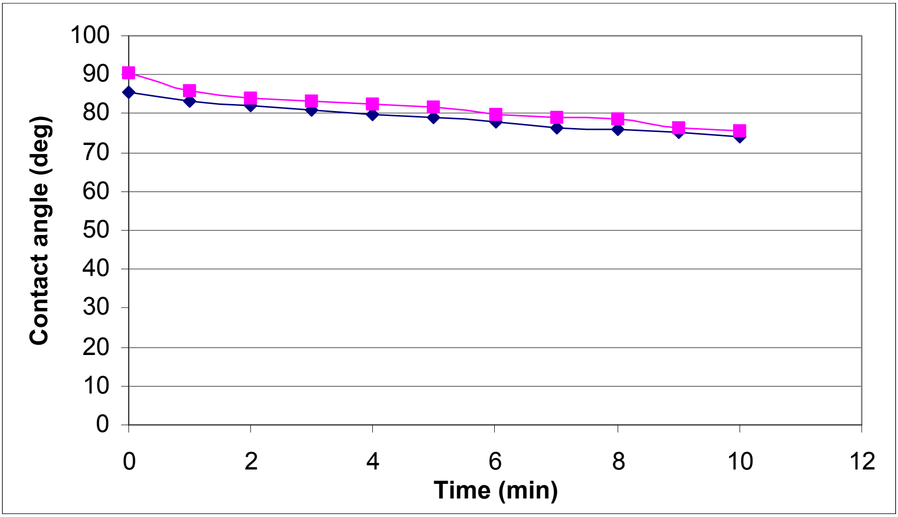

Figure 11. Time dependence of contact angle measurements

Wetting balance

Contact angle can be measured using the Wilhelmy plate technique [38, 39, 49, 50]. This technique measures the forces exerted as the solid-liquid phase boundary is moved along the sample surface as the plate is withdrawn from the liquid (Figure 12). The sample is attached to the balance (with a resolution of 1 µg) and the balance tared to remove the weight of the sample. The liquid (of known surface tension) is raised to meet the solid and the point of contact is determined and recorded as zero immersion depth. The plate continues to be lowered into the liquid to a set depth and the forces on the balance are continuously monitored. The process is then reversed and the solid is raised from the liquid. The contact angle θ may be calculated from the interaction force *F*, sample geometry (perimeter of the edge, *P* = 2 *x* length + 2 *x* thickness) and liquid surface tension (γ_l).

The forces involved are:

$$F = Wetting\ Force - Buoyancy \tag{4}$$

Buoyancy is accounted for by extrapolating the force back to the point of zero immersion depth. The force is given by:

$$F = \gamma_l P \cos\theta \tag{5}$$

When the contact angle is zero (i.e., perfect wetting) the value of F/P should reduce to the surface tension of the liquid.

Standard methods for wetting balance tests have been produced for determining the efficiency of the wetting of wires by solders [49, 50] but have yet to be standardised for the evaluation of substrates. When carrying out Wilhelmy plate measurements the following points should be considered:

- The edges of the plate immersed should be straight and meet at right angles;
- The plate should be attached vertically so that it meets the surface perpendicularly;
- There should be no bubbles adhering to the surface of the plate when immersed;
- The plate should not float in the liquid.

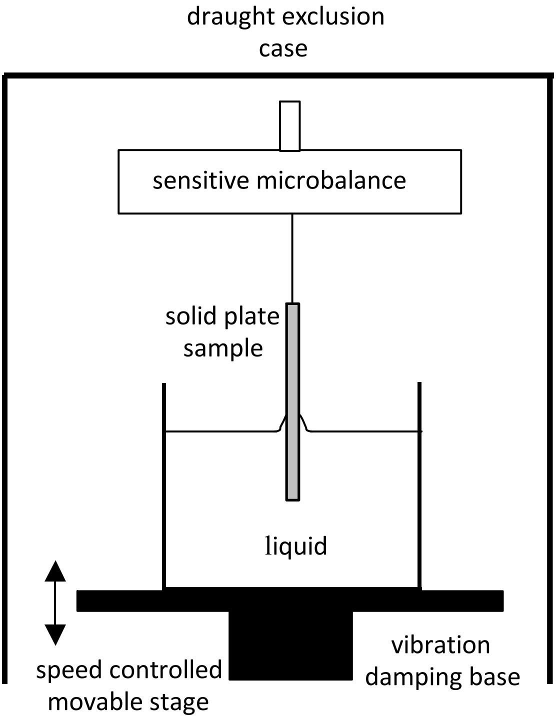

Figure 12. The Wilhelmy plate method

Mechanical properties of surfaces

Strong adhesive bonds require that the surface of the adherend is strongly attached to the rest of the substrate. Weak oxide films, coatings or boundary layers can initiate premature failure. This section describes techniques that can be used to assess the effect of surface treatments or environmental exposure on the mechanical properties and stability of surface layers.

Hardness and indentation

Hardness refers to the ability of a material to resist permanent indentation or deformation (or compressive loads) when in contact with an indenter under load (Figure 13) [51]. Hardness can be assessed on different scales depending on the depth that the indenter penetrates. The basic principle behind hardness testing is to load an indenter into a surface and measure the deformed imprint after retraction of the indenter. The indenter may be spherical (Brinell test [52]), pyramidal (Vickers [53] and Knoop [54] tests) or conical (Rockwell test [55]). The indentation is generally inspected using an optical microscope attached to the hardness test apparatus. Hardness data is defined in terms of the indenter geometry and for visco-elastic/visco-plastic materials the duration of loading. In the Brinell, Vickers and Knoop tests, hardness is the applied load divided by the unit area of the indentation and is expressed in kgf/mm^2. In the Rockwell test, depth of indentation is measured and converted to a hardness number (no associated units), which is inversely related to depth.

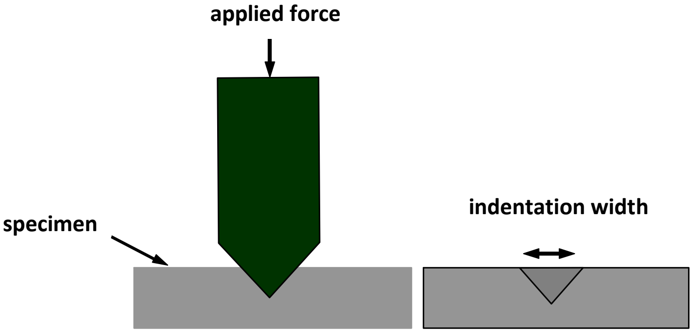

Figure 13. Hardness measurement

For standard hardness measurements, an indentation load of 30 N (or greater) is used and the size of the indentation is approximately 300 µm in size in the plane of the tested surface.

Micro-hardness testing involves smaller indenters and significantly smaller loads (10 mN to 10 N). The corresponding indentation is far smaller and is associated with local properties, whereas standard hardness tests provide bulk hardness measurements and may not distinguish the properties of the surface region. However, it is also more difficult to measure residual imprints for micro-hardness tests, as the residual imprints are relatively small.

Indentation tests instrumented for deflection and force, such as micro- or nano-indentation [56], provide more usable information on the mechanical properties of the near-surface material (including stiffness, plasticity, and fracture). Continuous stiffness measurements during indentation can be used to determine mechanical properties as a function of indentation depth. These techniques can provide very sensitive measurements of the mechanical properties of the surface layer. Analysis of the stiffness data can provide information on fracture and debonding. The combination of continuous stiffness indentation followed by AFM imaging can reveal much information about the surface. However, sensitive instrumented indentation instruments capable of high-resolution measurements are expensive and require specialist operators. Measurements are very sensitive to external vibrations and temperature fluctuations.

Hardness tests can be applied to a wide range of materials and used to characterise the mechanical structure of surface layers (e.g., oxide films or coatings) [56]. Hardness measurements have been shown to be sensitive to surface treatment [27], as shown in Table 4. The associated scatter in hardness measurements can be high, thus differentiation between the levels of treatment may be difficult. There are numerous hardness standards (e.g., [52-55, 57, 58]).

Surface Treatment	Microhardness (Vickers Hardness No.)	Ultra-micro Hardness (N/mm^2)
Aluminium		
As received (untreated)	80-110	1,400
Phosphoric Acid Anodising (PAA)	65-84	2,900
Chromic Acid Anodising (CAA)	79-135	19,000
Titanium		
As received (untreated)	258-352	10,800
Sodium Hydroxide Anodising (SHA)	234-248	800
Chromic Acid Anodising (CAA)	322-369	12,500

Table 4. Hardness measurements for surface treated aluminium and titanium [27]

Scratch testing

The adhesion scratch test is a commonly used method of assessing coating adhesion (see Figure 14) [51, 56]. The test is a variant on a hardness test and is designed to generate stresses at the interface between the coating and the substrate, which exceed the interfacial bond strength of thin, high adhesion coatings or films. A loaded diamond tipped stylus (10 to 60 N) is drawn across the target surface with a steadily increasing load (typically 100 Nmin^{-1}) until some well-defined failure occurs, usually flaking or chipping. The horizontal displacement rate is nominally 10 mm.min^{-1} with sample size being typically 25 mm × 15 mm.

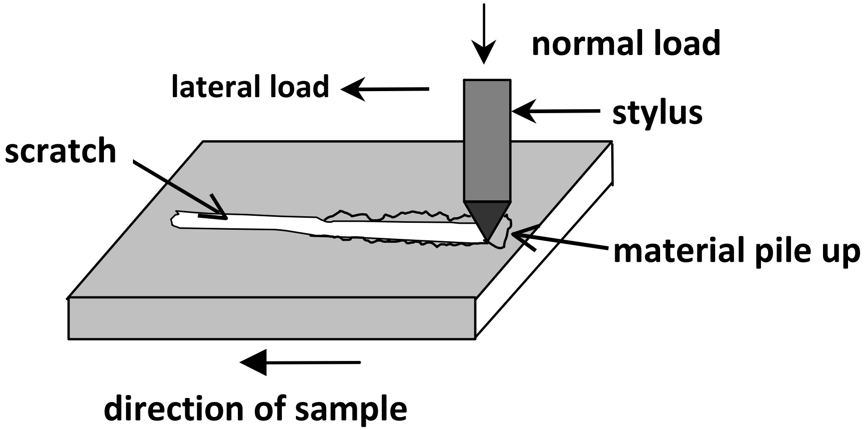

Figure 14. Scratch test method

During the test, the penetration depth and stylus position are recorded. These data can be supplemented with the vertical indenter load, the horizontal force on the indenter and acoustic emission to enable the coefficient of friction and the point of failure to be determined. The scratch can be subsequently analysed with a profilometer, SEM or optical microscopy to ascertain the scratch shape (residual depth, scratch width and pile-up height) and allow the failure mechanism to be identified. Varying the loading rate, the scratch speed and the indenter shape markedly affect the results of the test. Scratch testing equipment is commercially available. Measurements are comparative although there has been recent work to provide a scratch test calibration procedure, draft standards, and reference materials.

Modulus by surface acoustic wave

The propagation velocity of acoustic waves in a medium is proportional to the square root of (elastic modulus divided by density). Surface acoustic waves have very little depth penetration and therefore provide information on the properties of the surface layer rather than the bulk of the material. The laser surface acoustic wave (L-SAW) technique [59] focuses a short laser pulse onto the surface to generate a sound wave that propagates along the material and is detected by a piezoelectric foil. The characteristics of the propagating wave (velocity, wavelength, intensity) can be analysed, using complex functions, to infer the density, Poisson ratio and elastic modulus for both the substrate and the film.

Electrical and optical properties of surfaces

Surface resistivity

The electrical impedance of a surface will change with the surface properties and offers a means of monitoring changes to surface characteristics. A method of converting electrical properties to the surface characteristics is needed to relate measurements to bonding parameters.

Surface resistivity is defined as the electrical resistance of the surface of an insulator and is expressed in ohms/m^2 [60, 61]. An electrical potential is applied between two electrodes on the surface of the test and the resultant current is measured. The surface resistivity σ is determined [60] using:

$$\sigma = K_S \frac{V}{I} \tag{7}$$

where V is the applied voltage, I is the measured current and K_s is the test cell constant for surface resistivity based on cell geometry.

A configuration for measuring surface resistivity is shown in Figure 15. The current is only measured between the bottom two electrodes as shown in the diagram. The top electrode is guarded so that only current flowing between the two lower electrodes is measured (by a picoammeter). The key test parameters are applied voltage, length of electrification time, humidity, and temperature. The longer the voltage is applied, the higher the resistivity. This is because the sample charges exponentially with time. When measuring materials with high surface resistivity, background currents due to charging, static or triboelectric charge, or piezoelectric effects, may cause significant measurement errors.

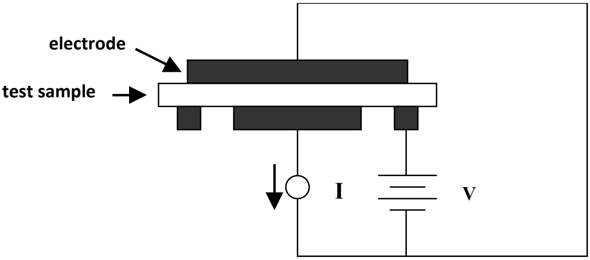

Figure 15. Surface resistivity measurement

The Alternative Current Method is a variation on the surface resistivity method where a bias polarity of positive polarity is applied, and the current is measured after a specified delay time. The polarity is then reversed, and the current is measured again using the same delay time. This process is repeated, and the resistance is calculated from a weighted average of the most recent measurements.

AC impedance techniques have proved successful for characterising oxide films and coatings. Based on an electrochemical method used for corrosion studies to measure oxide growth rates, the test surface is immersed in an electrolyte and an alternating voltage is applied. The resultant current is measured as a function of frequency. Surface impedance is determined from the resistance, capacitance and inductance of the surface and the resistance of the electrolytic solution. It is possible to relate film thickness, film composition and oxidation rate for surface anodisation treatments using impedance measurements. The AC impedance technique can differentiate between various surface treatments and levels of treatment for a given surface treatment.

Electrical techniques are sensitive to variations in the composition and thickness of the oxide film and surface contamination. The electrical and environmental conditions should be kept constant to ensure consistent data (e.g., surface impedance decreases with increasing humidity levels). Poor electrical contact between sample and electrodes will also have an adverse affect on the reliability of surface resistivity measurements. The electrode area must be equal to the contact area since any discrepancies will contribute measurement errors. Although the technique is unsuitable for on-line inspection, there is considerable scope as a developmental tool and for modelling oxidation processes.

Electrical measurements are used as non-destructive testing (NDT) methods for detecting the presence and monitoring the evolution of cracks in metals [62-65]. These techniques may also be suitable for monitoring changes in surfaces. The alternating current potential drop (ACPD) takes advantage of the fact, that when passing through a metal, an alternating current is not distributed uniformly through the depth of the metal. Instead, it is forced to flow mainly in a thin surface layer, an effect often referred to as the "skin effect" Thus, variations in the surface will cause measurable perturbations in the electric field [62-64]. The eddy current (EC) method as a non-destructive tool was developed in the 1940s. The technique has been widely used for crack detection in aerospace, power and oil and gas industries, and has been standardised as an NDT method in many applications.

The principle of eddy current is simple [65]. Eddy current measurements are generated in the sample by exciting an alternating current at a given frequency through a coil, often called a probe coil, located as near as possible to the component to be inspected. As a result of the eddy current in the component, changes take place in the impedance of a pick-up coil. The impedance of the coil is sensitive to the thickness of insulating coatings and is also affected by the presence of defects in the component. Therefore, under a given test condition where the design of the probe and the properties of the component are known or constant, the defect can then be detected and sized by measuring the impedance (phase and amplitude) characteristics in a pick-up coil above the surface.

Ellipsometry

Ellipsometry is an optical technique for probing the properties of thin films (less than the wavelength of the incident light) [27, 66, 67]. A polarised light beam (usually a laser) is directed onto the surface at an oblique angle of incidence. The phase change and intensity (amplitude) of the reflected light are measured. The interference of multiple reflections within surface layers provides information about the layer thickness *t*, optical constant *n* (index of refraction) and extinction coefficient *k* (a measure of absorption) for each layer interacting with the incident beam. The optical properties of isotropic materials can be represented by the complex index of refraction $\tilde{n}$ ($\tilde{n} = n + ik$). Ellipsometry can yield information about the thickness, morphology and chemical composition of layers that are thinner than the wavelength of the light source.

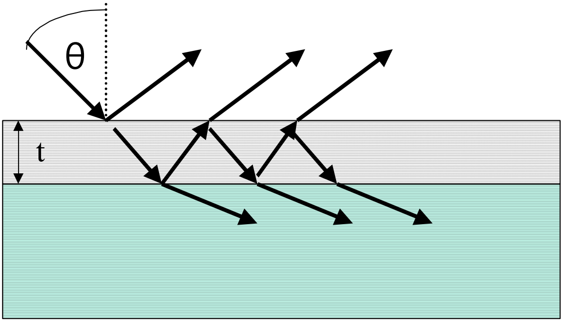

Figure 16. Schematic of multiple reflections

Most ellipsometers use a fixed incidence angle. However, Variable Angle Spectroscopic Ellipsometer (VASE) instruments give increased resolution [66]. VASE ellipsometers can also incorporate infrared spectrometry (IR-VASE) and provide a powerful technique for probing the optical properties, chemistry, and thickness of surface films. The molecular bond absorption bands for different surface treatments are distinguishable through IR ellipsometry data making it possible to fingerprint the surface chemistry (molecular bonds) present in the surface layers. The presence of a barrier layer or contamination will result in a different molecular bond absorption fingerprint.

Ellipsometry analysis requires a baseline set of known properties of the surface (measured on a 'clean' fresh surface and the surface film (e.g., Al_2O_3 ($n = 1.766$)) in the case of aluminium. The determination of film thickness is achieved by modelling the optical properties and requires a good knowledge of the optical properties of individual layers. Multi-layered systems can be modelled but this is a complex process and will lack accuracy unless information on each material layer is available. Ellipsometry measurements of the thickness of films [25] produced on aluminium using different levels of anodisation agreed well with transmission electron microscopy (TEM). Ellipsometry also provided evidence of differences in optical properties in the X- and Y-directions resulting from the milling process.

Optically stimulated electron emission

Optically stimulated electron emission (OSEE) is a method for monitoring surface contamination and surface treatments for metallic substrates [25]. It is highly sensitive to very low levels of contamination and has been successfully used for determining surface characteristics in multi-stage processing. OSEE operates by illuminating the area of inspection with ultraviolet (UV) radiation in the presence of a direct current (DC) electric field (see Figure 17). The UV radiation liberates electrons in the area of inspection by the photoelectric effect. The free electrons are collected on the positively charged anode with the magnitude of the resultant current being strongly related to the level of surface contamination. Contaminants absorb the UV radiation, thereby reducing the number of electrons emitted from the substrate, thus the greater the current the cleaner the surface. OSEE is particularly suited to studying contamination build-up following surface treatment and could be used to determine maximum time allowable between treatment and application of an adhesive (i.e., open time).

Six different aluminium surfaces were inspected using a portable OSEE unit. This instrument is suitable for both hand-held and on-line inspection (scan facility) and requires minimal operator training. A total of six measurements were taken for each condition and the results averaged. The units of measurement for the technique are based on a voltage relative to the condition exhibiting the highest output, which will depend on the nature of the substrate, cleanliness, etc., as well as the gain used to amplify the signal.

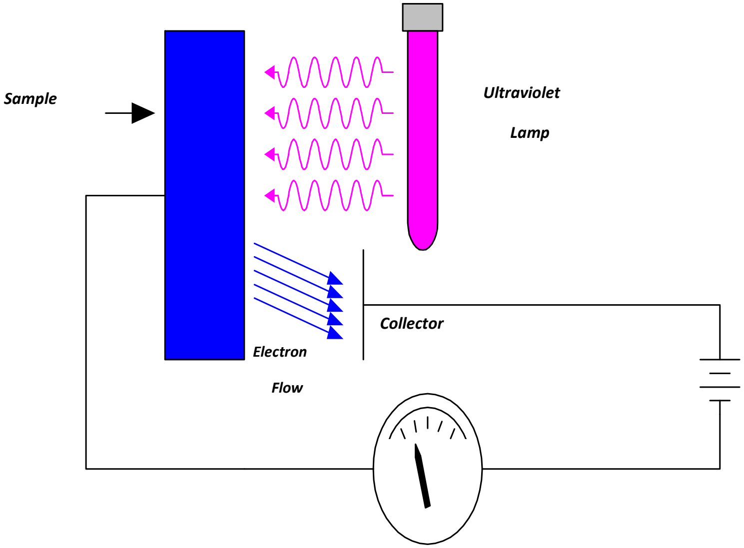

Figure 17. Optically stimulated electron emission (OSEE)

In general, as the cleanliness of the metal increases, the OSEE reading will increase due to a decrease in the attenuation of electrons by organic contaminants. However, as the thickness of the oxide layer increases, the OSEE will decrease due to an increase in electron attenuation by the oxide layer [24]. The results for the anodised treatments examined tend to correlate with these trends, Figure 18. The mill-finished material exhibits the lowest output due to the heavy oxide and organic contaminants on the metal surface. The highest readings were obtained for the lightly cleaned material, which is followed closely by the full electrolytic etch. Once the barrier and/or anodised film are applied, the OSEE output drops dramatically due to the increase in electron absorption by the thin films. There are minimal differences in the readings obtained for full clean + 25 nm barrier film ("optimised" system), light clean + 25 nm barrier and full clean + over-anodised - since the UV source is of a fixed wavelength so a substantial portion of the electrons are absorbed.

Note 1: Contaminants or thin films on the surface, depending on their own photoemission characteristics, can either enhance or attenuate the inherent emission from the surface as well. It also should be mentioned that there could be considerable variability in measurements depending on the uniformity of the surface with respect to cleaning and/or film thickness.

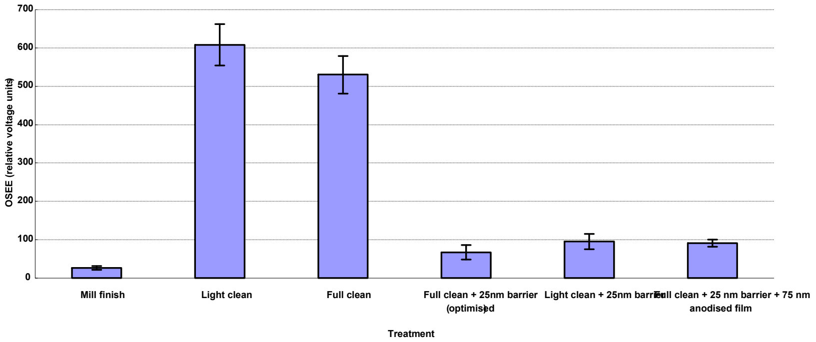

Figure 18. OSSE measurements on anodised aluminium (courtesy of Alcan, Ontario)

In summary, OSEE is suited for on-line inspection to determine the level of cleanliness or degree of treatment but unable to provide absolute measurements of surface activity for different treatments or ageing times. The technique is only able to detect barrier layers/cleaning levels through changes relative to initial substrate surface measurements. Hence, calibration is required for each surface to be assessed. Efficacy is compromised when contaminants fluoresce. OSEE is a non-contact, non-destructive technique that provides quantitative data. It is fast and relatively inexpensive, and straightforward to operate. The technique can be used to assess different methods of cleaning or the effectiveness of different cleaning agents.

Summary of surface inspection methods

Many techniques are useful for determining changes in surface properties for different levels of treatment, although the range of application of these techniques to many materials systems is generally limited. A study of applicability of several methods for a range of surface preparations was undertaken [25] and the results of this study are summarised in Table 5. Additional work was then undertaken to investigate some of these tests for following changes in surfaces due to ageing and accelerated conditioning [32].

Colorimetry is a versatile technique for detecting changes in surface morphology but, while it is possible to quantify changes in reflectivity and relate these changes to levels of treatment or various stages in production of multi-layered systems, the level of uncertainty can limit the ability to distinguish between similar levels of treatment.

Ellipsometry can be used to accurately determine layer thickness of multi-layered systems, provided the analyst has a good knowledge of the technique and optical properties of individual layers within the system.

The sessile drop method appears more reliable than the Wilhelmy plate technique in differentiating between surfaces with good and poor adhesion. The Wilhelmy technique requires considerable care in specimen preparation, whereas there are no special preparation requirements for the sessile drop method. Contact angle appears most useful for assessing bonding to polymers.

The portable units used for measuring surface roughness, surface resistivity and gloss required minimal operator training and were relatively inexpensive. These techniques can distinguish gross differences in treatments (e.g., untreated areas from treated areas) but are not sensitive enough to separate different levels of treatment or conditioning. OSEE is an on-line production tool for inspecting surface quality of metal substrates but is limited in its use for determining the surface activity of aged materials (i.e., off-line).

Corona discharge trials demonstrated the methodology and logic behind planning and conducting a matrix of tests in a systematic manner using statistical design of experiments (DoE) approaches [68]. Combined with surface characterisation techniques, such as colorimetry and the sessile drop method for measuring contact angle, this approach enables production engineers/researchers to assess the effects and non-linearity of key processing factors and possibly optimise process conditions.

Characterisation Technique	Surface Treatment				
	Grit Blasted	CAE	Anodised	Oil Lubricated	Corona* Discharge
Micro-hardness	No	No	No	No	No
Contact Angle	Yes	Yes	Yes	No	Yes
Profilometry					
Contact	Yes	Yes	Yes	No	No
Optical	Yes	Yes	Yes	No	No
AFM	Yes	Yes	Yes	No	No
Gloss Reflectivity	Yes	Yes	Possibly	No	No
Colorimetry	Yes	Yes	Yes	Yes	No
Ellipsometry	No	Yes	Yes	No	No
OSEE (on-line)	Yes	Yes	Yes	No	No
Surface Resistivity	No	No	No	No	No

Table 5. Suitability of characterisation techniques for different surface treatments
*(*Corona discharge treatment of glass fibre-reinforced polypropylene)*

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Chapter 3

Adhesive joint tests

- Introduction
- Overlap test methods for adhesives (tensile-shear)
- Peel test methods for adhesives
- Fracture test methods (cleavage – thick sections)
- Tensile test methods
- Flexure test methods

Introduction

Chapter 3 of the Guide focuses on (joint) test methods for characterising strength of adhesion between polymeric adhesives and substrates. Adhesive joints tend to be designed on the basis of perfect bonding with the assumption that the locus of failure will be in either the adhesive layer or the adherends. The properties of the interface tend to be ignored in the design, in part because reliable properties for the interface are difficult to obtain. It is common design practice to employ Finite Element (FE) methods to predict structural stiffness and stress distributions. The approach is often to model the continuum behaviour of the adhesive layer in order to predict joint stiffness and strength [4] but fracture methods may also be employed (Appendix 3). To accurately predict the performance of the adhesive joint using Finite Element Analysis (FEA) the following information is required:

- Mechanical properties for the adherends and the adhesive;
- Dimensions of the parts;
- Constraints and loads on the part;
- Failure criteria for the materials employed;
- Failure criteria for the bond at the interface; and
- Material models for adherends and the adhesive.

It is assumed that information on the material properties of the adherends and adhesives, joint dimensions, constraints, and loading are available and can be modelled accurately. Methods suitable for determining material properties (elastic constants, yield functions and ultimate strength) of the adhesive needed for design are described in other Good Practice Guides [1, 4]. Failure can originate in regions of the joint where the stress exceeds the cohesive strength of the adhesive, which depends on many factors such as temperature, strain rate, load history and chemical exposure. However, failure can also start at the interface where the local stress exceeds the interfacial adhesion strength and this needs to be considered. The strength of adhesion throughout the lifetime of the bond will depend on many factors, including:

- Mode of loading;
- Relative stiffness of the adhesive and adherends;
- Attractive forces between the adhesive and the adherend;
- Presence of defects or contamination;
- Degree of intimate contact between the adhesive and surface;
- Surface properties of the substrate;
- Material properties of adhesive and adherend in the interfacial region, which may differ from the bulk and change during service; and
- Degree of chemical or physical degradation of the interface.

The complexity of the interfacial region makes it impossible, except perhaps in very simple model systems, to predict the strength of an 'adhesive bond' from first principles. Therefore, to determine adhesion strength parameters, adhesive joint tests will be required.

There are many test methods available but their usefulness for establishing quantitative adhesion strength is variable [69]. Even in simple joint configurations, such as a single-lap shear joint, the resulting stress and strain distributions in the adhesive layer can be extremely complex with a mixture of shear, peel and hydrostatic pressure stresses occurring. Such situations are not easily amenable to closed form analytical methods and FEA may be needed to help interpret results. The preparation of joint test specimens is well recognised as having a critical role in the accuracy and reproducibility of test data and some guidance is reproduced in Appendix 2 [2, 3].

When considering a test method for establishing adhesive strength there are some basic requirements that should be considered. An appropriate test method should:

- Induce failure at the interface under study;
- Provide quantitative test data that are suitable for design calculations;
- Ensure a uniform, predictable stress distribution in the region of failure;
- Involve straightforward specimen preparation and robust test procedures; and
- Be accepted by users (e.g., through standards) and cost effective.

The choice of test method to study adhesion strength will depend significantly on the bending stiffness of the materials to be bonded and the likely loading vectors on the component. It is difficult to relate joint strength data from one configuration to a different configuration. Therefore, it is preferable to use joint tests that approximate the important modes of loading in the structure.

The basic modes of loading of adhesively bonded joints are (see Figure 19).

- Peel loads produced by out-of-plane loads acting on thin/flexiuble 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/thick adherends at the ends of the joints.

Tensile, cleavage or torsion loadings require two thick, rigid adherends. Peel loading requires at least one flexible adherend. Tensile-shear loading is the most versatile.

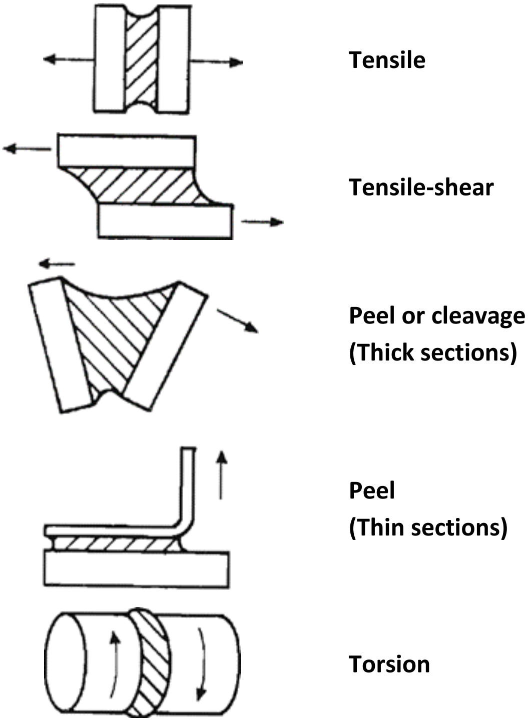


Figure 19. Basic adhesive loading configurations

Overlap test methods for adhesives (tensile-shear)

Shear test methods are widely used to obtain data on bond strength and durability [69]. Many of these tests are standardised and well accepted by the industry. Most shear test methods use specimens with flat adherends, bonded with an overlap, which are pulled in tension with the direction of loading parallel to the length of the overlap.

Single-lap shear test

[The single-lap shear test is ubiquitous in the field of adhesives and adhesion testing. National and international standards [70-75] set out the basic methods. Lap shear test results are extensively quoted on adhesive data sheets, often providing ‘evidence’ for ability to stick to different materials or of bond durability. The test specimen, shown in Figure 20, is loaded in tension until failure. BS EN 1465 [71] 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. It is good practice to use tabs to reduce out-of-plane deformation (i.e., bending).

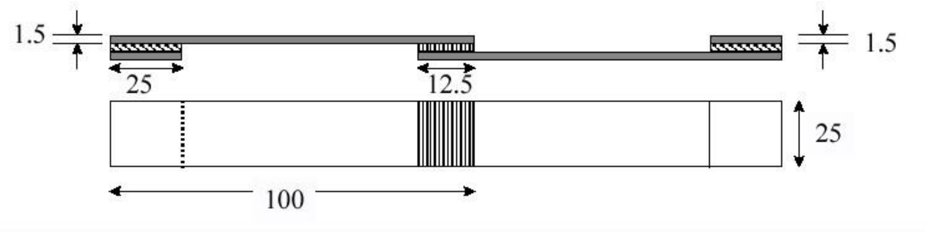


Figure 20. Single-lap shear test

The lap-shear strength τ is given by:

$$\tau = \frac{P}{bl} \tag{8}$$

where *P* is the maximum load, b is the joint width and *L* is the joint overlap length. The analysis assumes the adherends are rigid, and that the adhesive only deforms in shear. In fact, the resultant stress distribution, across and along the bond length is very complex and is dependent on adhesive and adherend properties, and joint geometry [76-80].

Single-lap joint tests are relatively straightforward from the perspective of specimen preparation and testing. However, these tests have recognised limitations for the accurate determination of joint design parameters. The basic mode of loading of the adhesive bond is in shear but, due to the eccentricity of the loading path, the critical end regions of the joint experience complex stress states with peel components. The directions and magnitudes of these stresses depend on numerous factors:

- Applied load and deflection (increasing either leads to an increase in stress);
- Mechanical properties of the adhesive (elastic modulus and yield stress);
- Mechanical stiffness of the adherends (determined by their thickness, elastic modulus and yield stress) - lower stiffness leads to higher peel stresses [4, 81]; and
- Geometry at end of overlap (shape of adherend corners, joint fillet) [76-80].

The geometry of the joint end can influence the location of the point of initiation of failure. Rounded adherend ends and fillets move the stress concentration from the corner of the joint into the fillet and failure may initiate through cohesive rupture at this region. For a single-lap joint, the maximum adhesive shear stress $\tau_{0,\ max}$ and peel stress, $\sigma_{0,\ max}$ can be calculated, assuming pure elastic behaviour, using the following Equations [10].

$$\tau_{0,max} = \frac{\sigma}{8}(1+3k)\sqrt{\frac{8G_a t}{E t_a}} \tag{9}$$

$$\frac{\sigma_{0,max}}{\sigma}\left(\frac{c}{t}\right)^2 = \lambda^2 \frac{k}{2}\frac{sinh(2\lambda) - sin(2\lambda)}{sinh(2\lambda) + sin(2\lambda)} - \lambda k' \frac{cosh(2\lambda) + cos(2\lambda)}{sinh(2\lambda) + sin(2\lambda)} \tag{10}$$

$$\sigma = \frac{P}{t} \tag{11}$$

$$k = \frac{cosh(u_2 c)\ sinh(u_1 L)}{sinh(u_1 L)\ cosh(u_2 c) + 2\sqrt{2}\ cosh(u_1 L)\ sinh(u_2 c)} \tag{12}$$

$$u_1 = 2\sqrt{2}u_2 \tag{13}$$

$$u_2 = \sqrt{\frac{3\sigma(1 - \nu^2)}{2Et^2}} \tag{14}$$

$$k' = kc\sqrt{\frac{3\sigma(1 - \nu^2)}{Et^2}} \tag{15}$$

$$\lambda = \frac{c}{t}\left(\frac{6E_a t}{E t_a}\right)^{1/4} \tag{16}$$

where:

P = load per unit width

L = length of overlap (bond length)

t = adherend thickness

E = adherend modulus

G_a = adhesive shear modulus (initial)

t_a = adhesive layer thickness

E_a = adhesive tensile modulus

ν = adherend Poisson's ratio

Finite element modelling is also used in the interpretation of stress and strain in the adhesive layer in bond joints. FE models allow the incorporation of non-linear properties (e.g., plastic yield), which are not included in analytical models but often play a critical role in the behaviour of joints, in the interpretation of the stress state. Figure 21 shows the effect of different adherend materials on the predicted peel and shear strain values in the standard lap joint specimen.

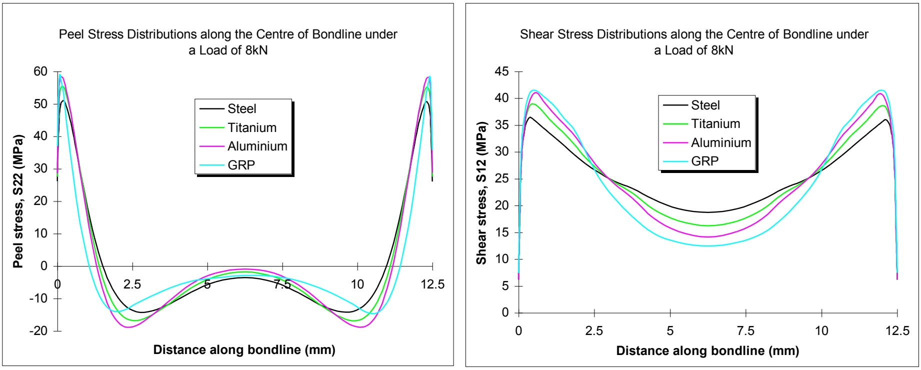


Figure 21. Effect of single lap adherend materials on strain distributions [80]

The single-lap joint provides the user with an “apparent” shear strength value. The apparent shear strength measured with a single-lap specimen cannot be assumed to predict the strength of joints of differing geometry. The thin lap shear test should be used only for the acquisition of qualitative data on adhesive performance.

Thick adherend shear test (TAST)

The thick-adherend shear test (TAST) is probably the most commonly used joint-specimen test for producing material properties under shear [82-85]. The thick and specially profiled adherends in this test produce more uniform stress and strain distributions in the adhesive than are obtained in a lap-shear test [85]. ISO 11003-2 [82] specifies a specimen with an overall length of 110 mm, a width of 25 mm and overlap length of 5 mm. The ISO standard recommends an adherend thickness of 6 mm and a bond-line thickness of 0.5 mm. Slots should be 1.5 mm wide. Load is introduced to the specimen preferably via two 12.7 mm diameter bolt holes. The hole centres are 80 mm apart. Care is needed to ensure that the holes are accurately drilled in the centre of each adherend, since small misalignments can result in unwanted rotation and uneven loading of the joint, thus compromising the test data. The test method is suitable for measuring shear modulus and shear strength of adhesive joints under ambient and hostile environments.

The pre-shaped adherends are more rigid than the bonded plates and reduce the peel stress in the test. However, specimen preparation costs are lower with bonded panels. A miniature TAST specimen, loaded in compression, has been developed at NPL that allows the testing of thinner adherends [86] – see Figure 22.

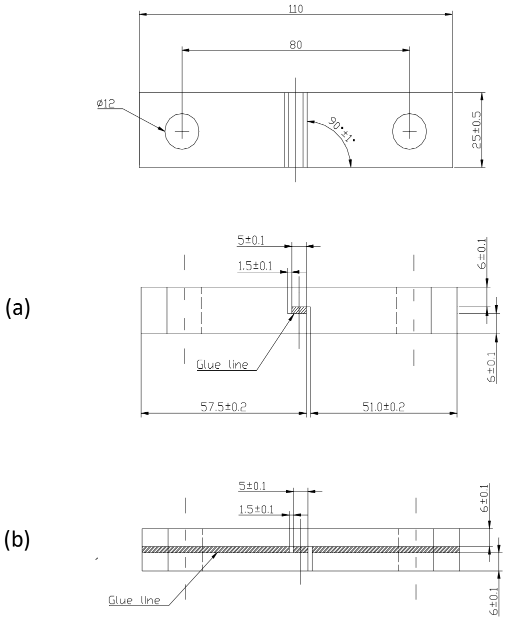


Figure 22. TAST specimen: (a) pre-shaped adherends, (b) bonded adherends (dimensions in mm)

The state of stress is predominantly shear but there are peel stresses at the end of the overlap. Failure occurs at the end of the bond line and is thus sensitive to the geometry of this region. The locus of failure tends to be close to the adherend and, thus, interfacial failure is a possibility. Cracks have been observed to run along the interface [84]. Higher joint extensions can be realised if the corners of the adherends and the spew fillet are profiled to remove stress concentrations. Stresses at the overlap ends are complex and difficult to calculate analytically but FE approaches have been used with some success [84, 85]. The stress state in the miniature specimen will differ from the standard TAST and this should be considered when interpreting results.

Double-lap joint tests

Double-lap joint tests were developed in attempts to eliminate eccentric loading, responsible for bending of the adherends and rotation of the bonded region [87]. Figure 23a shows a schematic representation of the symmetrical double lap joint. The central adherend is twice the thickness of the outer adherends. Although bending is reduced, peel stress at the outer adherends is unavoidable, since the load is applied to the outer adherends, via the adhesive, away from the neutral axis. Tapering the outer adherends can reduce the peel stress. Additional bending can also be introduced when clamping pressure is applied to the double lap end of the specimen, it is recommended that a rigid spacer be used to minimise this effect. The bending moment introduces tensile stresses across the adhesive layer at the free end of the overlap and compressive stresses at the other end. The centre adherend is free from the net bending moment but there is still an element of peel stress at the ends of the overlaps. The stresses at the joint end are sensitive to the mechanical properties of the adherends, the shapes of the joint ends and the geometries of the adhesive fillets.

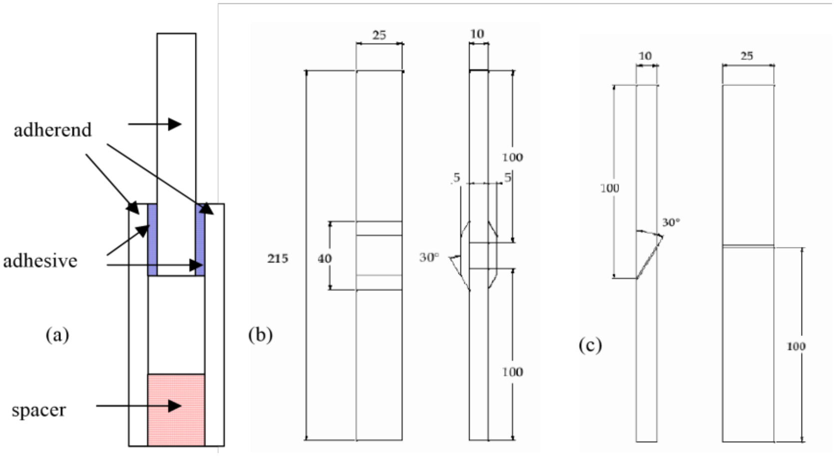


Figure 23. Overlap test geometries (a) double lap, (b) strap joint and (c) scarf

The strap joint consists of two thick adherends butted together. Straps (one for the single strap joint or preferably two for a double strap joint) are then bonded to the faces of the adherends to carry tensile loads in shear (in test specimens the gap between the butt-ends of the central adherends is free of adhesive). This geometry is also described in ASTM D3528 [87] as an alternative specimen to the double lap joint. The strap joint test reduces bending moments since the specimens are loaded through coaxial adherends. The square ended straps generate significant peel forces at the end of the strap. By tapering the straps peel forces at the ends of the adhesive layers can be reduced further.

The tapered strap joint, as shown in Figure 23b, can be used to generate adhesive design data [84, 88]. As with other lap joints, the adhesive in the strap joint is predominantly stressed in shear at the centre of the joint with peel stresses at the end of the overlap. Analyses of the strap joint suggest that a tensile stress concentration is inevitable at the end of the strap. The size and location of the stress concentration depends on the relative stiffness of the adherends and the local geometry at the end of the overlap. The shape of the fillet can determine whether the stress concentration is near the interface with the main adherend or the interface with the strap. This could be used advantageously to characterise the bond strength of an adhesive to either thick sections (central adherends) or thin sections (the straps). The central and strap adherends may be of different materials.

Double lap tests remove some of the disadvantages of the single lap test, but specimens are more expensive to prepare. The inner and outer adherends can be of the different materials and the location of the stress maximum can be controlled through the shape of the outer adherend ends and end fillets. Hence, this specimen has the potential to test the bonding of adhesive systems to materials that are available as either thick or thin adherends. It should be recognised that differences in bending stiffness of adherends could complicate the comparison of data determined for different materials.

Scarf joint tests

The scarf joint, Figure 23c, can be thought of as an ‘angled’ butt joint. When pulled in tension, a mixture of shear and tensile stress is generated in the adhesive layer. The proportions of shear and tension stress can be varied by varying the scarf angle [89]. A scarf angle of 90° to the axis of the specimen is a butt joint where tension dominates. An angle of 0° to the axis of the specimen is a lap joint where shear dominates. Scarf joints with lower scarf angles have higher strengths owing to their greater bonded areas and reduced tensile peel stress. The scarf angle and detailed geometry at the tips of the scarf sections influence the location of the stress maximum, near the interface that initiates failure [89]. The stress state at the stress concentration is complex and it may be difficult to quantify adhesion strength.

Other shear tests

There are many other test methods that impose shear stress on the adhesive layer. The butt and napkin ring torsion tests are known to provide a very uniform shear stress in the adhesive layer with minimal peel stresses [90, 91]. Shear strains to failure determined for adhesives in this test can be much greater than those determined in other tests. Torsion tests require test machines capable of applying loads in torsion and these are less common than tensile test machines. Failures seen in torsion tests appear to be cohesive in the adhesive rather than interfacial.

Pin and collar tests [92, 93] also test adhesives in shear. Specimens are easy to prepare and tests straightforward to perform by pushing the pin from the collar. However, there has been little investigation of the stress distribution in such specimens. A major concern is the possible sensitivity of results to the degree to which the pin is ‘centred’ in the collar.

Joint specimen tests based on bulk specimen shear tests such as the Arcan or Iosipescu tests can also be carried out on adhesives [94]. Failure will tend to initiate at a stress concentration at the end of the bond. The block shear test [95] is another possible method for testing adhesives but has found little favour owing to difficulties in interpreting and correlating results quantitatively.

Peel test methods for adhesives

T-peel tests

The T-peel test [96-98] is commonly used to assess the resistance of adhesive systems to normal force peel loading. The T-peel specimen is shown in Figure 24. The test requires flexible adherends i.e., those that can be bent through 90°. Tests are performed under constant separation speeds in a standard tensile test machine. The measured force-extension curve yields the maximum force and the peeling force (for steady state crack propagation). Peeling force (quoted as load per width) is normally the result quoted. However, work at NPL [98] suggests that the peeling force can be insensitive to environmental degradation and that the maximum force is a much more sensitive indicator of joint performance.

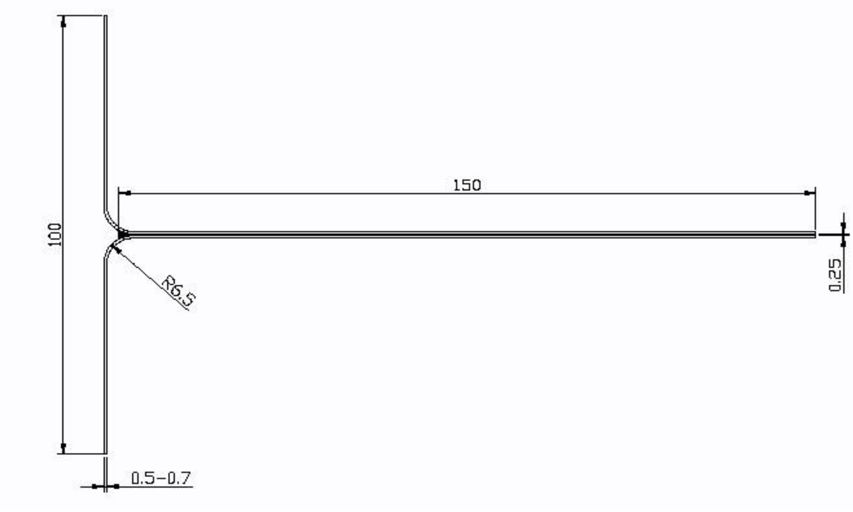


Figure 24. T-peel specimen (dimensions in mm)

The T-peel joint performance depends on joint materials and geometry. Most of the deformation in the test occurs in the adherends. Therefore, the thickness, stiffness, and plastic yield strength of the adherend material have major influences on the test results. The adherends bend during the test, changing the stress distribution. The degree of fillet in the joint influences both the strength and location of initial failure. Fillet size should be carefully controlled during manufacture [3]. The flexibility of the adhesive can alter the location of the stress maximum. With rigid adhesives, such as an epoxy, the stress concentration is at the end of the fillet. In the case of more flexible adhesives, such as a polyurethane [99] or 'moist' epoxy [98], the stress concentration is inside the fillet. The locus of failure and propagation route of the crack can vary between the interface and centre of the adhesive layer.

The evidence suggests that the T-peel test is unlikely to provide accurate interfacial strength data for design purposes. The crack propagation may not necessarily be along the interface. The maximum load borne by the joint is sensitive to many factors; in particular the local radius of curvature and peel angle of the adherend near the crack tip, and it is difficult to relate results to a material or interface property.

Flexible to rigid peel tests

There are several peel tests for assessing the bonding of a flexible adherend to a rigid adherend [100-106]. The main differences between the methods are the angles of peel and whether the peel angle remains constant during the test. Most of the energy is dissipated in the adherends rather than the adhesive. Accurate data on the elastic-plastic mechanical properties of both the adhesive and the adherends are important in the analysis of the test data [107-110]. Peel tests tend to be used to a greater extent for characterising flexible adhesives (including pressure sensitive adhesives) than they are for rigid structural adhesives. Peel strength is often quoted on adhesive data sheets. For structural adhesives, the T-peel test is more likely to be quoted than the methods outlined in this section.

The floating roller test method [103-105] determines the peel strength of a flexible-to-rigid bonded assembly. The roller mechanism enables a constant angle of peel to be maintained throughout the test. The test fixture can be adjusted to vary the peel angle. The average load obtained during stable peel is recorded as a characteristic of the bond.

The climbing drum peel test [106] is a standard test used to determine the bonding of sandwich structures. The drum fixture is used to peel the flexible skin from the rigid structure at a constant peel angle. Once again, the average peel load is measured. This method is used mostly in the aerospace industry.

Peel tests, while providing useful comparative data, are unlikely to provide quantitative measures of interface strength. There is no guarantee that the fracture path will be along the interface. Stress distributions depend significantly on local geometrical features such as crack sharpness and peel angle. Other than the aerospace industry, these tests seem to have found little favour in industry for generating data for the design of structural bonds. The conversion between peel load per unit area to stress requires assumptions about the extent of the peel zone that are likely to be inaccurate. Recent developments on the analysis of peel test results concentrate on the determination of fracture toughness properties [111].

Fracture test methods (cleavage – thick sections)

Fracture mechanics tests [13, 83-99] provide information on the growth of a fracture within a material. Stress is intensified at the tip of the crack. The fracture energy is determined from the stress intensity factor, crack length and the mechanical work applied. The quantities determined through fracture mechanics tests are the critical stress intensity factor K_c and the critical strain energy release rate G_c. The stress intensity factor is related to the geometry of the test specimen but K_c and G_c are both material properties. Analysis methods for bulk material fracture tests are well established, particularly for elastic materials. However, techniques for multi-material systems and materials that experience plastic yield are less well established, although theoretical developments are occurring to account for plastic zones [95] and rate/temperature dependence [99].

Fracture mechanics tests have been applied to adhesives. The common test methods are based on the double-cantilevered beam (mode I) and end notch flexure (mode II) tests. Mode I (crack opening) tests impose severe cleavage stresses on bonded joints. Fracture tests require an initial notch or pre-crack. The precise geometry of this notch will influence the results and is a source of uncertainty or variability in the tests. Notch geometry will have more effect on the initiation of crack propagation. Results from the initial part of the test are normally excluded from analyses with G determined from the regions of steady state crack growth.

Fracture tests are commonly performed to assess joint performance in hostile environments and results show sensitivity to the preparation of bonded surfaces. Fracture toughness is recognised as an important adhesive property, contributing to mechanical and impact performance.

Boeing wedge test

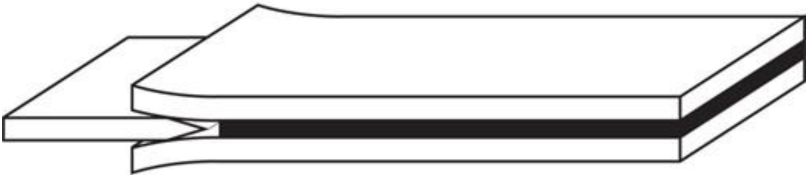


Figure 25. Wedge cleavage test (Boeing Wedge) test

The wedge cleavage test [112, 113], commonly referred to as the Boeing wedge test, uses the introduction of a wedge between two flat surfaces to force the adherends apart and impose cleavage stresses in the region of the crack tip, as shown in Figure 25. The wedge imposes a fixed displacement to the adherends, and the crack opening force is driven by the elastic stored energy in the adherends. Crack length is monitored with time, often using ruled scales attached to the adherends. The wedge test is simple and cheap to perform. No test machine is required since it is self-stressed. The stressed specimen can be exposed to hostile environments and the presence of chemicals at the crack tip can be expected to accelerate degradation much more effectively than in tests where these species must diffuse into the bond layer. Commonly, a crack growth limit is reached within several days making this test attractive for rapidly assessing durability. This test is considered a reliable method for assessing the environmental durability of adherend surface preparations.

Fracture energies *G* can be determined from the crack length *a* wedge displacement *w* adherend modulus *E* and adherend thickness *h*:

$$G = \frac{Ew^2h^3}{16}\left[\frac{3(a+0.6h)^2+h^2}{(a+0.6h)^3+ah^2}\right] \tag{17}$$

The accuracy of the fracture energy determined can be compromised by plastic deformation of the adherends (reducing cleavage forces) and adhesive. This test is not considered particularly accurate for fracture toughness measurements. The driving force depends on wedge insertion distance and the stiffness of the adherends. Although specimens can be manufactured with the initial crack at the interface there is no certainty that the crack will continue to run along the interface. FEA suggests that the regions of maximum strain and stress in the specimen, prior to crack propagation, are close to the interface.

The wedge test has been used to assess the comparative durability of adhesive systems and results suggest that crack growth rates in this simple test can distinguish between different surfaces and surface pre-treatments [69].

Compact tension test

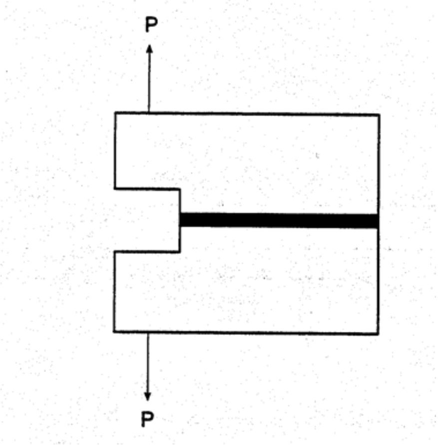


Figure 26. Compact tension test

Compact tension test [114] specimens are manufactured by bonding shaped adherends with the same geometry as the solid compact tension specimen, Figure 26. Specimens are 25 mm wide with a 25 mm long bond line. The depth of adherend is typically 12 mm at either side of the bond line. The specimen is loaded at one end of the bond producing a cleavage force. The test is run at a constant loading rate or crosshead speed until the joint is completely failed. The maximum load is recorded.

Double cantilever beam tests

The double cantilever beam (DCB) test [115, 116], Figure 27, is used to measure the initiation and propagation energy of a Mode I crack. The critical strain energy release rate G_{Ic} depends on crack length and is calculated using:

$$G_{Ic} = \frac{4P^2(3a^2 + h^2)}{Eb^2h^3} \tag{18}$$

where *P* is the applied load, *E* is the Young's modulus of the adherend, *b* is the specimen width, *a* is the crack length and *h* is the adherend thickness. Specimen preparation is straightforward. Thick adherend parts can be bonded and tested directly. Where the beams are formed from thin sheet material end tabs need to be attached to allow the beams to be gripped. The dependence of specimen compliance and strain energy release on crack length adds to the complexity of conducting and analysing this test.

The specimen compliance in the tapered double-cantilevered beam (TDCB) test, Figure 28, is independent of crack length [115, 116]. Compliance *C* is directly related to load *P*, width *b*, adherend modulus *E* and bending moment *m*, and G_{Ic}, proportional to the rate of change of compliance with crack length *a*, is given by:

$$G_{Ic} = \frac{4P^2m}{Eb^2} \tag{19}$$

The taper height is chosen such that *m* is constant with crack length a from the relationship:

$$m = \frac{3a^2}{h^3} + \frac{1}{h} \tag{20}$$

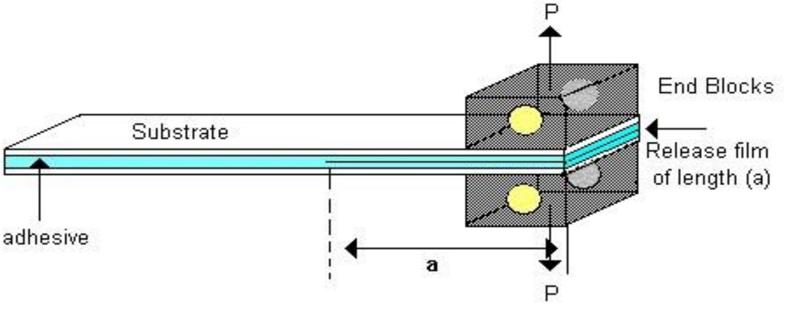


Figure 27. Double cantilever beam (DCB) specimen

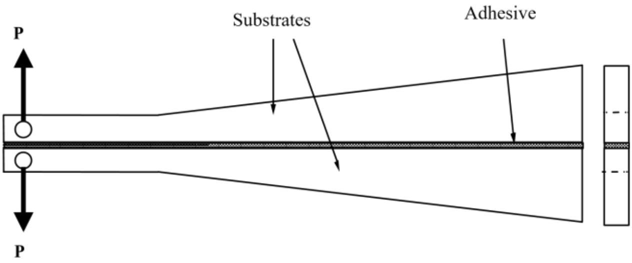


Figure 28. Tapered double cantilever beam (TDCB) specimen

Although TDCB specimen fabrication is more expensive than the DCB specimen the testing is much more routine and, provided that the adherend materials have high yield stress, the adherends should be reusable. Thin adherends can be evaluated using the TDCB test provided they are bonded to rigid components tapered to produce the necessary adherend dimensions. TDCB tests have been used to assess environmental durability. As with all fracture tests, information on the strength of the interface can only be obtained if the crack propagates along a surface.

End notch flexure test

The end notch flexure (ENF) specimen [96] is essentially the double-cantilevered beam specimen loaded in flexure (Figure 29). This test characterises Mode II (in-plane shear) fracture toughness. For small displacements (and negligible transverse shear deformation), strain energy release is calculated using:

$$G_{IIc} = \frac{9P^2a}{16Eb^2h^3} \tag{21}$$

ENF test is being developed as a standard method for Mode II fracture toughness for loading situations where Mode I do not dominate.

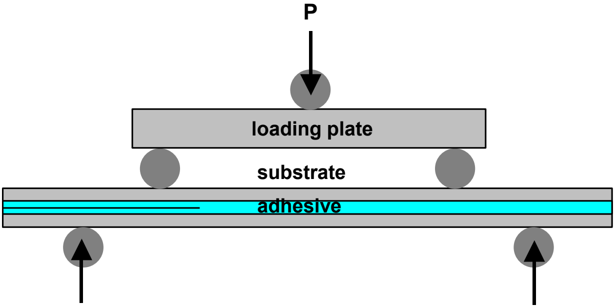


Figure 29. End notched flexure (ENF) test

Tensile test methods

Pull-off tests

Resistance to peel failure can be assessed from tensile pull-off tests. These [69, 118-120] are used widely to evaluate the adhesion of coatings to substrates. This type of test is used to assess adhesives, for example in the construction industry where it is used to determine the development of bond strengths, since it is simple to perform and can be carried out in-situ (either vertically or horizontally) on virtually any type of surface. The pull-off test is attractive as it is quick and simple to perform, requires low cost, commercially available equipment and produces a quantified measure of the adhesive strength from the maximum force applied to the sample. The test method and sample are illustrated in Figure 30.

A test pull stud is bonded to the surface under investigation. The stud is clamped in the test instrument. In some commercial instruments pneumatic pressure is applied, pulling the substrate against a restraining surface, until the stud is pulled off the surface and the maximum pressure is recorded. A calibration look-up table is used to determine the failure stress. Conventional tensile test machines can also be used to perform pull-off tests. The pull off test takes place in a short period of time; maximum load is reached in less than 1 s. The dynamic response of the transducer may introduce measurement errors.

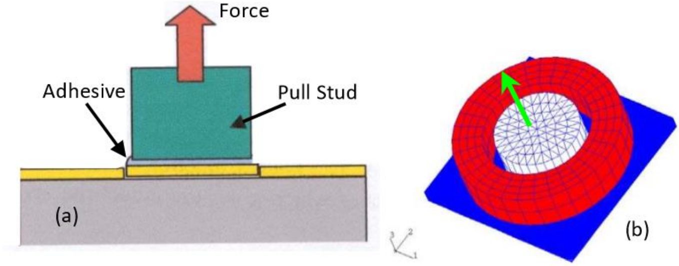


Figure 30. Pull-off test: (a) schematic of substrate; interface and pull stud, (b) FE model showing substrate, stud and 'restraining ring

Sample preparation is straightforward. Adhesive can be applied to either the substrate or the pull stud. These are then pressed together with the pull stud perpendicular to the sample and held in position whilst the samples are cured.

- It is important that the sample should not fail at the interface between the pull stud (or aluminium dolly) and the adhesive. The adhesive and pull stud need to be compatible and the surface of the pull stud prepared to achieve sufficient bond strength. It has been found that gritblasted aluminium provides a suitable surface for epoxy adhesives.

- The surfaces of the pull stud and the substrate should be flat and bonded so that they are parallel and the bond-line thickness is constant throughput the sample. Nonuniform bond thickness will induce cleavage stress in the sample leading to premature failure (see Table 6).
- Stress analysis indicates that the thickness of the adhesive layer has little significant effect on the stress distribution within the adhesive layer [120]. There is therefore little need to control the bond line thickness of the sample beyond ensuring that it is suitable for the type of adhesive being tested. The bond-line thickness can be controlled by mixing a small quantity of glass beads into the adhesive.The adhesive layer thickness should be constant in all regions of the sample.
- A spew fillet is likely to form around the pull stud due to displacement of adhesive from the bond region when pressure is applied. It is recommended that the fillet is not removed as this may damage the bond-line.

The test provides a value of pull-off strength as a ‘stress’ in MPa. This is the applied force divided by the bonded area (calculated from the radius of the pull stud). However, it is recognised that the stress will vary within the bond-line as the substrate will deform during the test. FE stress analysis, Figure 31, shows that the stress in the centre is very low and increases along the radius with the maximum being achieved at the edge of the bond (in the fillet should one be present).

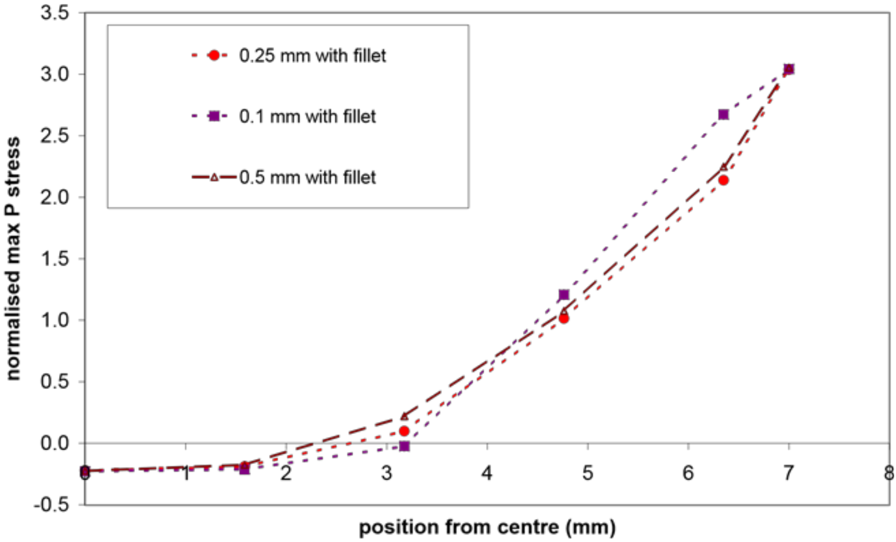


Figure 31. Analysis of maximum principal stress distribution normalised by the average stress in pull-off specimens with different bond-line thickness

The stress distribution in the bond-line depends on the bending stiffness of the substrate. The stress distribution becomes more uniform as the stiffness of the substrate increases. Under equal loads, adhesives bonded to substrates with low bending stiffness will experience higher stresses than they would if bonded to substrates with high bending stiffness. Therefore, care must be taken when comparing the bonding performance of an adhesive on different materials. In situations where the substrate is significantly more flexible than the pull stud (most thinner gauge materials), then the maximum stress is predicted to occur at the interface between the fillet and the adherend. However, as Figure 32 shows, failure is often cohesive within the bond-line, leaving the fillets firmly attached to the substrate.

Figure 32. Pull-off test specimen

Figure 32 also indicates that adherends can be deformed to a significant extent in the pull-off test. A significant amount of cleavage stress will be generated around the rim of the bond-line. Experience of using the pull-off tester suggests that in many cases the sample fails through cohesive failure of the adhesive – the results of the tests therefore provide a minimum value for the adhesion strength (see Table 6). Adhesion failures were associated with poor surface treatments.

Table 6 shows some test results for the pull-off test. The level repeatability in the measurements varies between 10% and 30%. The test shows significant differences between ‘good’ and ‘poor’ quality surfaces but is less able to distinguish between similar surfaces. The results show the importance of the care with which adherends are machined on the results – guillotining produces slightly bent adherends that lead to premature failures. Calibration drift may be an issue with this test. It is recommended that a stable and repeatable adhesive-adherend system (identified through testing of multiple batches) is identified and used periodically to check the performance of the test unit.

Surface and Treatment	Adhesive A and Aluminium, Pull-Off Strength (MPa) (Failure Mode: I = interfacial Adhesion, C = Cohesive)	
Aluminium (see Table 3)	**Guillotine/Sawn**	**Spark Eroded**
Mill finish	10.7 ± 0.8 (I)	12.1 ± 4.0 (I)
Light clean	13.0 ± 0.5 (C)	21.6 ± 3.8 (C)
Light clean + anodise	14.0 ± 0.5 (C)	19.6 ± 0.8 (C)
Full clean	14.8 ± 0.4 (C)	18.1 ± 5.4 (C)
Full clean + anodise	14.3 ± 0.7 (C)	24.0 ± 4.8 (C)
Full clean + over anodise	13.3 ± 1.3 (C)	23.6 ± 4.4 (C)
Steel Surface and Treatment	**Adhesive B + Galvanised Steel Pull-Off Strength, MPa**	
Mill finish	13.8 ± 0.9 (C)	
Light clean	5.0 ± 0.9 (I)	
Light clean + anodise	3.4 ± 1.1 (I)	

Table 6. Pull-off test results

Butt joint tests

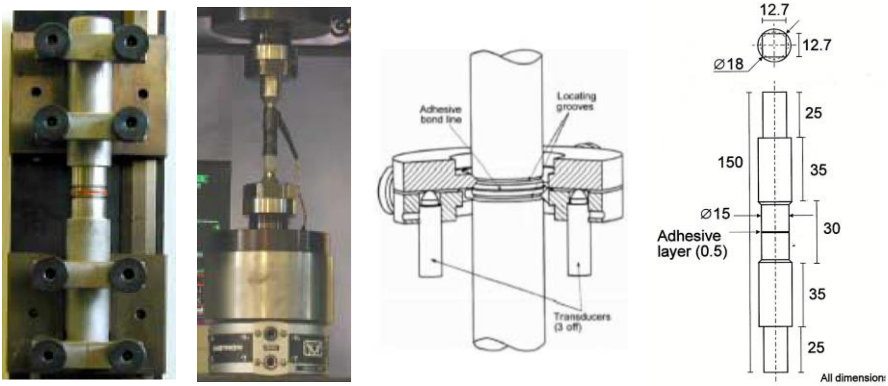
The butt joint test [121-124] is a severe test of an adhesive; specimens are prepared by bonding two rods or bars of equal cross-section together end-on. The joint is pulled in tension to obtain the butt tension strength. Tensile tests on butt-joint specimens have been used with suitable instrumentation to determine elastic properties and plastic property results that serve to check the validity of elastic-plastic models [4]. One use of this test has been to investigate adhesive yield strengths since the adhesive layer experiences high levels of tensile and hydrostatic stress but low shear stresses due to the constraints imposed by the rigid adherends [121]. This severe stress state also applies at the interface, thus providing information on the interface performance. There are several standards for butt joint tests but, as small misalignments can introduce high cleavage stress, it is difficult to obtain reproducible and accurate data unless considerable care is taken.

Accurate alignment of test specimen and loading assembly is critical in this test. The specimen is loaded through collet grips [124] that are rigidly mounted in the test assembly to minimise any lateral deformation of the specimen during loading. Without this constraint to lateral movement, the specimen is observed to bend during plastic deformation causing a non-uniform strain in the adhesive. Alignment is critical at all stages:

- During preparation of the test specimen where the accurately machined cylindrical adherends should be aligned carefully so that they meet precisely to form a straight rod with parallel bonded ends. The axial alignment of the butt joint specimen is closely controlled during manufacture using a precision manufactured V-block assembly jig, Figure 33, to ensure that the sample is bonded end-on. The adherend ends are precisely machined as 20 mm diameter cylinders from 25 mm rods. Slip gauges are used to position adherends and control their separation to obtain the desired bond line thickness (typically 0.5 mm). The specimen is clamped tightly during cure to prevent movement during this process.

- The grips of the test machine should be carefully positioned so that the direction of force application is precisely through the centre of the long axis of the test specimen. An alignment fixture is used to position one grip and enable the specimen to be mounted precisely in the test assembly. Figure 33 shows the fixture and specimen that are used to position the grips. The dummy specimen is strain gauged around the circumference (at equal angular separations) and the strain readings monitored when load is applied. The alignment fixture is adjusted until all the strain readings become equal to within a tight tolerance indicating that the test machine is aligned. Once aligned, the grips are locked rigidly in place and a final check is made of the dummy sample. If this is successful then the dummy sample is removed and test samples can be clamped.
- During tests, the extension at different locations around the circumference of the sample should be monitored to confirm that the loading remains axial. A suitable specimen geometry together with extensometers for the measurement of strain in the adhesive are shown in Figure 33. The specimen consists of two 25 mm diameter, cylindrical adherends that are bonded together at end faces. The v-shape groove in each adherend near the adhesive layer serves to locate an extensometer consisting of two rings with knife-edges on their inner surfaces. Each ring locates in one of the grooves in the test specimen. One of the rings supports three precision displacement transducers that are equally spaced around the ring. The core of each transducer contacts the surface of the second ring. The three individual displacement transducers provide a check on the axiality of the loading during the test. The readings will diverge should the specimen be misaligned. A value for the nominal axial strain in the adhesive under tension is determined from the average displacement divided by the adhesive layer thickness. A small correction can be applied for the contribution from the elastic deformation of the adherends to the total displacement recorded by the extensometer.

The standard butt joint sample consists of cylindrical adherends with flat faces. The sharp, square corners at the rim of the adherends leads to stress concentrations in the outer rim of the adhesive layer where failure will initiate. The sharp rim can be rounded to reduce but not eliminate the stress concentration. FE analyses show the peak stress in the bond-line to be approximately 10% higher than the average stress obtained by dividing the applied force by the bonded area [120]. This stress concentration, although lower in significance than found in many other joint tests, can cause problems with interpretation of results.



Bonding Fixture **Alignment Fixture** **Extensometry** **Butt Joint Specimen**

Figure 33. Tensile butt joint testing

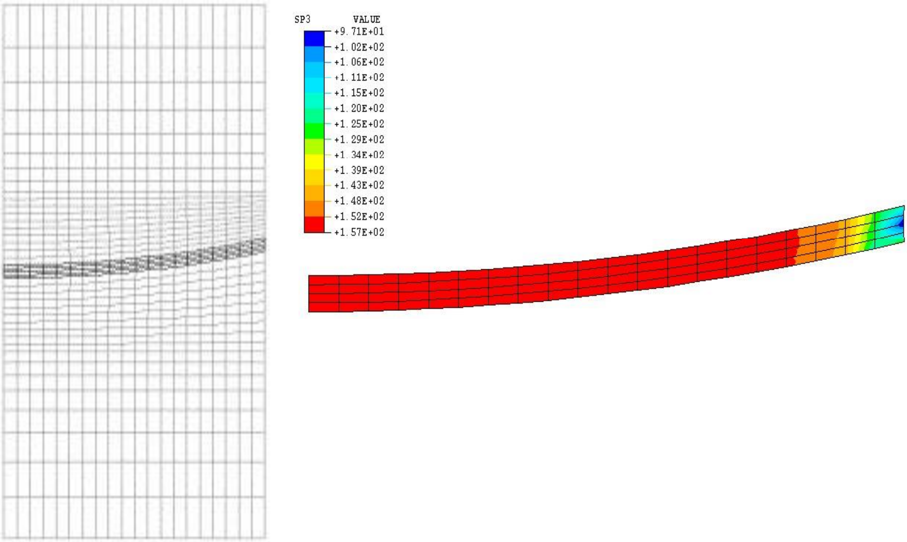


Figure 34. Profiled butt joint bond-line: (left) FE Mesh (rotational symmetry), (right) Maximum principal stress contour plot of the adhesive

Other profiling options, to reduce the stress concentration problem, were investigated with FEA. The most promising profile of those considered was when the butted adherend ends were machined as interlocking concave and convex spherical surfaces with 50 mm radii of curvature – producing a bowl-shaped adhesive layer with constant thickness, Figure 34. The radial distribution of tensile stress distributions predicted in the profiled butt joint specimen is compared the stress in a square ended butt joint in Figure 35. The region of highest stress in the profiled sample extends from the centre of the specimen over most of the adhesive layer. Stress values near the rim are lower. The highest stress predicted is close to the average stress (calculated as 51 MPa from the force divided by the bonded area).

Tensile butt joint tests performed on toughened adhesives using the profiled specimen showed considerable deformation after the adhesive yielded plastically, provided that the test system was correctly aligned (no deviations between individual extensometer readings). If the test system was not perfectly aligned, then the extensometer readings deviated, and the sample failed prematurely through cleavage. The well-aligned tests tended to fail cohesively indicating that the bonding strength exceeded the strength of the adhesive. The maximum load obtainable in these tests was limited by the yielding behaviour of the adhesive rather than failure.

The tensile butt joint test offers, provided sufficient care is taken, many advantages for studying adhesion strengths. However, there are many issues that will prevent widespread use of this technique:

- Tests are expensive to perform as to achieve the good levels of accuracy much care must be taken with sample preparation (including machining) and the setting of the test machine.
- The adherend dimensions must be large enough to produce samples of sufficient length and diameters, which excludes many materials from consideration by this test.

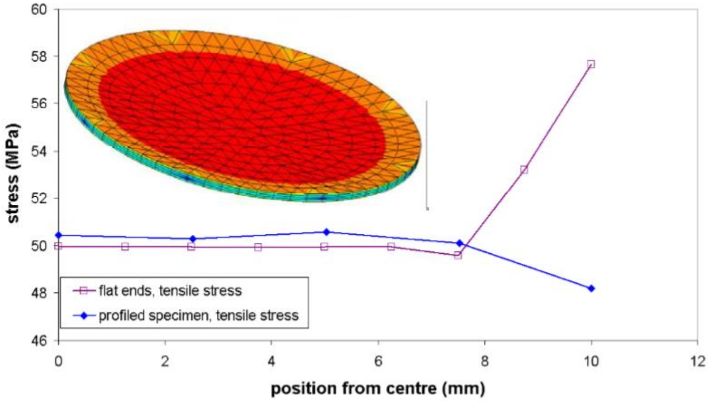


Figure 35. FE stress predictions in the butt joint test

Tensile pull-out tests

In fibre-reinforced polymer composites, the adhesion of the fibres to the resin is a key determinant of the strength of the system. Debonding of fibres from the matrix leads to failure of the composite. Pull-out tests were developed to assess the fibre-matrix adhesion to optimise fibre properties [125, 126]. Micro-pull tests determine the force required to pull embedded fibres from the resin. In order to provide improved interpretation of the data, analytical routines, based on elastic shear lag analyses, were developed to calculate stresses along the interfaces [126]. At first inspection, this test seems promising for adhesives. Specimens would be easier to prepare, and testing would be straightforward with a simple fixture. The largest stresses are at the interface, biasing the test towards interfacial failure and the initiation of failure would be easily identified from the sharp fall in load.

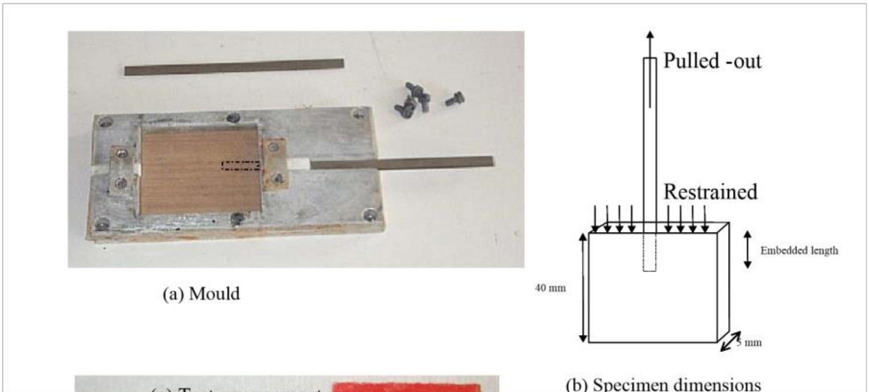
Most of the theoretical work addresses cylindrical fibres but practical adherends are rarely cylindrical. Therefore, an expression for calculating interfacial shear stress (IFSS) values, τ_i, along the surfaces of thin rectangular section shim adherends was developed, based on shear lag calculations for cylindrical fibres [120, 126].

$$\tau_i = n \frac{F}{wh} \frac{cosh[n\,(L_e - x)/r]}{2sinh(nL_e/r)} \tag{23}$$

$$n^2 = \frac{E_a}{E_s(1 + \nu_a) ln\left(\frac{R}{r}\right)} \tag{24}$$

L_e is embedded length, E tensile modulus, ν Poisson’s ratio, x distance along shim, w the shim width and h the shim thickness. The subscripts s and a represent shim and adhesive, respectively. R and r are equivalent radius values for the adhesive block and shim, respectively. These equivalent radius values were calculated from the equivalent circumferences of the cross-section surfaces.

Pull-out test specimens were prepared in a mould (Figure 36a) where rectangular blocks to the dimensions indicated in Figure 36b could be cast. The channel in the mould keeps the adherend shim perpendicular to the top face of the adhesive block. Sliding the shim to different positions in the channel controls the embedded length. The clamps lock the shim in place once the embedded length has been set. Release film prevents the adhesive bonding to the mould. The sample is held within a clamping jig and the shim pulled from the block in a tensile test machine, Figure 36c. The force-extension curve is recorded, and the point of failure inferred from the point where the load drops, Figure 37.



(a) Mould

(b) Specimen dimensions

(c) Test arrangement

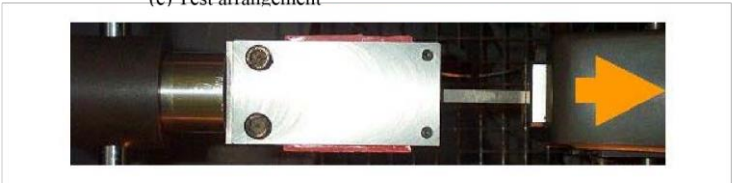


Figure 36. Pull-out specimens

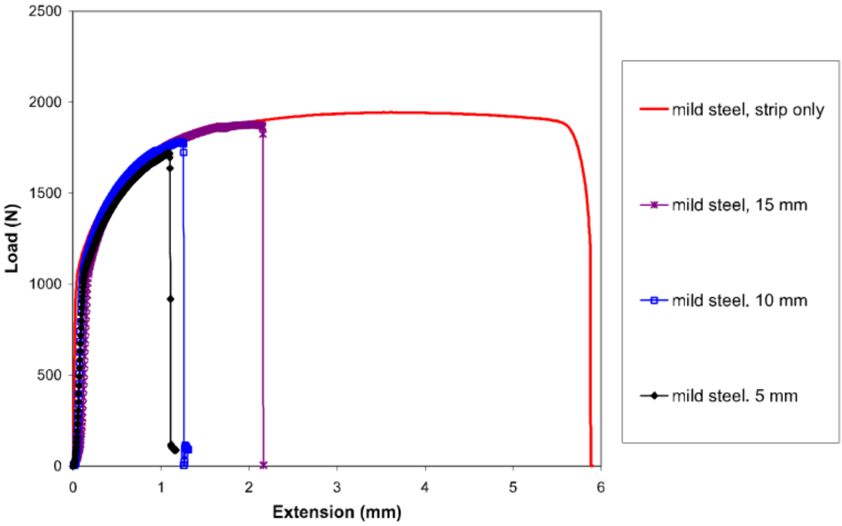


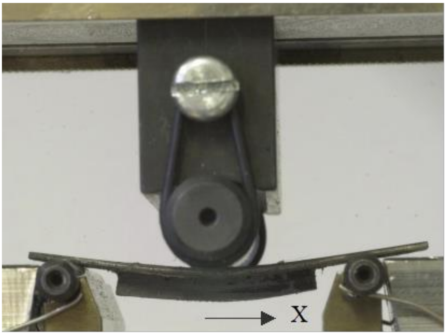
Figure 37. Typical pull-out test force-extension curves

Tests were conducted on a variety of shims and embedded lengths, supported by FEA [120].

- Tensile stresses in the shim sample are large; most of the response in the measured force extension curve shown in Figure 36 is due to plastic yielding of the adherend. In many cases, particularly with longer embedded lengths, the shim failed before the bond.
- The analytical expression is based on a linear elastic analysis and becomes invalid when the shim starts yielding. Hence, the simple closed form analysis of the test results is inaccurate and more complex analysis is required.
    - Failure loads are not directly proportional to embedded length.
    - IFSS values at failure (calculated from failure loads and Equations 23 and 24) are not consistent for different embedded lengths.
    - The IFSS values predicted from Equations 23 and 24 are far lower than those predicted by FEA.
    - FEA suggests similar peak stress levels at failure in each case.
- In some samples the sample failed by cohesive failure of the adhesive block and not failure at the interface.
- Repeatability in the load at failure was poor.
- Manufacturing adhesive blocks of the required thickness (5 mm) can be difficult owing to overheating during exothermic cure.
- The test is limited to adherends that can be prepared as thin samples (e.g., 1 mm thick strips.

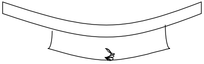
Therefore, for the many reasons outlined above, it is very difficult to recommend the pull-out test for determining interfacial adhesion strength.

Flexure test methods

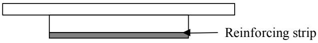


(a) Three-point bend test

(b) Unbacked specimen – interfacial (delamination) failure



(c) Unbacked specimen – cohesive failure



(d) Backed specimen

Figure 38. Three-point bend test, ISO 14679 [127]

When a laminate is flexed, stresses are imposed at the interfaces between different layers and delamination will occur if the laminate strength is exceeded. This principle can be used to assess adhesive joints and coatings. Bend tests are well-established techniques for assessing adhesion of coatings and paints to surfaces [56]. The four-point bend test is often used for thin coatings with the point of failure being detected by visual observation of cracks forming on the coating surface or by acoustic emission techniques. However, four-point bend tests may not produce failure with well-bonded, flexible coatings as peel stresses may not be sufficient to rupture the bond.

The three-point bend test, ISO 14679 [127] has been developed for adhesives. Figure 38a shows a three-point bend specimen and loading jig. A thick rib of adhesive is applied to the middle of the adherend. The adherends used are rectangular with a length of 50 mm and width of 10 mm. These adherends should be thin in comparison with the adhesive rib so that they bend easily and so that the rib of adhesive imparts a reasonable degree of stiffening to the structure. The rib of adhesive applied to the adherend is 25 mm long by 5 mm wide; the adhesive thickness should be approximately 4 mm and uniform, Figure 39.

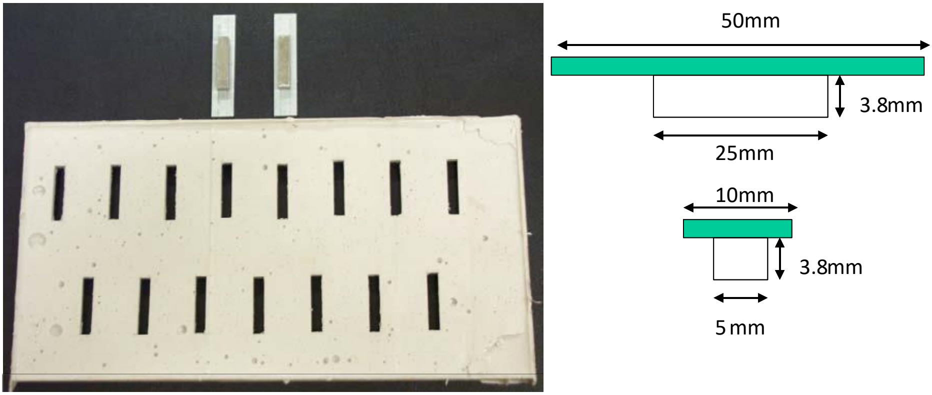


Figure 39. Example of a mould for preparing test specimens

The two lower rollers support the adherend on either side of the rib. The upper roller pushes on the top of the sample. As the sample is depressed, the load increases. The sample bends and stress are generated at the interface between the adhesive and adherend. Delamination should initiate at the end of the bond where the stress is a maximum. The delamination leads to a reduction in force, as shown in Figure 40. In weakly bonded systems or when very low modulus adhesives are tested the drops in load may be very small and difficult to identify.

The test sample should fail by delamination of the adhesive rib from the adherend, Figure 38b, and does so in most cases. However, in some cases, (e.g., flexible adhesives that are well bonded to the adherends) the forces at the interface are insufficient to delaminate and the samples eventually fail through cohesive rupture at the base of the rib (Figure 38c).

A modification to the test, bonding a stiffener strip to the outer edge of the adhesive rib, Figure 38d, can be made to increase the peel stresses at the end on the rib and thus bias failure towards the interface. In these modified samples, failure was observed to initiate at the adherend-rib interface as desired.

Consistent samples can be prepared by using shaped moulds. Moulds can be prepared by casting silicone rubber plaques around solid inserts corresponding to the required dimensions of the sample (plastic beams cut to the required rib dimensions can be glued to spare adherends to produce the inserts). The mould shown in Figure 39 produced samples with a 3.8 mm thick adhesive rib. When preparing the modified specimen thin 25 mm by 5 mm steel plates were inserted into the mould before the adhesive was dispensed. The thickness of the plates was 0.9 mm, and the resulting thickness of the adhesive 'sandwich' layer was 2.9 mm. Adhesive is dispensed into the mould before the 50 mm adherends are placed in position and pressed down using a flat plate. The samples are then cured according to normal cure schedules before they are released from the moulds. Excess adhesive is removed after the samples are cured. One drawback with this method is that the adhesive layer needed is relatively thick and it may be virtually impossible to make representative samples this thick with some types of adhesives, for example anaerobics. Furthermore, if the adhesive cures rapidly large temperature rises may occur in such thick adhesive samples, damaging the material.

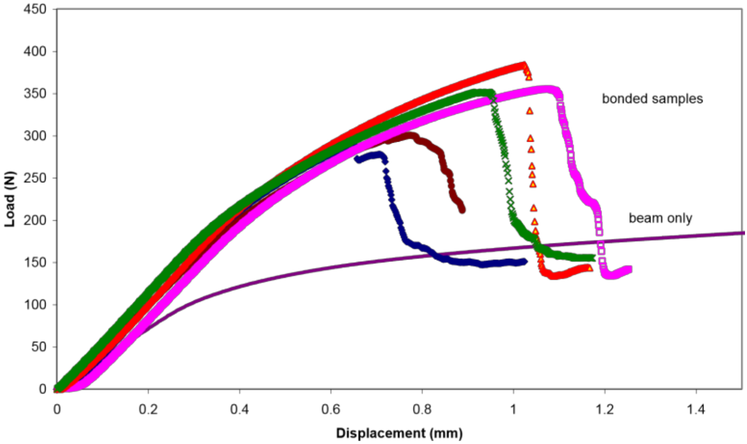


Figure 40. Measured force-extension response

Figure 41 shows typical test results from the 3-point bend test. The maximum force has been determined from the load at the drop in the force extension curve. Equation 25 was used to calculate interfacial shear strength from the maximum load. The results are an average of five specimens per surface treatment/condition; the error bars indicate the experimental scatter, which masks any differences between the treatments. The conclusion is that all the treatments perform similarly and resist degradation under the conditioning used.

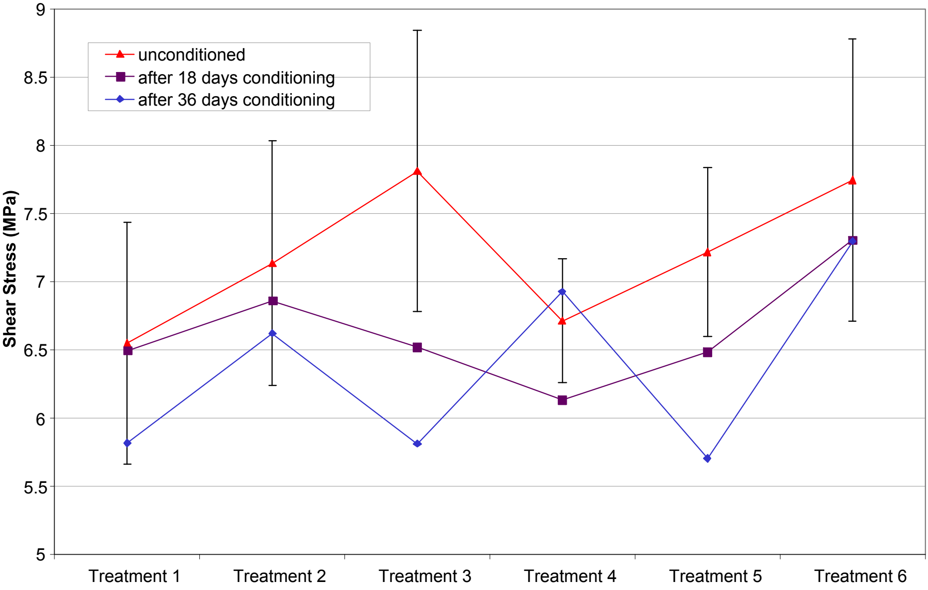


Figure 41. Three-point bend results for aluminium samples (see Table 3 for treatments)

The standard specimen, consisting of adherend and adhesive rib, can be modelled as a two-layer beam. Analytical equations [128] have been developed to calculate the shear stress τ at the interface. These are based on the bending moments and elastic properties of the beam and rib:

$$\tau = \frac{FE_2b(h-h_1)}{2b_1\left(G_2 - \frac{G_1^2}{G_0}\right)}\left[\frac{h+h_1}{2} - \frac{G_1}{G_0}\right] \tag{25}$$

where

$$G_0 = E_1b_1h_1 + E_2b(h-h_1)$$

$$G_1 = \frac{E_1b_1h_1^2}{2} + \frac{E_2b(h^2-h_1^2)}{2}$$

$$G_2 = \frac{E_1b_1h_1^3}{3} + \frac{E_2b(h^3-h_1^3)}{3}$$

The modified specimen, with metal stiffening strip added to the base of the adhesive rib, can be modelled as a 3-layer beam. The analytical calculation for shear stress (Equation 25), modified to account for the presence of the stiffener [120], is:

$$\tau = \frac{F E_2 b(h - h_1)}{2b_1\left(G_2 - \frac{G_1^2}{G_0}\right)}\left[\frac{h + h_1}{2} - \frac{G_1}{G_0}\right] \tag{26}$$

where

$$G_0 = E_0 b_1 h_0 + E_1 b(h_1 - h_0) + E_2 b(h - h_1)$$

$$G_1 = \frac{E_0 b_1 h_0^2}{2} + \frac{E_1 b_1 (h_1^2 - h_0^2)}{2} + \frac{E_2 b(h^2 - h_1^2)}{2}$$

$$G_2 = \frac{E_0 b_1 h_0^3}{3} + \frac{E_1 b_1 (h_1^3 - h_0^3)}{3} + \frac{E_2 b(h^3 - h_1^3)}{3}$$

F = applied force
b = width of the adherend
b_1 = width of the rib (and is equal to the width of the stiffener)
h = total depth of the adherend plus rib (or the + rib + stiffener)
h_0 = depth of the stiffener
h_1 = depth of the rib (or the rib + stiffener)
E_0 = modulus of stiffener
E_1= modulus of the adhesive h total depth of the adherend plus rib (or adherend + rib + stiffener)
E_2 = tensile modulus of the adherend

FE analyses were performed to determine the effects on the test data that would occur through varying various aspects of the test (e.g., rib thickness, fillet shape). These showed that the presence of a curved fillet was to be avoided, as this would move the area of peak stress in the sample from the interface (at the end of the bond-line) into the outer edge of the fillet to produce a cohesive failure. The peak stress in specimens with square ends (both the standard and the backed version) occurs at the end of the bond at the interface and specimens failed through delamination as required. Rib thickness affects the magnitude of stress at the interface for comparable deflections and the relationship is linear over the range of manufacturing variation in the rib thickness [120].

The interfacial shear stress predictions from the FEA results were compared with those experimental results in Figure 41, calculated from Equation 23. The analytical calculations predict significantly less strain the stress at the interface than the FEA calculations. The level of shear stress determined in Figure 41 (ranging from 6 MPa to 8 MPa) is much lower than obtained from lap shear measurements of adhesive on aluminium which are much closer to the FEA prediction of shear stress at failure > 25 MPa. The large underestimate of stress from the analytical calculations is partly due to them calculating the shear stress at the interface in the centre of the beam rather than at the ends of the bond-line. The analytical formulations are based on linear elastic assumptions that are unlikely to be valid at failure.

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Chapter 4

Interpretation of test results

- Mode of failure
- Influence of material properties
- Residual stress
- Detection of failure
- Curing adhesive
- Quality assurance

Mode of failure

The failure of an adhesive bond may occur due to:

- Interfacial failure between the adhesive and the adherend surface, called 'adhesive failure';
- Cohesive failure of the adhesive;
- Cohesive failure of the adherend;
- Failure of the surface layer of the adherend.

Inspection of the fracture surface can indicate which mode of failure predominates. Samples should be examined after testing to establish the mode of failure. The absence of adhesive on large regions of the surface indicates adhesive failure. The presence of adhesive on both the surfaces indicates cohesive failure. Both halves of the sample should be examined; the surface of the adhesive can indicate failure of the substrate through the presence of bonded material or help confirm adhesive/cohesive failure from the topography. It should be noted that in instances where the adhesive fails cohesively very close to the adherend surface then the thin adhering layer might not be readily visible without more detailed examination (e.g., by microscopy or spectroscopy).

The value of examining both failure surfaces is demonstrated in Figure 42, which shows fracture surfaces for a flexure test sample and a pull-off test sample. Both substrates are galvanised steel and have had the same surface preparation. Specimens were manufactured and cured on the same day bonded using the same epoxy adhesive. Each specimen was conditioned for 44 days in a hot/wet environment (70 °C/85% relative humidity). The specimens were tested on the same day.

The flexure test sample shows a region of adhesion failure (upper left end) where fracture initiates. The mode of failure then changes to cohesive failure very close to the interface. The location of the change from adhesive to cohesive failure is most obvious on the surface of the adhesive rib. In the centre of the sample is a region where the steel has corroded. There is rust on the corresponding face of the adhesive indicating a weak surface layer that failed rather than the adhesive. In contrast, the pull-off sample shows cohesive failure. There are regions on the substrate where there is adhesive that is obviously still adhering (black patches). The surface of the adhesive is rough in appearance, suggesting that there are few areas where interfacial failure has occurred.

Further information may be obtained by combining imaging and chemical analysis measurements. For example, SEM and EDX measurements made on the flexure sample are shown in Figures 43 and 44. The EDX element maps show regions of enhanced iron and depleted zinc corresponding to the observed locations of the corrosion. The carbon content is low except at the edge of the bonded region, indicating the absence of cohesive failure of the adhesive both in the corroded region and the area of the sample next to the corrosion.

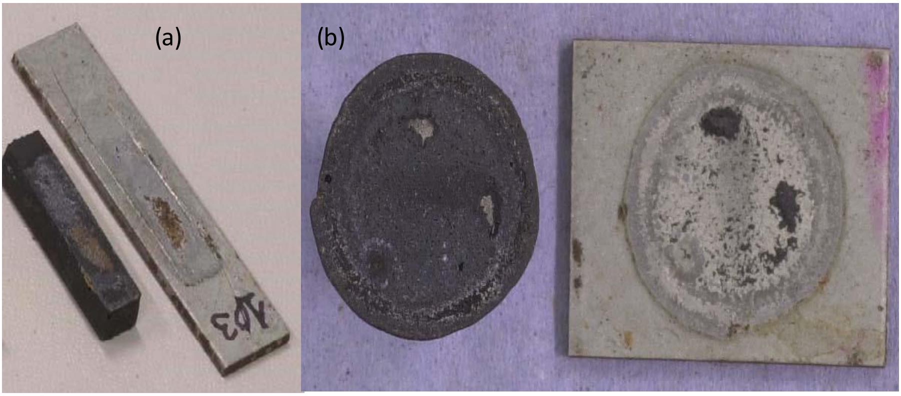

Figure 42. Fracture surfaces of galvanised steel/epoxy test samples (a) three point bend and (b) pull-off

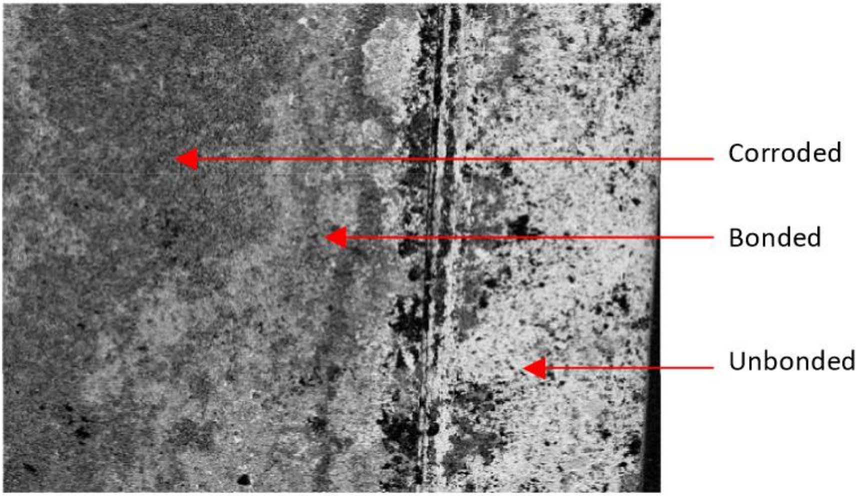

Figure 43. SEM image of sample from Figure 42

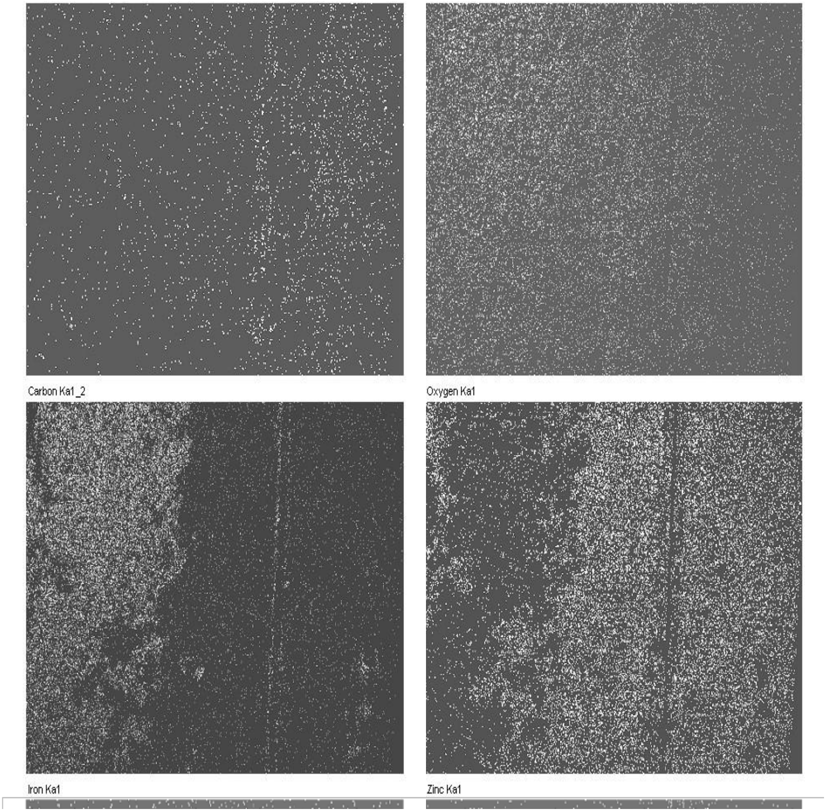

Figure 44. EDX element intensity maps of region shown in Figure 43

Influence of material properties

Plastic yielding

The results of various joint tests can be analysed, using functions derived from linear elastic mechanics, to determine local stress or strain values at the point of failure. However, in strong structural joints very often the plastic yield stress of either the adhesive or the adherend will be exceeded in localised regions before the joint fails. Adherend yielding is most likely to occur with joint tests using thin adherends (e.g., three-point bend, single lap shear or peel tests), thick sections are less likely to yield. When yielding occurs, strain at the location of yield will increase very rapidly compared to strain elsewhere until failure occurs. The linear elastic assumptions behind many of the equations used to analyse the test results will no longer be valid and the true stress or strain at failure will be underestimated as stress localisation at yield cannot be accounted for by linear models.

Degradation of material properties

- Static loads (creep);
- Intermittent loads (fatigue);
- Exposure to chemicals (including moisture);
- Thermal cycling.

The purpose of most adhesive joint testing programmes is to establish the service performance of the bond. Specimens can either be exposed to conditions until they fail (e.g., creep rupture tests) or tested after specific durations to establish the degradation in bond strength. Often conditions harsher than the service conditions (e.g., higher loads, temperatures or chemical concentrations) will be used to accelerate the ageing of the joint. Degradation may occur in the strength of the interface and/or the mechanical properties (modulus, yield stress, cohesive strength) of the adhesive and/or the adherend. Results of durability tests may need to separate these effects in order to provide reliable design data.

Exposure of adhesives to moisture is common. Adhesives will absorb moisture and the absorbed moisture may affect the properties of the adhesive and the interface. Moisture will plasticise epoxy adhesives – reducing the modulus, yield stress and ultimate tensile strength as shown in Figure 45. A fully saturated adhesive joint will have a very muchreduced joint strength due to the reduction in the cohesive strength of the adhesive even if the interface strength is unaffected.

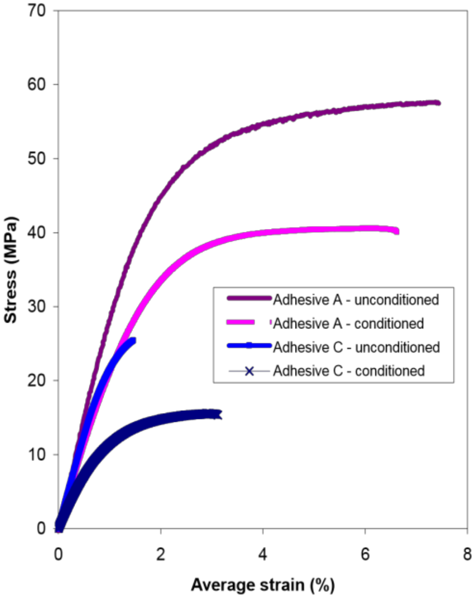

Figure 45. Effect of conditioning on the mechanical properties of the adhesive

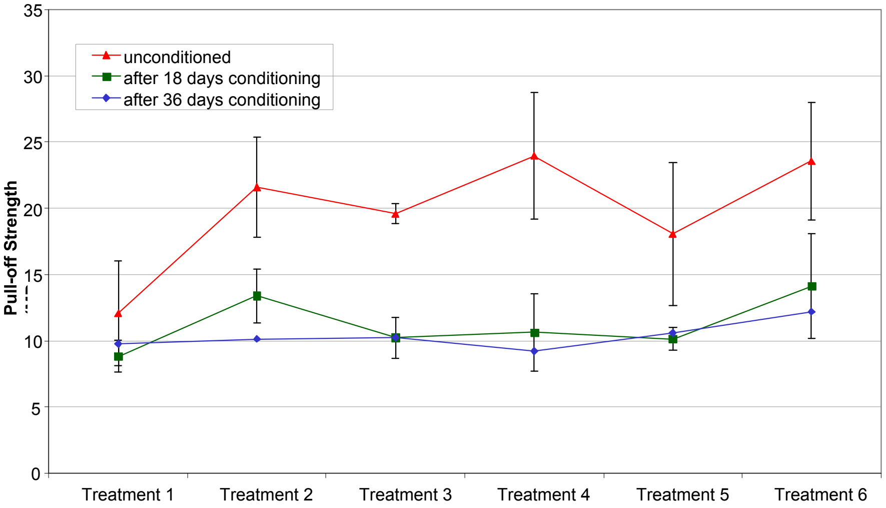

Figure 46. Pull-off test results for aluminium/adhesive A samples (see Table 3 for treatments)

As an example of the effect of degraded material properties on bond strength results, Figure 46 shows pull-off test results for aluminium test samples corresponding to the same surface treatments/conditioning combinations as the results from the three-point bend test presented in Figure 41. The adhesive in each case was adhesive A in Figure 44. In Figure 41, there was little difference between the results of the test combinations. However, the results in Figure 46 show:

- The unconditioned, untreated sample (Teatment 1) has significantly lower bond strength than the unconditioned treated samples (Treatments 2-6), which are comparable. Treatment 1 fails adhesively and the others cohesively (Table 6);
- The strength of the untreated sample is unaffected by conditioning; and
- Conditioning reduces the strengths of Treatments 2-6 until they are comparable with the untreated samples.

Normally, it would be expected that the treated samples would be more environmentally resistant than the untreated samples. FE analyses were made of the conditioned and unconditioned systems (using the appropriate materials properties shown Figure 45). These showed that, at the typical failure loads for the unconditioned (22 MPa) and conditioned (12 MPa) samples, the peak stress values in the adhesive layer were 66 MPa and 41 MPa, respectively – comparable to the tensile strength of the adhesive and suggesting that the loads measured are at the limit of the cohesive strength of the adhesive.

Residual stress

Adhesive joint tests assume that all stress in the adhesive layer is generated by the application of load through the adherends. However, residual stress in the adhesive layer may already be present following manufacture of the specimens. Residual stress may be generated in an adhesive layer through:

- Shrinkage of adhesive during cure; or
- Differences in the residual thermal expansion of the adhesive and adherends generating strain when the joint is cooled from the cure (or post-cure) temperature.

The extent of shrinkage may be determined through dilatometry or internal strain measurements of the adhesive during cure and cooling. These measurements will provide data on both the cure shrinkage and the coefficient of thermal expansion (CTE). Shrinkage whilst the adhesive is still a viscous liquid/paste will have little effect on internal stress generation and, therefore, the technique for monitoring cure shrinkage only needs to be capable of measuring the behaviour following the gel point of the adhesive, where it changes state from viscous liquid to visco-elastic solid.

Optical Fibre Bragg Grating (FBG) strain sensors, Figure 47, can be embedded within adhesives during cure. The strain transfer between the adhesive and the optical fibre alters the spacing of the diffraction grating printed on the fibre. The change in grating spacing changes the wavelength of light reflected through constructive interference. The shift in wavelength is proportional to strain. The strain signal needs to be corrected to account for thermal expansion of the fibre and, therefore, simultaneous temperature measurement is needed.

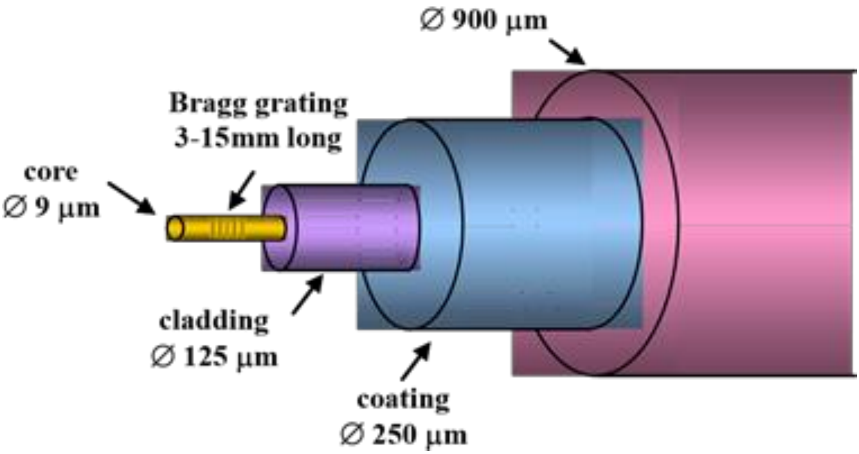

Figure 47. Fibre Bragg grating

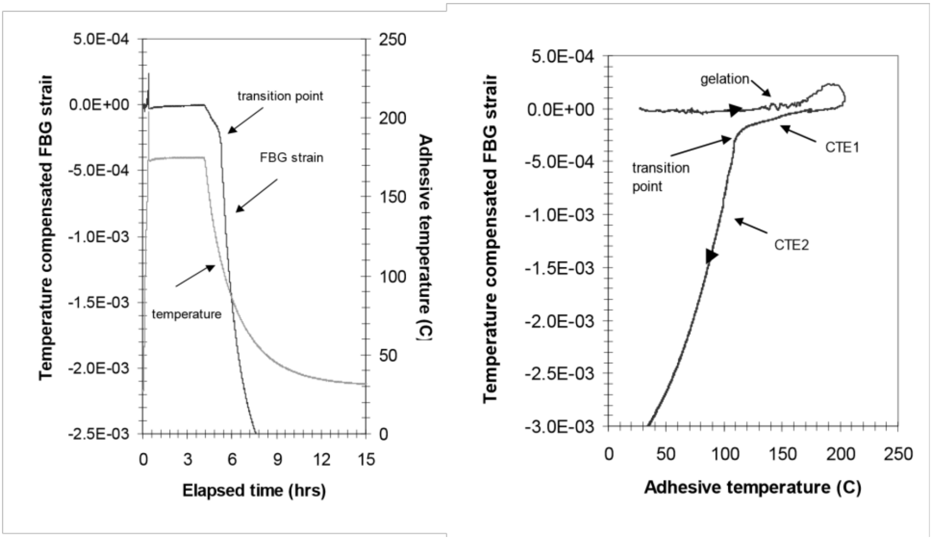

Figure 48. FBG strain measurements of an epoxy adhesive during cure (a) plotted against time and (b) plotted against temperature

The cured sample was subsequently cycled between room temperature and the cure temperature. On the first cycle a significant reduction was observed in the strain on heating beyond the glass transition temperature. The cooling cycle and subsequent heating/cooling cycle did not exhibit this feature, which was attributed to the relief of internal stress by annealing.

Figure 48 shows FBG measurements on a one-part epoxy adhesive cured at high temperature (180 °C). The temperature profile shows the adhesive heating towards the cure temperature, accelerating as the exothermic cure reaction occurs, settling to the stable cure temperature and then falling as the sample is cooled. The temperature compensated strain is virtually constant, except around the exotherm peak where the temperature compensation may be lagging the actual fibre temperature and then decreases as the adhesive shrinks during cooling. The 'knee' in the strain curve occurs at a temperature of ca. 110 °C, consistent with the glass transition temperature determined using dynamic mechanical thermal analysis (DMTA). The lack of any change in strain during the curing period indicates a very low cure shrinkage, which is expected for a filled thermoplastic. The CTE of the adhesive is higher than most metals and, therefore, on cooling the adherends will shrink less than the adhesive. This will lead to tensile stress in the adhesive, as the adherends will generally be stiffer than the adhesive and constrain it from full shrinkage.

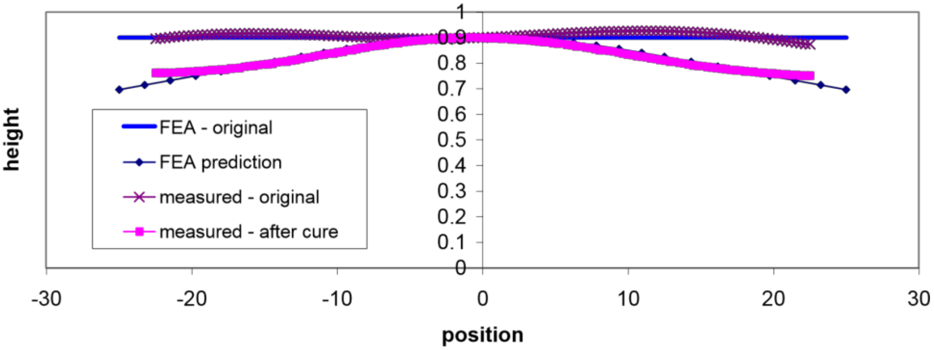

Figure 49. Residual stress induced deformation of a thin three-point bend adherend

Figure 49 illustrates the effect that residual stress can have on test samples. The profile of an adherend for the three-point bend test was measured before and after the adhesive was cured. Additionally, an FE analysis was run to predict the effect of thermal shrinkage on the sample deflection and the internal stress. The deflection of the thin adherend is measurable and agrees well with the FE prediction. The stress analyses predict that the peak residual stress in the sample is approximately 20 MPa, which is a significant proportion of the 60 MPa tensile strength of the adhesive. The residual stress is greatest in the centre of this sample and, hence, does not contribute significantly to the region of maximum stress at the end of the bond where failure initiates.

It is not only residual stress in the adhesive that may affect the performance of the adhesive joint. Stress may be induced at the surface of the adherend, which could damage any surface layer or coating and weaken the bond. For example, the large residual stress transferred to the surface of the sample may explain the location of the corrosion observed in Figure 42a. The tensile stress may have ‘opened’ the zinc surface, creating higher porosity where water could reach the steel, and damaged the protective capability of the zinc layer.

Detection of failure

An important output of an investigation into the adhesion strength of an adhesive joint is the identification of the point where failure initiates. Work on testing adhesive joints shows that, in many cases, joint failure initiates before the ultimate extension or load of the joint is reached [89, 99]. Designing for a long-term service life requires that joints never be loaded to the extent that these failures can initiate (whether through cohesive rupture or adhesive failure). Therefore, identification of the failure initiation point is a critical part of any joint test. By knowing this point, analyses can be performed to calculate stress and strain distributions immediately prior to the initiation of the crack. At the more easily identified point of maximum load in the test, a fracture will be present in the bond that will introduce inaccuracies in any analyses.

Shape of the force-extension curve

The formation of a crack will result in a redistribution of load and, in principle, failure could be detected from a change in the shape of the measured force-extension curve at the point of crack initiation. The slope of the loading curve should fall as a crack forms because the load bearing area of the bond is reduced. In many tests, such as the three-point bend, the initiation of fracture seems to be readily apparent (e.g., Figure 39). However, in other tests the joint stiffness reduces as load is applied, for example as a toughened adhesive yields plastically. In these situations, particularly if crack growth is slow, it may be very difficult to distinguish the effects due to crack formation from those due to yield. Finite element predictions may be used to establish the load at which the measured force-extension response of a joint deviates from the predicted behaviour. However, for these analyses to have any reliability, the models of the material properties and boundary conditions need to be extremely accurate.

Visual inspection

It should to be possible to detect a crack forming and growing by viewing the bond. Obviously this method is restricted to the edge of the bond and cracks forming in the interior of the bond-line will not be visible until the crack has grown to the edge of the joint. High magnification of the region of the joint where failure is expected would be required to view the beginning of a crack. Photography or video recordings could be used to record the formation and growth of the crack [89, 99]. These would need to be referenced to the force-extension or stress-strain curves for identification of the conditions generating the fracture. Synchronisation of the time index of the test data and the photographs is one mechanism through which the data could be analysed.

There are can be difficulties with visual detection of cracks particularly where heterogeneity and surface structures at the joint edge can mask the presence of a crack until it has grown somewhat. The edges of the adhesive bond could be polished to reduce these effects and a penetrant dye used to improve the contrast between the crack and the surface. Tests need to be performed at slow strain rates in order to capture the crack growth meaningfully, unless high-speed photography facilities are available. However, a fast propagating crack is likely to be associated with sudden, catastrophic failure of the joint – the situation where the maximum load is most likely to correspond to the load for the initiation of failure.

Acoustic emission of fracture energy

The fracture of an elastic material or bond is accompanied by a release of energy. A portion of this energy will be sound energy. Thus, fracture can be heard, a loud crack occurs as a brittle adhesive bond fails due to the large amount of acoustic energy emitted by the fast propagating crack. The sound intensity emitted by the initiation of a slow propagating crack in a tough adhesive is low and may be difficult to detect aurally above any background noise. Sensitive acoustic transducers placed near the bond line should detect acoustic emission from a crack forming. Analysis of the acoustic signal may yield additional information on the crack location and growth rate. Acoustic emission is a technique being applied to many systems (e.g., composites, coatings) as a research and structural monitoring tool. The technique relies on the operator having sufficient experience to be able to identify particular defect types from the AE data. The technique is essentially a laboratory tool, however, AE is used for proof and qualification tests of pressure vessels prior to installation and routine inservice inspection.

Strain mapping techniques

The initiation of a crack will alter the distribution of strain in its immediate vicinity. Local surface strain measurements should give an indication of the initiation of a fracture, even if the fracture originates within the bulk of the adhesive.

Strain mapping can be carried out using a variety of techniques. Modern fast computing technology allows for strain mapping through techniques involving image correlation of surface features, such as speckle interference, fringe patterns bonded to the surface (e.g., Moiré [129]) or natural patterns on the surface of interest. The absolute accuracy of strain maps in small regions of critical stress may be questionable but the initiation of a crack should have sufficient effects on the strain map to be readily apparent.

Internal strains can be characterised through Raman microscopy provided that the materials are transparent to the wavelengths used. Stretching a molecular bond alters its characteristic vibrational properties. With a suitable calibration curve, Raman spectroscopy can be used to determine strains in local regions [126]. X-ray diffraction can characterise strain in metal adherends close to the adhesive interface. These strains can be used to calculate stress in the adherend close the interface and, by inference, provide information on the interface [130]. Photoelasticity measurements can also be applied where one of the adherends is transparent [131].

The formation of a crack at the adhesive-adherend interface will relieve stress locally and thus reduce the strain in the adherend. These changes in strain can be detected using the strain mapping methods outlined above. However, where the adherends are rigid, strain gauges can be attached to the adherends to monitor strain. This technique is known as 'back face strain gauging' and can be a cost effective monitoring method. Strain gauges will also enable the identification of the point of plastic yielding of the adherends, which is a likely precursor to failure of the joint.

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Chapter 5

Concluding remarks

Potential measurement methods for characterising and quantifying the interfacial adhesion strength properties that are key to the strength of materials systems have been discussed in this review. There is, at present, no consensus as to best practice in considering the interface in a design or quantifying the adhesion strength in a structure. Industry tends to assume a perfect interface when designing adhesive bonds. This is due to both the complexity of models needed to include the interface and the lack of reliable data.

Tests for adhesive joints, fracture toughness and engineering coatings that can provide information on the interface strength have been described. Although there is a plethora of methods available, few of them are capable of providing good quality quantitative data that are readily usable in design calculations. Analysis of test results presents problems as there are difficulties in defining the state of stress, strain or fracture energy close to the interface due to the complex stress distributions that are difficult to model accurately (particularly when material responses are non-linear) and the possibility of material properties close to the interface being different to those of the bulk material. In many situations it may be sufficient to perform qualitative ranking studies to determine the most effective adhesive/adherend/surface treatment system within a specified set of loading and service condition variables.

There are few joint tests that are applicable to both thick and thin adherends. Obviously, thick adherends can be machined to the dimensions required for tests but this increases the cost of testing. The results from lap joint tests are difficult to interpret owing to the complex distribution of peel and shear stresses near the end of the overlap. Joints with higher stiffness (due to thicker adherends) such as the thick adherend shear, double lap or strap joint ought to give more reliable results. The tapered strap joint offers the possibility of inducing failure at either the thick, central adherend or the thin straps depending on the joint geometries chosen. This could enable the evaluation of both thin and thick section bonds using the same test configuration.

Peel tests can provide information on bond strengths but these are difficult to relate to a general design situation as converting load per unit width to stress requires making assumptions about the size of the peeling zone. Often fracture in peel tests occurs in the adhesive layer rather than at the interface. Fracture toughness tests also offer the capability of characterising the interface. The wedge test is simple and cheap to perform but, whilst well suited for comparing the durability performance of different surface treatments, does not provide accurate data on surface fracture energies. The double-cantilevered beam tests (DCB and TDCB) provide more accurate data. A possible limitation is that fracture may run into the adhesive rather than along the interface.

The pull-off and three-point bend tests are simple tests, suitable for screening purposes, which appear to maximise stresses at the interface between the adhesive and substrate. These stress distributions are complex and although analysis techniques have been developed to interpret the results the accuracy of these is open to question. The butt tension test can provide good quality quantitative data but great care needs to be taken to produce reliable results.

The conditions causing initiation of failure are critical in the interpretation of test results and for the safe design of bonded structures. Techniques for detecting the start of a fracture need to be utilised when testing the material systems in order to maximise the usefulness of the data obtained. Inspection of the fracture surfaces to determine the mode of failure, even if only by visual inspection, needs to be done to aid the understanding of results. The cohesive strength of the adhesive and adherends needs to be considered in the interpretation of test results. It is possible that what appears to be a degradation of interface performance on conditioning may be a consequence of reduction in the mechanical properties of the materials involved.

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Appendix 1

Adhesion

Mechanisms of adhesion

The term adhesive is used to describe a polymeric material that bonds two surfaces or components together to form a joint, particularly one that will bear load. A true adhesive bond to a surface is formed through inter-molecular attraction between molecules in intimate contact. Bonds form due to (in order of increasing bond strength):

- Van der Waals attractive forces
- Polar interactions and hydrogen bonding
- Covalent, chemical bonding

The primary mechanisms involved in the development of an efficient bond are:

Mechanical adhesion

Mechanical adhesion is related to the degree of roughness and as a consequence friction of the adherend surface. A certain amount of bonding can be expected purely from the mechanical interlocking of the two contacting surfaces, increasing the total surface area available for chemical bonding and creating a convoluted failure path where the adhesive penetrates crevices on the adherend surface. Although the tensile strength of the bond can depend on the re-entrant angles on the adherend surface, shear strength increases significantly with increased roughness.

Wetting

Wetting out refers to the ability of an adhesive to flow and cover a surface to maximise the contact area and the attractive forces between the adhesive and bonding surface. If sufficiently intimate contact is achieved between the adherend and adhesive a physical interaction develops between the atoms of the two surfaces, which results in wetting. Formation of a physical bond results from highly localised intermolecular forces. Adhesives that have surface energies less than that of the adherend will readily wet the surface and yield good bonds. Wetting may be due to acid-base interactions, weak hydrogen bonding or Van der Waals forces (dipole-dipole and dispersion forces). The extent of wetting depends on the differences in surface free energies of the solid, liquid and subsequent interface.

Chemical bonding

Chemical bonding is the formation of a stable linkage by the reaction between a functional grouping on the adherend surface and a compatible group in the adhesive. Adherend surfaces are usually given surface treatments (e.g., chemical etching, corona discharge and plasma treatments) to create compatible groups. These treatments serve to increase the concentration of oxygen and nitrogen containing functional groups on the adherend surface since these species are considered to enhance adhesion by encouraging the formation of strong covalent or hydrogen bonds.

In thermodynamic terms, to break an adhesive bond energy must be applied to the system such that the input energy (ΔE) is greater than the free energy of the two new surfaces created less the interfacial energy.

$$\Delta E \geq W_A + W_S - W_{AS}$$

where W_A is the surface energy of the adhesive, W_S is the surface energy of the substrate, W_{AS} is the interaction energy.

In theory, all bond strengths could be calculated from these surface energy properties. Wettability (and contact angle) measurement techniques enable determination of surface energies resolved into dispersive and polar terms [132-135] and with the correct choice of probe liquid acidic and basic properties of the surfaces can be quantified. Unfortunately, the surface energy approach has only limited application to predicting the strength of a joint since, as mentioned above, true interfacial failure is unlikely. Further complications arise since the contact area, due to roughness at a molecular scale, is difficult to determine although adsorption techniques have been used [136]. Furthermore, the surface energy approach ignores any direct chemical interaction between the adhesive and adherend and also fails to consider any changes in the composition of the surface layers due to the presence of the second layer. Wettability is therefore a useful method for assessing suitability to bond but would be difficult to relate directly to bond strength. Phenomena such as mechanical keying often provide enhanced bond strengths to the physico-chemical bonds.

An alternative technique for characterising energies of interaction between surfaces is the determination of detachment forces and contact radius using sensitive surface force apparatus [137]. Highly sensitive force measurement systems enable the measurement of extremely small adhesion forces. The measurements require that the surfaces are molecularly smooth. This is generally achieved through the use of cleaved mica sheet substrates. Monolayers of polymer or surfactant can be deposited on the mica surface and interactions measured directly. This type of measurement can yield accurate adhesion strengths but is limited in the types of materials that can be studied through the requirement of molecularly smooth surfaces.

Workers in the adhesives field, such as Sharpe [138] and Bikerman [132], have argued that the statistical improbability of a fracture propagating solely along a molecularly rough surface means that true interfacial failure never occurs. Thus, the phenomenon of ‘interfacial failure’ is more accurately described as near surface failure. However, other authors [133] have argued that failure at the interface can be thermodynamically favoured and that the surface energies can be correlated with bond strength with polar components of the surface energies playing a critical role. Whatever the terminology employed, understanding the behaviour of interfaces throws up many challenges.

Stresses in the region of the interface are difficult to determine accurately owing to the difficulty in modelling load transfer between dissimilar materials. The properties of surfaces may differ both chemically and physically from those of the bulk material (e.g., oxide layers on metals). Furthermore, there is some evidence that the proximity of another surface may further modify the properties of interface layers of the adhesive through mechanisms such as molecular re-orientation, preferential diffusion/adsorption of components or altered chemical reactions (e.g., through catalysis or differences in the thermal history during cure). The concept of an interphase region has been used to describe material characteristics near an interface [139, 140]. The properties of the adhesive in this region can be significantly different to the bulk adhesive [139-142]. The differences between bulk and interface properties depend on the adhesive, surface material and surface preparation. Substantially higher modulus values have been reported for the near surface material, attributed to the transfer of materials from the surface oxide layers into the adhesive [139] as have decreased modulus values near the interface [140-142], thought to be due to incomplete cure and residual stresses in the near interface region. The extents of the interfacial zones in the adhesive layer have been estimated as relatively thick (greater than 0.1 mm) in comparison to bond thickness [132, 139-142]. The keying of adhesive into surface roughness features can also be considered as forming an interphase region whose properties affect the mechanical performance of the joint [143]. In this Guide the term ‘interface’ is used to encompass the true interface, the interphase and the near surface area.

Appendix 2

Joint preparation and testing

Introduction

This Appendix summarises good practice in adhesive joint preparation and testing which was originally presented in Good Practice Guide No. 47 [3].

Handling and storage of adhesives and adherends

A number of issues relating to the handling and storage of adhesives and adherends need to be considered as many of these factors can affect the performance of the material. Issues to be considered include:

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

Handling materials (particularly 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 of adhesives

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 humidities above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [8]. 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 in regard to storage 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 following points should be considered for the storage (and transport of adhesives):

- 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.
- Repeated exposure of the adhesive is a frequent cause of adhesive ageing and deterioration.
- 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). Containers that have been opened must be sealed immediately after use.

- Adhesives should not be stored below freezing unless recommended by the manufacturer. In some systems components of the adhesive may phase separate at sub-zero temperatures, degrading the adhesive performance.
- Exposure to repeated freeze/thaw cycling could limit the shelf life of an adhesive stored at low temperature.
- 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.
- A refrigerated adhesive may be used several times during the shelf life of the product. It is advisable to take samples of the product under the storage conditions. However, to handle the adhesive, this may not always be possible. When the adhesive is taken from a sub-ambient (often sub-zero) temperature and allowed to warm, the amount of time at ambient for these adhesives should be kept to a minimum (advice on the length of time can be obtained from the adhesive manufacturer). 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.
- 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.

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. Temperature should be monitored to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. 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.

Handling and storage of adherends

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 humidities above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [8]. The overall effect is to reduce the adhesive performance and induce premature failure of adhesively bonded joints.

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.

Preparation of adherends

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 section 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. Adherend surfaces, unless specified otherwise, should be prepared according to ISO 4588 [144] for metals and ISO 2818 [145] for polymer matrix composites (PMCs). Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e. adherend bending)

- 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.

Many substrates (e.g., fibre-reinforced polymer composites) or surfaces (e.g., porous anodised films) will absorb moisture from the atmosphere. The presence of moisture can interfere with bond formation and may adversely affect the properties of the adhesive during the cure process. As a result, the joint strength may be compromised. Moisture released from the substrates 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 G_c of a rubber toughened epoxy adhesive by a factor of 10 [146]. 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.

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. There are many procedures available – further information can be found in “Guide to The Structural Use of Adhesives” produced by The Institution of Structural Engineers [13] and specific treatments are defined in various standards, e.g., BS 7079 [14], BS EN 12768 [15], ASTM D 2651 [16], ASTM D 2093 [17] and BS EN 1840 [18] – see also [19-22]. 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.

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 fixtures

A bonding fixture is recommended to ensure correct bond length (e.g., 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 Figure A2.1 [147]. This 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 six 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.

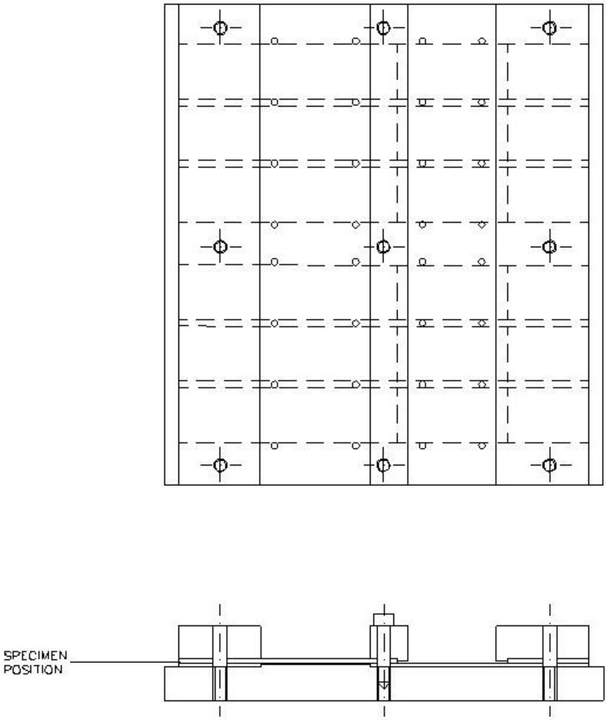

Figure A2.1. Single-lap joint bonding fixture (accommodates six specimens)

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. Care must be taken to avoid contaminating bonding surfaces with mould release agent. Where possible solid films should be used to eliminate the risk of contamination.

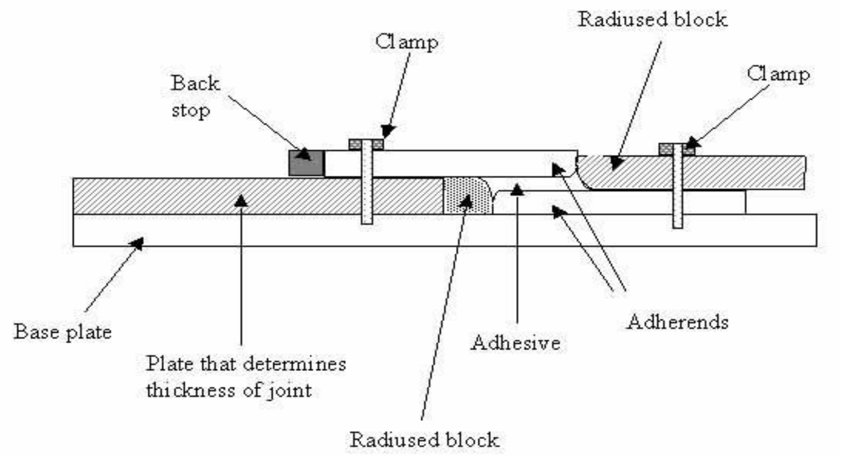

Figure A2.2. Fixture for controlling single-lap joint adhesive fillet

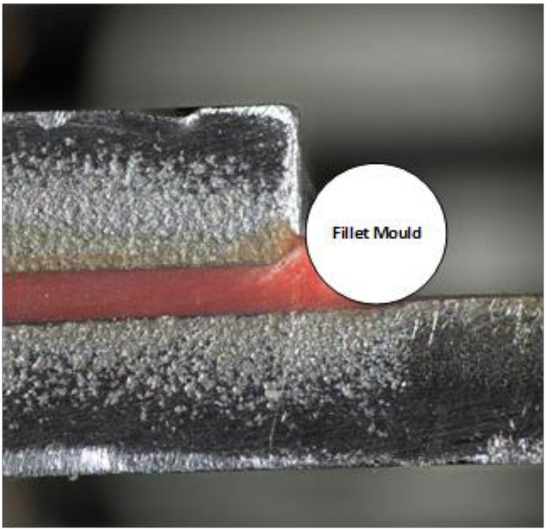

Figure A2.3. Single-lap joint with a concave fillet

Adhesive fillets

Adhesive fillets can have a significant effect on the performance of an adhesive joint. The size and shape of these fillets should be controlled carefully to ensure repeatable results from tests. Fillets tend to reduce stress concentrations formed at the bond-line ends and therefore increase the “apparent” shear strength of joints, compared with square-ended bond-lines. 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.

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 A2.2 or a special tool shaped to fit within the bonded joint; as shown in Figure A2.3. The tool can be held in place using heat resistant tape. Figure A2.4 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., film adhesives). The high viscosity associated with highly filled 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 2 mm, 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. These additional features will add considerably to the costs of specimen manufacture.

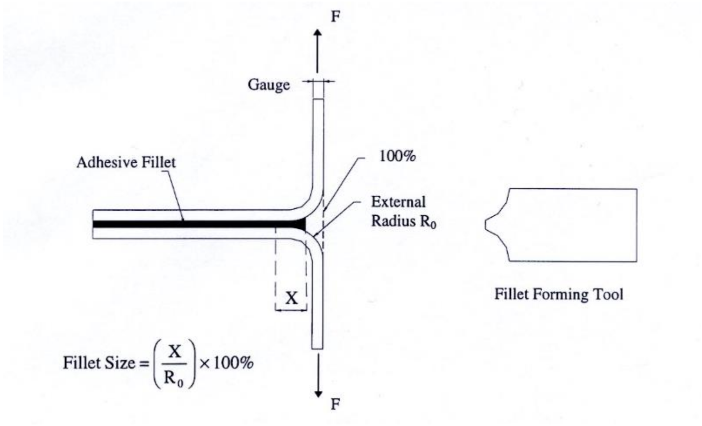

Figure A2.4. Tool for controlling fillet size in T-peel specimens

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),
- Thin wire spacers (e.g., stainless steel) inserted between the adherends 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), or
- Ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1 wt. % by mass). 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.
- Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness.

Controlling the bond-line thickness of very viscous adhesives is difficult due to the lack of flow. The preferred method is to use thin wire spacers. However, this method of bond-line control is not always reliable.

Dispensing of adhesives

The adhesive should be carefully dispensed to ensure that the correct volume is used to avoid underfilling the joint (leaving gaps that weaken the joint) or overfilling the joint (and having to remove the excess).

Multi-part adhesives should be thoroughly mixed in the correct ratio before being applied to the adhesive joint. Often material is supplied in twin pack cartridges and dispensed through mixing nozzles, which provides good control over mixing. However, if the parts require mixing by hand, then the two parts should be weighed out before mixing to ensure control of the mix ratio. The two parts of the adhesive are normally different colours and complete mixing is usually indicated by a uniform colour with no streaks. The period of mixing should be kept within the working life of the material to ensure that the adhesive does not start to cure and therefore flows correctly when it is applied and when the bond is closed. To ensure consistency when making batches of samples, it is important to ensure that the samples are made with as little difference as possible in the times between mixing and making the first and last joints. The whole operation should be completed well within the working life of the adhesive.

The procedure for manufacturing the joint should minimise any inclusion of air in the adhesive that could lead to voids in the joint. It is best to apply the adhesive, whether through a nozzle or by spatula, in a single pass to avoid folding the adhesive over on itself.

It may be necessary to remove excess air from the adhesive prior to manufacturing specimens. This can be done by vacuum out gassing if there are no volatile components in the adhesive system. The pressure should be reduced slowly to avoid ‘foaming’ of the adhesive. Stirring while under vacuum improves the effectiveness of air removal. Adhesive can also be centrifuged to remove air, but this runs the risk of components in the adhesive with different densities, such as fillers, becoming separated by the process.

Curing of adhesives

There are a number of key points that should be considered when curing adhesive joint specimens [1, 3, 5]:

- 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 test coupon should be similar to that in the final bonded structure. The same is also true of bulk adhesive specimens used to generate mechanical property data [1]. 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 [5].
- 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, different types of joint specimens may heat at different rates and therefore the final temperatures of the adhesive joint at the end of the cure period can be significantly different.
- 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. This is a particular problem with thick bond-lines.
- 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 previously, handling adhesives can be hazardous to human health, thus 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 [148]. 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 (see also NPL Measurement Good Practice Guide No. 32 [149]).

Quality and assurance documentation

Quality assurance tests

After the adhesive is cured, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the bonded area. The trained eye can detect specimen misalignment (i.e., twist and non-parallel edges), unfilled areas and voids.

Joints containing adhesive depleted regions around the edges or ends of the bonded area should not be tested. Thick, thin, or uneven bond-line can also be detected visually or by using a micrometer or optical microscope.

Checks should also be made on the adhesive fillet to ensure that the fillet complies with test specification. It is also advisable to check that the adhesive is fully cured and has bonded to the surface of the adherend. The texture and hardness can be a clue as to the effectiveness of the curing process. Several techniques are available for the non-destructive inspection of adhesive joints:

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

Documentation (record keeping)

It is recommended that an accurate record of the adhesive and adherends be maintained. It is good practice to perform routine checks each month to ensure that the stored materials are within the time limits recommended by the adhesive manufacturer. The storage record of the adhesives and adherends should include details on:

- The material source, manufacturer’s code number, batch or lot number,
- Form and packaging (i.e., for adhesives one-or two-part, or film, etc.),
- Shelf life and recommended storage conditions,
- Observations of the condition of the material on receipt (e.g., surface conditions of the adherends, including corrosion and surface defects such as scratches),
- Actual storage conditions (including temperature),
- Handling history during the shelf life (i.e., dates of use and length of time at room temperature if refrigerated),
- Manufacturer’s data sheets (material certification), and
- The records should also include test data from acceptance tests and routine quality checks carried out to assess material performance.

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.

The adhesive joint manufacture records should include details of:

- Date of manufacture;
- Person doing the manufacture;
- Adherends used;
- Adhesive used (including batch number and details of previous use);
- Surface preparation of the adherends and details of preconditioning; and
- Manufacturing details
    - Assembly method (jigging)
    - Fillet control
    - Bond-line thickness control measures
    - Curing conditions (i.e., temperature and humidity)
    - Manufacturing conditions
    - Post manufacture quality checks

Test records should include the information for the test specimen recorded previously as well as:

- Details of test machine and instrumentation employed (including calibration records)
- Test conditions
    - Load or strain rate
    - temperature

Mechanical testing of adhesive joints

This section considers the affect of test parameters (i.e., test machine alignment, load train stiffness, methods of gripping test specimens, 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. The guidance considers monotonic (static) loading but is relevant for other modes (i.e., cyclic fatigue and creep) and environmental conditions (i.e., elevated humidity and temperature).

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). 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.

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 [150, 151]. Alignment specimens can be in the form of a rectangular or circular bar (see Figure 51). 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 [150]. 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 51).

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

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 [1]. 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. 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.

An approximate measurement of strain and hence stiffness can be obtained from measuring the crosshead displacement of the test frame. The strain is the ratio of crosshead displacement and the bond-line thickness, and 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 to take into account the stiffness of the loading chain. 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.

Speed of testing

Polymeric adhesives are visco-elastic materials; that is their mechanical properties 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.

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Appendix 3

Finite element analysis

Introduction

Finite element analysis (FEA) is a useful tool for interpreting the mechanical behaviour of adhesive joints. Force-extension responses can be predicted, and maps of the stress distribution produced to identify critical regions where the local stress or strain may exceed a failure criterion for the material. The behaviour of bonded adhesives has been modelled through different approaches using finite element (FE) analysis. In many cases the adhesive and adherends are modelled using continuum elements, assuming the adhesive is perfectly bonded to the adherends [69]. For example, Cotton et al. [126] determined the interfacial shear strength of a system by assuming the two components were perfectly bonded and that the substrate maintained contact without any slippage. The assumption of a perfect bond means that the finite element analysis takes no account of the adhesion properties of the interface.

There does not appear to be an obvious way of predicting interface strength, as often the input data needed requires knowledge of the interface strength, or the failure stress/strain of the adhesive. But there are ways of accounting for adhesion in an FE analysis, by modelling the way in which the adhesive fails. This Appendix highlights some of the options available. There are two main approaches for modelling the failure of an adhesive interface. One approach is to model the growth of a crack (fracture mechanics energy-based approach). A second method is to model adhesive de-bonding (stress or strain-based approach).

Fracture approach

In the fracture mechanics approach, a sharp crack is assumed to exist within the material and failure occurs through propagation of this crack. In an adhesive joint, de-bonding may initiate in regions of stress concentration, then grow and cause joint failure. A de-bonded area can therefore be classed as a crack. This approach does not predict the crack initiation stress or energy. The general approach is to relate the crack growth rate (*da/dt*) through the joint to the applied strain energy release rate *G* or the stress intensity factor *K*. Within the literature, authors have considered measured and/or predicted values of *G* [152-154] and *K* [155, 156]. Calculation of the energy release rate *G* is based on linear elastic fracture mechanics (LEFM). This is valid when the plastic zone around the crack tip is very small. LEFM is not necessarily always applicable to adhesives. Toughened adhesives are relatively ductile and therefore do not always exhibit sharp crack behaviour. For non-linear situations, *G* is replaced by *J*, the non-linear energy release rate [152, 156]. *J* is applicable to any stress-strain relationship and is equal to *G* when the material is linear elastic. *J* can be written as a path-independent contour line integral giving rise to the name, the J-integral. The J-integral takes account of plastic zone size and is therefore more appropriate than *G* when the plastic zone size is large. The determination of *G* for linear elastic materials is relatively straightforward, while determination of the J-integral for non-linear materials is more complex. With FE methods, stresses, strains, and displacements along a path can be calculated using the J-integral [152, 156]. As the J-integral is path-independent it can be calculated away from the crack tip and is therefore not so affected by approximations in modelling stress and strain fields at the crack tip.

Simulations of crack propagation advance the crack front when the local energy release rate rises to a critical value. To apply such methods, one must introduce an initial flaw. In FE packages, such as ABAQUS [157], crack propagation can only be modelled along a predetermined surface. Potential crack surfaces must be modelled using contact surface definitions. Surfaces may be partially bonded initially and then may de-bond during crack propagation. The three de-bonding criteria are: crack opening displacement; critical stress criterion at a critical distance ahead of the crack tip; crack length as a function of time. After de-bonding, the interface behaviour reverts to standard contact, including any frictional effects.

Guild et al. [158] used the virtual crack growth method to model crack propagation. Cracks were introduced into the mesh by renumbering nodes of adjacent elements. Crack tip elements were not used, as point values of stress at the crack tip are not meaningful. The preferred direction of crack growth because Mode I opening could be deduced from examination of the vectors of maximum principal stress. The virtual crack growth method for assessing energy release rate is based on the concept that the energy released in growing the crack is equal to the energy which would be required to close the crack. Forces and displacements at the crack tip are found from FE simulations and calculations of energy can be made subsequently. The shape of the growing crack can be used as a further indication of the likely direction of further crack growth.

Interface elements can also be used to predict crack growth. In the work of Chen et al. [159] interface elements, defined via a user subroutine, were located along the de-bonding interface. This method is based on the indirect use of fracture mechanics in which a softened decohesion material model is provided with traction/relative displacement relationships that are constructed so that the enclosed area is equated to the critical fracture energy. Initial flaws are not required, with initiation being governed by a strength criterion. Two material properties were required for interface elements: G_c, the total energy from experiments and S_t, the assumed interfacial material strength. Both a linear and a cubic decohesion material model were used. In the linear model there is a triangular relationship between traction and the relative displacement (see Figure A3.1). The triangular area is the critical interlaminar fracture energy, G_c. The stress/strain relationship is defined by the interfacial tensile strength, σ_t, the cracking strain ε0, and the maximum strain, ε_c. In the FE simulation, the strain can exceed ε_c but the equivalent stress is zero and the interface is de-bonded. The elements where softening takes place are in the region $\varepsilon_0 < \varepsilon < \varepsilon_c$. The cubic relationship for the decohesion model is shown in Figure A3.1.

Cohesive zone models can be used to analyse the failure of an interface. These have been employed in the study of fracture in many ways (e.g., as phenomenological element breaking rules for fracture, and as models for the process zone ahead of a crack tip). Jagota et al. [160] describe cohesive zone models for polymer interfacial fracture, which have been implemented as cohesive elements for use with FE codes. The authors suggest that when incorporated between element edges (or faces in 3D) cohesive elements extend conventional FE in a way that allows independent specification of interfacial fracture and bulk constitutive behaviour. The cohesive elements describe the deformation and failure of the interface between two bulk finite elements by specifying the tractions that resist relative motion. Bennison et al. [161] used the cohesive zone model with FEA to analyse a compression shear strength test to determine adhesion. Cohesive elements were placed along the interface to capture intrinsic and near crack dissipation associated with interface de-bonding. The cohesive elements define the tractions acting across this interface in terms of opening and sliding displacements. The authors concluded that analysis of the compression shear strength test shows that cohesive elements can be used to treat adhesion/fracture problems where large inelastic deformations characterise the polymer response to loading.

Liechti and Wu [162] used the cohesive zone modelling approach to model rubber/metal debonding. The authors found that failing ligaments clearly provided some bridging between crack faces. The failing material in this study was represented by non-linear Kelvin units (non-linear springs and dashpots in parallel). The rate-dependent cohesive force was expressed as the sum of the non-dissipative force (springs) and the dissipative force (dashpot). At a particular ligament extension damage starts to occur at the interface and the stiffness of the interface decreases. At a critical extension, the ligament breaks, and the cohesive force vanishes. In the FE model part of the bond line was not connected to the substrate to account for the initial crack length and traction-free boundary conditions. The remainder of the bond line was replaced with an array of equally spaced non-linear Kelvin units to enable crack growth to occur along the interface. Calibration of the cohesive zone model was accomplished via numerical simulations of the observed de-bonding experiments. The values of the parameters of the cohesive zone model were chosen such that the solution for the crack extension history corresponded to the measured de-bonding history at one loading rate.

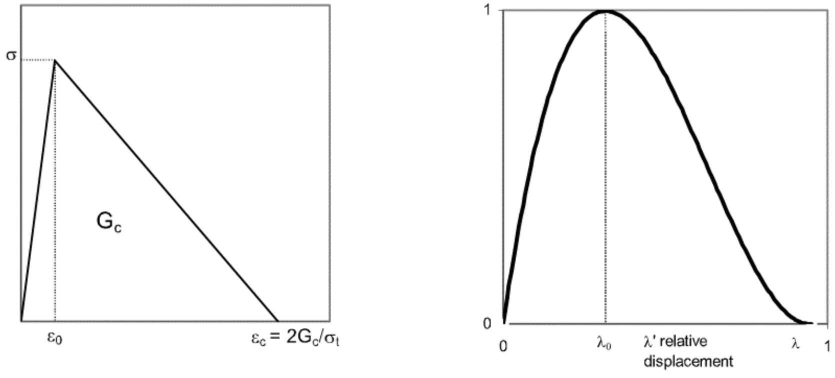

Figure A3.1: Decohesion models: linear model (left) and cubic model (right)

Coupling elements have also been used to study adhesion. Sebastian [163] modelled FRP plates de-bonding from concrete beams. A non-linear FE analysis used a layered bending membrane hybrid element for the beam and the plate, along with a coupling element for the adhesive connection. The coupling element was programmed to rupture and to disallow further interaction between the hybrid elements on either side once a predetermined stress level corresponding to the shear bond strength of the plate to concrete connection had been activated. The program can incorporate non-linear constitutive models for the materials present. The author suggests that the FE program can be improved: the analysis used was a stress-based failure criterion for connection failure, but the author suggests that a fracture mechanics-based criterion may be more appropriate.

An alternative de-bonding method can be employed in ABAQUS using the *de-bond option [157] to specify that de-bonding is to be considered. An amplitude-time function (see Figure A3.2) is used to give the relative magnitude of force to be transmitted between the surfaces at time $t_0 + t_i$ (t_0 being the time when de-bonding begins). When the fracture criterion is met at a node, the force at that node is ramped down according to the de-bonding data. The force at the node must have a value of 1.0 at zero time and must end with a magnitude of zero at the final time (i.e., node de-bonded).

Damage modelling is another method for modelling failure of an adhesive. Feih et al. [164] modelled a bending test. The adhesive was modelled with isotropic elasticity and von Mises plasticity. Failure of the adhesive was assumed to occur when the maximum tensile plastic strain was reached under tension. Cracking onset of the adhesive was modelled by reducing the transferable stress to 10%. This simplified damage method simulates the stress redistribution inside the adhesive and was employed as a user subroutine within an FE model. Stabilised integration point degradation was used to degrade the element properties at the integration point once failure was recorded at that point (one element might have different properties at its integration points over a certain period). In order to avoid numerical problems due to a singular stiffness matrix, full degradation is modelled using a very small degradation factor of 0.01 rather than zero.

ABAQUS/Explicit also offers two damage models: a shear failure model driven by plastic yielding and a tensile failure model driven by tensile loading. These failure models provide simple failure criteria that are designed to allow stable removal of elements from the mesh because of tearing, ripping or tensile spalling of the structure. These models can be used in conjunction with the Mises or Johnson-Cook plasticity models. Each model provides several failure choices including the removal of elements from the mesh.

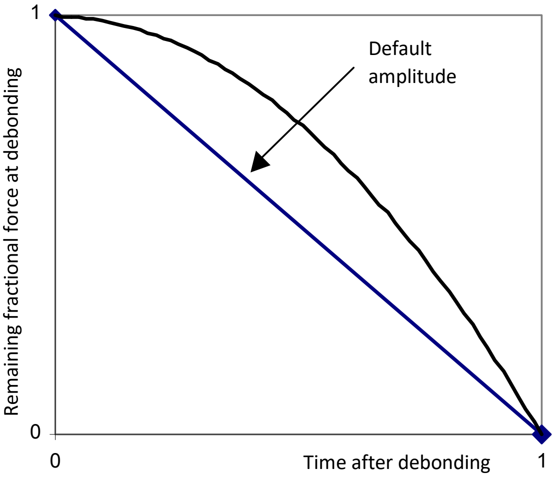

Figure A3.2. Amplitude-time function used to provide the relative magnitude of force to be transmitted

In ABAQUS it is also possible to define breakable bonds that connect the surface of contact pairs from two contact surfaces. Either a time to failure or a damage failure model is used to simulate the post failure response of the bonds. These breakable bonds are design for modelling spot-welds and a spot weld failure criterion is used to model failure. This is a function of the force required to cause failure in tension (Mode I loading) and force required to cause failure in pure shear (mode II loading). This method could possibly be used to model de-bonding of the adhesive/adherend interface. Other possibilities for modelling the adhesive interface include using non-linear spring elements to connect the adhesive elements to the adherend elements or including a narrow row of elements at the adherend/adhesive interface, with different properties to the bulk adhesive to simulate the interface. This would probably require a very refined mesh of elements and knowledge of how the interface should behave. It might also be possible to use contact surfaces with softened behaviour, i.e., as the surfaces move apart the contact pressure between them decreases until it reaches zero (separation).

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Abstract

The design of adhesively bonded structures requires accurate material property data. These data, which are often best obtained from bulk test specimens, can provide an indication of a material's cohesive strength, however, designers also need to consider the interfacial strength between the adhesive and substrates. Information on interface strengths is normally obtained from adhesives joint tests. The complexity of stress distributions in adhesively bonded joints, however, invariably leads to difficulties in obtaining quantitative interface strengths that are applicable to other loading configurations. As part of a BEIS (formerly DTI) funded Measurements for Materials Systems project, "Interfacial Adhesion Strength", several test methods for quantifying adhesion strength have been evaluated to assess their ability to quantify the strength of adhesion between adhesive and adherend. An experimental and Finite Element (FE) study was conducted on each of the selected methods.

As the properties of the surfaces being bonded are critical for the manufacture of strong, durable adhesive bonds, techniques for inspecting surfaces have also been assessed. There is no universal surface or adhesion test suitable for all situations – methods need to be selected based on the materials (adherend and adhesive), expected structural loading and service conditions, degree of accuracy required and budget (time and cost) for the assessment. This document provides guidance in the selection and use of surface characterisation and adhesion strength test methods.

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.

Breaking stress: Stress at the moment of rupture of a specimen.

Brittle failure: Failure in which the fracture surface exhibits no permanent material deformation to the naked eye (e.g., stretching, elongation and necking down).

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).

Composite: Solid product consisting of two or more distinct phases, including a binding material (matrix) and a particulate or fibrous material.

Contamination-free: Absence of foreign matter, both on a treated surface (i.e., cleanliness), or which could migrate through the bulk to a bonded interface with time.

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.

Degradation: Change in the chemical structure of a polymeric material involving a deleterious change in properties.

Delamination: Separation of layers (i.e., planar defect) in a laminate.

Deterioration: Permanent change in the physical properties of a material evident by impairment of these properties.

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.

Hyperelastic: Large strain elastic behaviour modelled using a strain energy potential.

Interface: The region where two materials are in intimate contact. Interfaces can be solid-solid (cured adhesive joint), solid-liquid (uncured adhesive joint), solid-gas (exposed adherend), liquid-gas (open adhesive) or liquid-liquid (insoluble liquids).

Interphase: The region near an interface where the properties of materials are altered from their bulk properties due to the presence of the interface.

ISO: International Standards Organisation.

Laminate: Product made by bonding together two or more layers (plies) of material or materials.

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

Materials model: Constitutive equations linking stress and strain properties.

Modulus: Material property denoting the stress required to extend a specimen by unit strain. (modulus = σ/ε).

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.

Permeability: Property of a material transmitting gases and liquids by passage through one surface and out at another surface by diffusion and sorption processes.

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

Poisson’s ratio: The ratio between axial and transverse strains in a uniaxial tension test as defined in Equation (3), used in the definitions of elastic moduli.

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.

Prepreg: Admixture of resins (with or without fillers), additives, and reinforcements in woven and filamentous form, ready for moulding.

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

Reinforced plastic: Polymer (plastic) with high-strength fibres embedded in the composition, resulting in some strength properties greatly superior to those of the base resin.

Roughness: Micro-roughness represents the fine structure of a surface with dimensions 0.1 µm or less. Macro-roughness, which may also have a significant influence on bond performance, suggests the coarser structure of a surface with dimensions greater than 0.1 µm.

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.

Sealant: An interlaminar layer of polymeric material applied for the purpose of filling gaps and insulating the interior of the joint from external environments. Sealants are not used to provide load bearing capacity although adhesives can also act as sealants.

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.

Soundness: Freedom from weak and loosely attached surface layers.

Stability: The stability of surface layers and oxides, towards water, organic compounds, and elevated temperatures, as a function of time following treatment.

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 (S-N) 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.

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

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

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

Uniformity: Visible or measurable consistency of the other characteristics, and of the regularity of a treated surface area.

Visco-elastic: A material whose properties combine elastic (or recoverable) and viscous (or irrecoverable) components. Visco-elasticity leads to strain rate and temperature dependent properties and is also responsible for damping.

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.

Wettability: A measure of the attractiveness of a solid surface towards a liquid, encompassing aspects such as energy and chemistry.

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 Good Practice Guide aims to provide guidance to technologists, laboratory staff and quality assurance personnel on how to characterise and quantify the adhesion of adhesives to substrates in order to design bonded structures. A general familiarity with laboratory operations and mechanical testing, but not specifically adhesives testing, is assumed. The objective of this Guide is to familiarise the operator with the options available for testing and the factors that can influence the test and design results. It is highly recommended that validation tests be performed to gain confidence in design calculations.

Finite Element Analysis (FEA) is used extensively in the evaluation and design of structures and sub-components. FEA also provides a useful tool for evaluating test methods and interpreting the results. Reliable FEA predictions require accurate materials properties data, which are often best obtained from bulk test specimens. Such data can give an indication of the cohesive strength of the materials, but designers also need to consider the strengths of the interfaces between the adhesive and substrates. Methods for obtaining such data are outlined in this guide. There is no single test that is suitable for every material and loading condition. Often there is a trade-off between the relative accuracy and cost of testing. This becomes increasingly important where a wide range of adhesives or surface treatments may need to be screened, particularly for long-term durability. The following types of test method are described:

- Overlap Test Methods (Tensile-Shear)
- Peel Test Methods
- Fracture Test Methods (Cleavage – Thick Sections)
- Tensile Test Methods
- Flexural Test Methods

Greater detail is given to the test methods investigated in the Measurements for Materials Systems project ‘Interfacial Adhesion Strength’ – pull-off test, butt tension and three-point bend test – but much of the guidance in this document is generally applicable to all joint tests.

Surface preparation is recognised as a critical step in the adhesive bonding process and considerable effort is frequently expended in optimising surface treatments for bond strength and durability. Correct surface preparation is essential ensuring the long-term structural integrity of bonded structures under hostile service conditions. Unsatisfactory surface preparation will result in premature and unpredictable interfacial failure of bonded joints. An essential part of ensuring good adhesion and long-term durability is the ability to control and monitor the surface morphology and chemistry of the substrate materials during surface preparation and storage. Methods of assessing the surface of adherends, to provide a route to quality assurance of bonds, are described.

Emphasis is given to techniques for testing interfacial adhesion strength and assessing surface properties, which can be used simply, quickly, and cheaply, suitable for quality assurance testing or large-scale screening of materials or processes.

The information presented in this guide has been gathered under BEIS (formerly DTI) funded programmes of research on the Performance of Adhesive Joints (PAJ) and Measurements for Materials Systems (MMS).

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Chapter 1

Scope

Scope

The performance of a materials system, such as an adhesive joint, coating, or composite, is influenced by the properties of the bulk materials and of the interfaces within the system. The design of adhesively bonded structures requires accurate materials data and knowledge of the performance of the interfaces. Materials property data are often best obtained from bulk test specimens. Such data can give an indication of the cohesive strength of the adhesive, but designers will also need to consider de-bonding at an interface as a potential mode of failure. However, quantitative properties of the interface are seldom considered in the design process owing to the lack of validated data and the absence of design models and tools. Information on interface strengths can be obtained from adhesive joint test. The plethora of available methods, mixture of failure modes, difficulty in detecting the start of failure and the complexity of evaluating stress distributions in joints, however, creates difficulties in obtaining quantitative interface strengths that are applicable to all loading configurations.

Surface preparation is recognised as a critical step in the adhesive bonding process and considerable effort is frequently expended in optimising surface treatments for bond strength and durability. Correct surface preparation is essential ensuring the long-term structural integrity of bonded structures under hostile service conditions. Unsatisfactory surface preparation will result in premature and unpredictable interfacial failure of bonded joints. An essential part of ensuring good adhesion and long-term durability is the ability to control and monitor the surface morphology and chemistry of the substrate materials during surface preparation and storage. Methods of assessing the surface of adherends provide a route to quality assurance of bonds.

This Good Practice Guide seeks to describe methods for testing interfacial adhesion strength and assessing surface properties. Emphasis is given to techniques, which can be used simply, quickly, and cheaply, suitable for quality assurance testing or large-scale screening of materials or processes. The information presented in this guide has been gathered under BEIS funded programmes of research on the Performance of Adhesive Joints (PAJ) [1-5] and Measurements for Materials Systems. The outputs from this work are available through the Adhesives Design Toolkit website (www.adhesivestoolkit.com), a free information resource for adhesives.

Chapter 2 describes methods that can be used for assessing and quantifying surface conditions. There are many surface preparation methods available to treat the wide array of materials that may be bonded; it is not the purpose of this Guide to recommend any specific treatment.

Chapter 3 describes many of the methods that could be considered for determining the strength of adhesion between the adhesive and adherend. Adhesion mechanisms are discussed in Appendix 1. Good quality test specimens are critical for obtaining accurate and reproducible test data. Preparation of adhesive joints must be undertaken using carefully controlled procedures, since manufacturing variability contributes significantly to the degree of repeatability in the test data. General guidance on adhesive joint manufacture is given in Appendix 2 and Good Practice Guide No. 47 [3].

Interpretation of adhesive joint data, even if the results are accurate and reproducible, may not be straightforward. Chapter 4 outlines some important points to be considered when interpreting results. Methods of including adhesion strength in predictive finite element design calculations are discussed in Appendix 3.

Further information and guidance on the testing of adhesives and adhesive joints may be obtained from other NPL Good Practice Guides [1-5] and various textbooks [6-13].

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Chapter 2

Surface preparation and assessment

- Surface preparation
- Surface assessment methods
- Surface analytical techniques
- Imaging techniques
- Quantifying appearance
- Surface energy
- Mechanical properties of surfaces
- Electrical and optical properties of surfaces
- Summary of surface inspection methods

Surface preparation

Good surface preparation is crucial to ensuring optimum bond strength and reliable long-term performance of bonded joints, particularly under hostile service conditions [6-13]. Unsatisfactory surface preparation will result in premature and unpredictable bond failure at the adhesive/adherend interface. Surface preparation is recognised as the most critical step in the adhesive bonding process and considerable effort is often expended in optimising the surface treatment.

The purpose of surface treatments is to [3]:

- Remove contaminants that may interfere with bond formation;
- Remove weak surface layers;
- Produce a surface morphology that enhances the surface area available for bonding and/or allows mechanical keying;
- Chemically modify the surface to increase surface energy and chemical compatibility with the adhesive.

The selection of surface treatment is largely dependent on the substrate, the required strength and durability of the joint and economic considerations (such as costs and time involved in preparation). Surface treatment processes often consist of a series of different steps. Surface treatments can be classified as either passive or active. 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.

It is important that the process of surface preparation only affects the chemistry and morphology of a thin surface layer of the adherend(s) and does not alter the mechanical and physical properties of the underlying substrate. There are many procedures available – further information can be found in “Guide to the Structural Use of Adhesives” produced by The Institution of Structural Engineers [13] and specific treatments are defined in various standards, e.g., BS 7079 [14], BS 5350-A1 [15], ASTM D2093 [16], ASTM D2651 [17], and BS EN 13887 [18] – see also [3, 19-22]. Advice is usually 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 (Control of Substances Hazardous to Health) specifications.

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 (for examples 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 [3]

Surface assessment methods

Surface characterisation of the adherend prior to bonding is an essential part of the evaluation of surface treatments to ensure optimum bond strength and environmental resistance for specific service conditions. These techniques can provide important information on:

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

Surface and chemical characterisation techniques can provide qualitative and quantitative information relating to the strength and durability of bonded joints [6, 8, 11, 23-27]. This section examines physical and chemical techniques that can be used to characterise surfaces with particular attention given to those techniques that can be used for on-line inspection (i.e., quality control) and are accessible by small industrial concerns [25]. The practicality and limitations of these techniques are discussed, and relevant data is provided to demonstrate the effectiveness of the technique for different substrate/surface treatment systems.

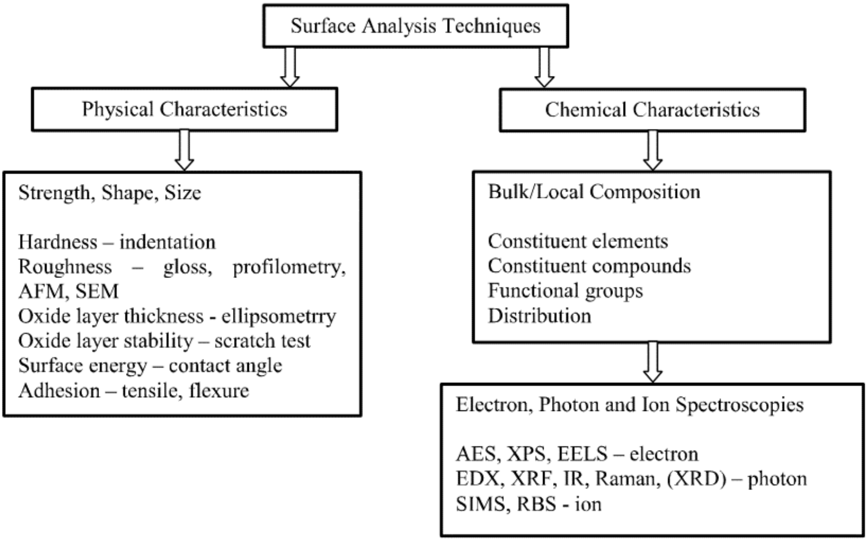

Figure 1. Surface characterisation techniques

There are many physical measurement and chemical analysis techniques that can be used for surface characterisation of metals, plastics (including fibre-reinforced plastic composites) and other substrates -.see Figure 1. The section describes techniques that can be used to evaluate surface treatments applied to ensure optimum bond strength and environmental resistance for specific service conditions. The techniques available to the researcher range from the inexpensive and simple (e.g., visual inspection) to the expensive and specialised (e.g., photoelectron spectroscopy).

Spectroscopic techniques require significant investment in both facilities and trained operators. Hence, they are generally only available at large manufacturing and research facilities. Although many research technology organisations (RTOs) and academic institutions offer spectroscopic measurement services many adhesive bonders are discouraged from regular use by the costs and need to schedule/transport samples.

Surface analytical techniques

Chemical characterisation, either elemental or functional group analysis, can be achieved using spectroscopic techniques. The various techniques can provide qualitative and quantitative information on chemical composition and varying information on molecular structure and physical-chemical characteristics, which determine the attractive forces towards adhesive molecules and, hence, bonding performance. The techniques have different levels of capabilities for scanning areas or providing depth profile information. Except for the optical techniques (IR and Raman), the measurements are performed with samples in an evacuated chamber, which adds to the cost and complexity of the technique. Many techniques require ultra high vacuum (UHV) conditions to avoid scattering or generation of secondary x-rays or electrons from gas molecules. Under UHV, there may be loss of volatile material from the surface and alterations to the microstructure of the surface being investigated.

Infrared spectroscopy (IRS)

Description	Use/Limitations
Absorption of IR photons (transmitted or reflected) occurs at specific frequencies associated with internal vibrations of groups of atoms in molecules.	IR provides: • Qualitative and quantitative chemical analysis data, particularly useful polymer chemistry. • A fingerprint of the adhesive or coating composition in any physical state that can be compared with databases of spectra to enable characterisation of molecular state. Since it is an optical (photon in/photon out) technique it is not necessary for such studies to be carried out in vacuum. IR is a commonly available laboratory technique.

Raman spectroscopy

Description	Use/Limitations
Raman is based on the inelastic scattering of monochromatic light. A laser excites the material, usually in the visible region of the spectrum. The frequency of scattered light is analysed and compared to incident values.	The technique is similar to IR in determining the nature of molecular structures and is a complementary technique to IR when characteristic frequencies are weak or for highly absorbing materials. Samples require minimal preparation, but need to be stable to high intensity light and contain no species that fluoresce when excited by visible radiation.

Energy dispersive x-ray analysis (EDX)

Description	Use/Limitations
X-rays emitted from a surface material upon exposure to a primary beam of electrons are characteristic of the atom from which they originated.	Detection and analysis of the characteristic Xray lines of various elements can be obtained using an EDX system attached to an SEM. EDX generates elemental distribution maps, enabling both qualitative (boron to uranium) and some quantitative (sodium to uranium) elemental analysis. Samples must be studied under vacuum.

X-ray photoelectron spectroscopy (XPS)

Description	Use/Limitations
Measures the energies of photoelectrons emitted from atoms of a sample when irradiated with soft (or low energy) X-rays.	XPS is surface-sensitive and used for quantitative elemental analysis, capable of detecting all elements with the exceptions of hydrogen and helium. It can discriminate between different oxidation states and different chemical environments, and be used in conjunction with inert gas ion sputtering to determine the variation in chemical composition with depth. Some polymeric material samples are sensitive to ion beam damage. Samples must be studied under vacuum.

Auger electron spectroscopy (AES)

Description	Use/Limitations
High-energy electron beam bombardment of the surface results in the emission of Auger electrons at characteristic discrete energies.	AES is a surface sensitive, non-destructive technique for identifying the elements, with the exception of hydrogen and helium, in the first few atomic layers and is able to provide quantitative data (by comparison with known standard samples). Combined with inert gas ion sputtering, AES can obtain depth composition profiles and scan the specimen surfaces with high spatial resolution (0.5µm). AES is not particularly suited to insulating materials. Samples must be studied under vacuum.

X-ray fluorescence spectroscopy (XRF)

Description	Use/Limitations
Irradiation with X-rays results in the release of photons with element specific characteristic energies.	XRF provides qualitative and semi-quantitative elemental analysis for elements above aluminium in the periodic table. It requires very flat samples. Samples must be studied under vacuum.

Secondary ion mass spectrometry (SIMS)

Description	Use/Limitations
The bombardment of a surface with a beam of high-energy ions results in the ejection of molecular fragments, atoms and ions from the surface that are subsequently analysed.	SIMS provides surface elemental analysis and depth concentration profiles on areas from several mm to sub micron. It can detect all elements and isotopes including hydrogen and hydrogenated compounds with very high sensitivity (parts per billion). Quantitative analysis is complex and requires reference standards. Samples must be studied under vacuum.

Rutherford backscattering spectroscopy (RBS)

Description	Use/Limitations
A beam of positive ions is directed at the target surface and produces ions, which are scattered by the sample nuclei, to be measured and analysed.	RBS is used for the quantitative, non-destructive compositional depth profiling and thickness measurements on thin films. The depth resolution is 10 to 20 nm and RBS can probe several thousand atomic layers and is ideal for surface analysis up to 2 µm depth. The erosion and the radiation degradation of the sample material by the particle impact are negligible. Samples must be studied under vacuum.

Imaging techniques

Often the condition of a surface is checked by visual inspection to detect signs of any problems, such as damage (e.g., scratches) and contamination (e.g., grease, marks) that might compromise bonding. The surface appearance and morphology will be assessed to determine whether surface treatments have been applied uniformly (e.g., the roughening caused by grit blasting a smooth surface). Many surface treatments affect the surface morphology at scales too fine to be resolved by the human eye and there are techniques available (summarised below) to view surfaces at higher resolution. These techniques produce images, which give qualitative information of the surface. Often these images are compared with reference images to aid interpretation. Techniques that can provide quantitative information on surface appearance are described in the next section.

Optical microscopy

Description	Use/Limitations
Observation of the surface through magnifying lenses.	Optical microscopes are commonly available laboratory equipment. Resolution is limited by the wavelength of light to around 1 µm, which is not sufficient to view many surface features. Illumination conditions can be altered (e.g., to generate fluorescence) and light signals filtered (e.g., polarising filters) to enhance images.

Scanning electron microscopy

Description	Use/Limitations
A highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated and formed into an image, the intensity of which is governed by surface topography.	Qualitative three-dimensional (3-D) imaging of surface features, Resolution of SEM is approximately 100 times greater than for optical microscopy and features can be seen to approximately 5 nm. SEM is suitable for all materials, but non-conducting materials must be given a thin conductive coating (e.g., gold sputtered), which can mask the true surface morphology. To avoid degradation of the signal by scattering of the secondary electrons SEM measurements are normally performed under vacuum.

Transmission electron microscopy (TEM)

Description	Use/Limitations
TEM focuses a beam of electrons on the specimen. The transmitted signal is projected onto a phosphor screen and an image is created.	Extremely high resolution (0.2 nm) is possible. However, electrons cannot penetrate a specimen very deeply so sample thickness is a major problem. UHV is needed.

Atomic force microscopy and scanning tunnelling microscopy

Description	Use/Limitations
Atomic Force Microscopy (AFM)/Scanning Tunnelling Microscopy (STM) measures the morphology and topography of surfaces on the atomic scale. A sharp tip, mounted on a cantilever, is scanned across the surface (in contact or non-contact modes) using interaction forces. The vertical movements of the cantilever are analysed.	Atomic force microscopy is a popular method used by many industrial R & D sectors to measure surface topography and materials properties with spatial resolutions of 5 - 20 nm and height dimensions in the range from 100 nm down to 5 nm and exceptionally to 0.2 nm. STM can produce a 3-D image for visual inspection of conductive surfaces and also measure relative surface stiffness giving local hardness and modulus information. However, investigated areas are small and scans take significant time to perform. No special atmosphere is required for AFM but the technique is sensitive to vibrations.

Quantifying appearance

Gloss measurement

Reflectance measurements can be made using a gonio-photometer. A beam of light (often a laser) is projected onto the target surface at a specific angle and the amount of light reflected is measured. For a perfectly smooth, mirror surface all the light is at the same angle, the specular angle. Surface roughness causes incident light to be scattered at angles other than that of specular reflection, such that the scatter increases with roughness. Analysis of the angular distribution and intensity of the reflected and scattered light allows parameters related to the surface roughness to be defined. These parameters include the total integrated scatter (ratio of total scattered power to total reflected power, which is related to RMS roughness R_q) and the bi-directional reflectance distribution function (scatter density normalised to incident power which contains valuable information on the distribution and size of surface features). In order to achieve this an array of detectors or a detector moving in an arc relative to the surface are used. Such an arrangement is cumbersome and lacks portability.

Simpler hand-held instruments, gloss meters, are available to quantify the degree of specular reflectance from a surface, (see Figure 2). These instruments follow standards specifying the angle of incident beam and the spot size (ASTM D523 and ISO 2813 [28-29]). A standard black glass is used as a reference for comparisons. Gloss meters have been successfully used with flat and curved surfaces for determining the surface finish of polymer products and painted surfaces for quality control purposes [30]. These instruments provide quantitative data on the uniformity and quality of surface treatments. The technique can be used to differentiate between surface treatments and the quality of the surface treatment. Commercial handheld instruments measure reflectivity at fixed angles to the substrate (20°, 60° or 85°). The technique can be automated for on-line inspection. Whilst gloss measurement shows sensitivity to gross changes in the surfaces, for example easily distinguishing grit blasted regions from untreated, it lacks the sensitivity to discriminate between different levels of surface treatment [25].

Colour measurement by reflectance spectroscopy

Colorimetry can be used to detect differences between surface treatments and the degree or intensity of the surface treatment (e.g., exposure time). Colour is sensitive to surface morphology and chemical composition. When light falls on a surface with an oxide film, several phenomena occur at the interface. Some of the light is reflected back at the film-air interface and does not enter the oxide layer, whilst the transmitted component enters the oxide layer. A portion of the transmitted light is reflected from the metal-oxide interface. Interference of the two reflections will occur, affecting the perceived colour of the surface. The equipment is basically a spectrophotometer that measures the intensity of wavelengths in the reflectance spectra (see Figure 2).

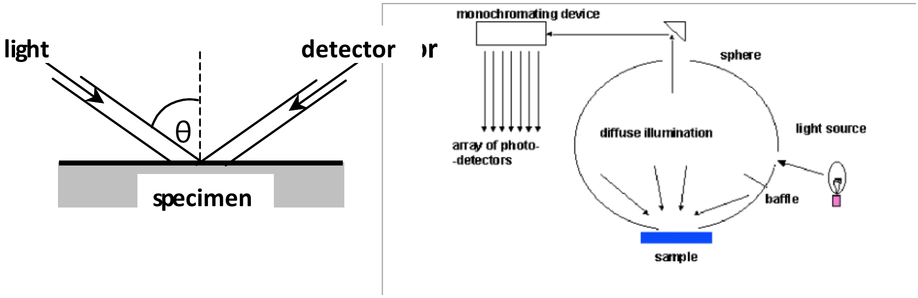

Figure 2. Schematics of reflectance measurement instruments: gloss meter (left) and colorimeter (right)

Colour measurement [31] requires a spectrophotometer to illuminate the sample with white light and to calculate the amount of light that is reflected by the sample at each wavelength interval by passing the reflected light though a monochromatic device that splits the light up into separate wavelength intervals. The instrument is calibrated using a white tile whose reflectance at each wavelength is known compared to a perfect diffuse reflecting surface. The reflectance of a sample is expressed between 0 and 100 (as a percentage). Reflectance values obtained are relative values and, for non-fluorescent samples, are independent of the quality and quantity of the light used to illuminate the sample. The spectrophotometer exposes an area on the surface to a light source with a daylight colour temperature and compares the percentage reflectance within the visible spectrum (360 – 750 nm wavelength, in 1 nm steps) to that of reference white and black colour tiles.

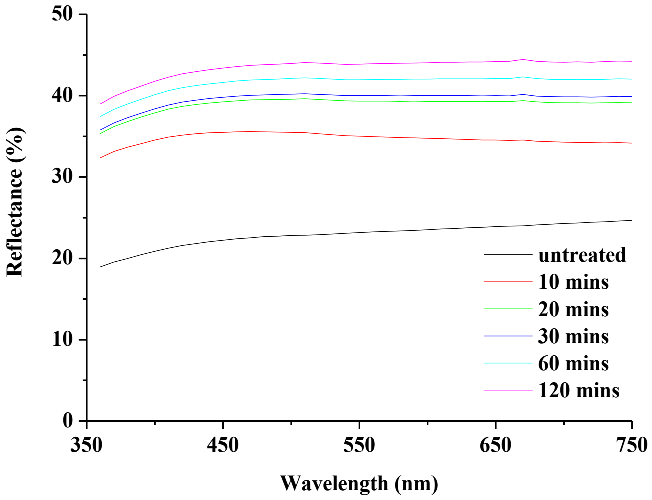

Figure 3. Reflectance spectra for untreated and CAE aluminium

Colorimetry results have shown that surface reflectance measurements have some promise and can detect differences between some surface treatments [25]. Figure 3 shows colour reflectance spectra measurements made for an aluminium sample subjected to different levels of chromic acid etch (CAE) treatment [32]. The reflectance increases with increasing treatment time, corresponding to increasing oxide film thickness. As the oxide layer grows, the reflection becomes brighter. There is a large change between the untreated surface and the treated surface but the differences between different levels of treatment are less significant.

A series of colour measurements, shown in Figure 4, were made [31] on a set of six samples of aluminium with different surface treatments (described in Table 3). Each point on the curve is an average of measurements on five different samples. For clarity, error bars are only shown for Treatment 1 (untreated), the degree of variability is typical of those found for the other curves. The results indicate that the colour measurements can distinguish significant differences between the untreated surface and the treated surface. However, given the degree of uncertainty, it is probably not possible to state with confidence that there are any significant differences between the other surface treatments apart from treatment 6 where the spectral response is significantly flatter than for the other treatments.

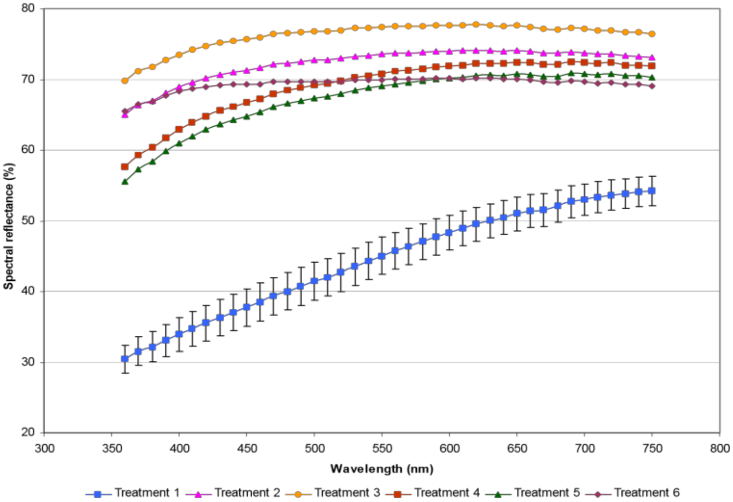

Figure 4. Colorimetry measurements for different aluminium surface treatments

These samples were exposed to hot/wet conditioning (70 °C/85% relative humidity) to degrade the surfaces. Figure 5 shows the effect of 0, 18 and 36 days conditioning on the colour spectra of samples with Treatments 1 (untreated, mill finish) and 2 (light clean). There is no significant change on conditioning for Treatment 1. For Treatment 2 there is a change between fresh and conditioned samples but no significant differences between the two conditioning durations (this was also the case for Treatments 3-6).

Treatment	Finish	Code	Description
1	Mill finish	MF	Untreated aluminium
2	Light clean	PC	Lightly degreased to remove oils - most of the surface structure and oxides still remaining.
3	Full clean	FC	An extensive electrolytic acid etch - equivalent of the optimized system without an anodised layer.
4	Full clean/anodised	FC + 25 nm	Full clean + 25 nm barrier film ("optimised" system with a full etch and a 25 nm barrier anodised film).
5	Light clean/anodised	PC + 25 nm	Light clean + 25 nm barrier film (a variant with a lower level of etch).
6	Over anodised	FC + 25 nm + 75 nm	Full clean + over-anodised - braid (a variant with extended anodisation period to produce a 25 nm barrier + 75 nm porous anodised film).

Table 3. Aluminium surface finishes

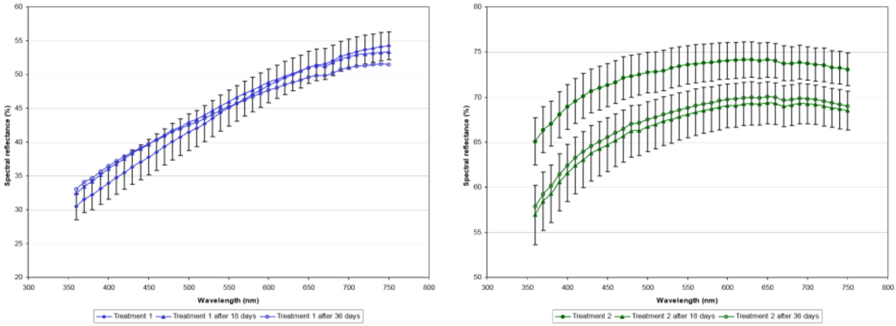

Figure 5. Effect of conditioning on colour of aluminium surfaces mill finish (left) and light clean (right)

Colour reflectance measurements were also investigated as a method for establishing the thickness of an oil coating on a steel surface. The results shown in Figure 6 indicate that reflectance shows a good discrimination between clean and oiled surfaces. These are no significant differences evident between the different levels of oil. This situation persists during conditioning. The spectral response of the clean steel surface shows changes with conditioning but there are few significant changes for the oiled surfaces. This could be a consequence of the protective nature of the oil film.

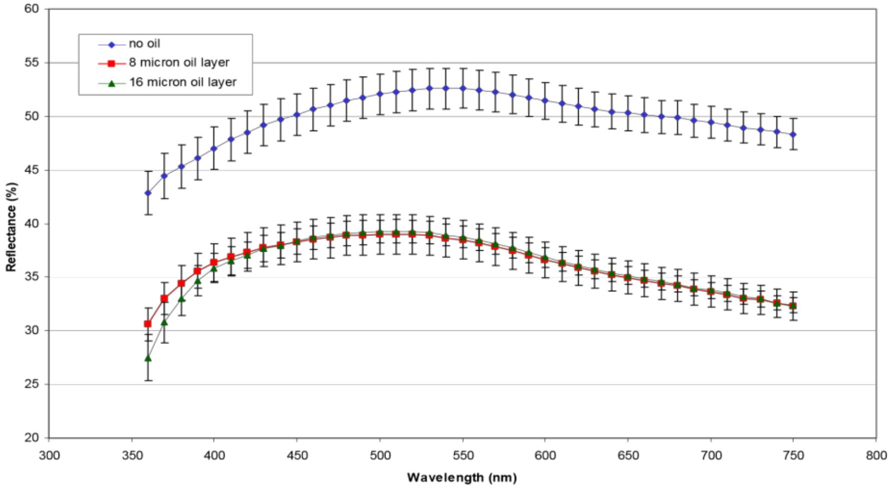

Figure 6. Effect of oil film thickness on spectral response

Surface roughness

Surface treatments are often undertaken to roughen the substrate to increase the surface area available for bonding and to provide sites for mechanical interlocking. Techniques suitable for evaluating surface topography (roughness) include:

- Contact (stylus) profilometry: hand held roughness meters typically have a measurement range of 100 nm to 4,000 nm.
- Optical profilometry; phase shift white light interferometers have typical measurement ranges of 0.2 nm to 100 µm
- Atomic force microscopy (AFM); typical measurement range 10 nm to 7000 nm.

The reliability of any measurement technique will decrease as the lower limit of resolution is approached (i.e., where the surface is smooth according to the depth scale of the instrument).

Surface topography [33] is defined as that which distinguishes a real surface from a perfectly flat, featureless one (i.e., polished). Filters are used to separate the long-wave (waviness) components of the measurement from the short-wave (roughness) components of the surface profile. Waviness is defined as the more widely spaced component of the surface texture. Roughness features are <10 µm in size and are superimposed on the waviness. Data at the beginning and end of each profile is usually discarded to remove any potential edge errors. Statistical parameters are then obtained from these profiles, which help to characterise the surface. The most common parameters used are R_a and R_q, the average roughness deviation and root mean square (RMS) roughness deviation, respectively.

Roughness parameters are calculated relative to a reference line established as the position at which half the trace lies above and half below.

$$R_a = {}^{1}\!/_{L} \int_0^L |y^2(x)| \, dx \tag{1}$$

$$R_q = \sqrt{{}^{1}\!/_{L} \int_0^L |y^2(x)| \, dx} \tag{2}$$

where *L* is the sampled length, *x* is the position along the sampled length and *y*(*x*) are the roughness profile values (see Figure 7).

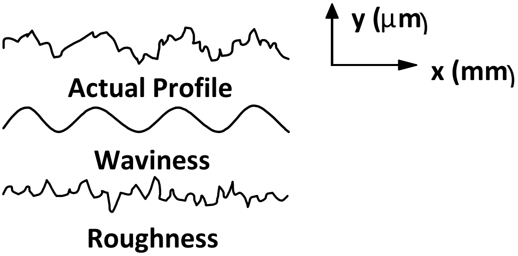

Figure 7. Surface topography definitions

Another useful measure that is occasionally used is peak count, given in peaks per unit length where a peak is any maximum to minimum which extends through a given region around the reference line.

Contact (stylus) profilometer instruments move a loaded probe (usually a diamond) across the target surface and record the vertical movement caused by the irregularities [33-37]. These measurements are analysed automatically using Equations 3 and 4 to produce a roughness value. A typical instrument is shown in Figure 8. This technique is most suitable for hard surfaces, since soft materials can be damaged, and reducing the load may result in some surface features not being registered. Lateral and vertical resolution is affected by stylus shape (usually a cone with a spherical tip (Figure 8) and angle of 60° or 90°), stylus tip radius (typically 5 µm - sharper tips increase damage) and surface profile. Instruments to measure surface roughness are limited by spatial frequency, some features being too wide and others too narrow to be detected, as well as the limits on minimum height of surface features that is detectible. Peaks narrower than the stylus are recorded as broader and the base of narrow troughs may not be reached. This technique can also have difficulty measuring highly curved or convoluted surfaces with steep slopes.

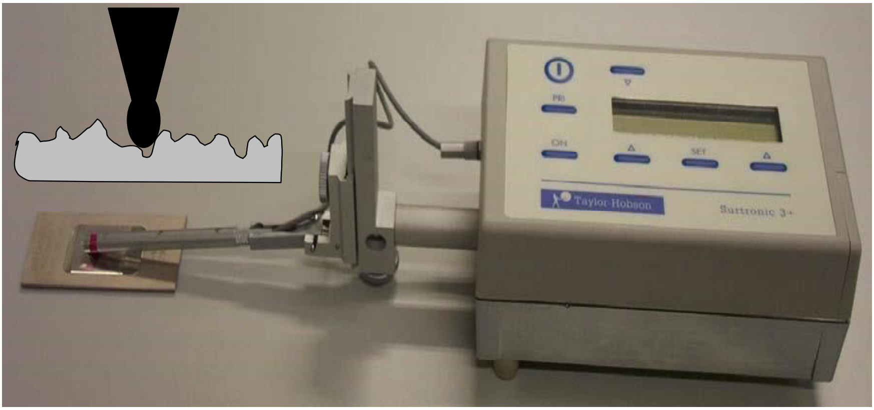

Figure 8. Stylus profilometer

Recent developments have seen the emergence of non-contact (or optical) profilometers, such as laser triangulation systems and optical interferometers for measuring 3-D surface topography. These instruments are being used to measure roughness, finish and texture of surfaces ranging from polished optics to rough surfaces, such as rolled steel and aluminium, plastics, and ceramics. The cost of the instrument increases with resolution and high-resolution equipment is relatively expensive Currently there are no supporting standards for optical systems, although there is a plethora of instruments on the market. It has been shown that stylus measurements provide comparable roughness values to optical profilometry techniques (e.g., 3-D interferometric optical phase shift white light interferometer).

Figure 9 shows an example of the use of roughness measurements. Roughness measurements were made to study differences between six samples of aluminium with different surface treatments (Table 3). The measurements on fresh, unconditioned samples indicate that this simple test indicates significant differences between the untreated sample (Treatment 1) and the other treatments. There are less significant but likely genuine differences between the anodised surfaces (Treatments 4-6) and the cleaned, unanodised surfaces (Treatments 2 and 3). Treatment 4 is nominally the optimum treatment for bonding and has the highest roughness. The effect of hot/wet conditioning (70 °C, 85% RH) is to reduce the roughness of the treated samples towards that of the untreated material (which is not significantly changed by the treatment). After 36 days the roughness of the surfaces is indistinguishable [32].

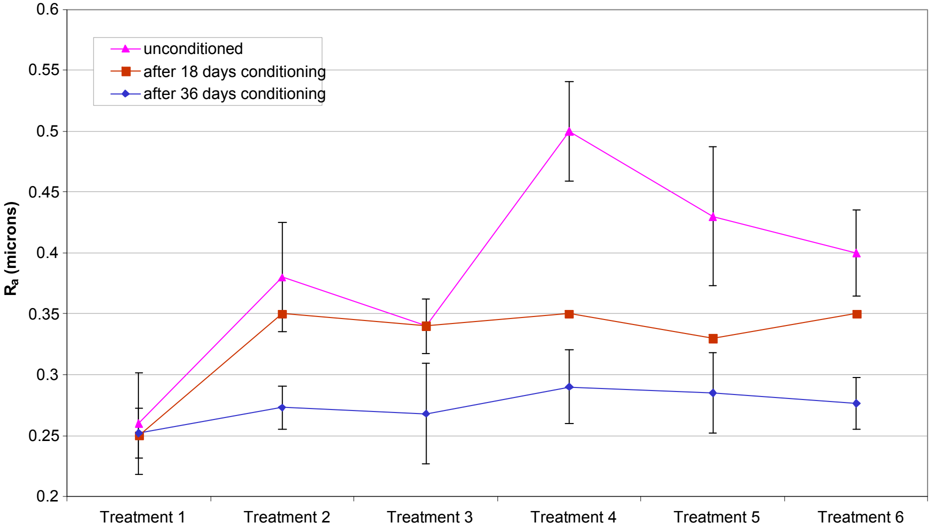

Figure 9. Surface roughness measurements

Surface energy

Wetting is the spreading over and intimate contact of a liquid (adhesive) over a solid surface (substrate). If sufficiently intimate contact is achieved between the two phases, a physical attraction develops causing the liquid to conform to the surface on a macro and micro scale, displacing air and thus minimising interfacial flaws. Good wettability of a surface is a prerequisite for ensuring good adhesive bonding, and hence a high surface energy provides a good bond. Considerable effort has been expended in developing simple wettability tests to assess surface energy/tension prior to bonding [6, 23, 38, 39].

Contact angles are closely related to wettability and adhesion, providing information on the heterogeneity and roughness of the adherend surface and the effect of surface treatments. A liquid (adhesive) will wet a solid (adherend) when its surface energy is lower than the solid surface energy. The equilibrium force balance at the solid-liquid boundary is given by Young equation for contact angles greater than zero (see Figure 10). The lower the contact angle, the greater the tendency for the liquid to wet the solid surface, until complete wetting occurs at a contact angle of zero. Large contact angles are associated with poor wettability and low adhesion forces.

The Young equation relates the contact angle θ, and the surface free energies of the liquid-vapour, solid-vapour, and solid-liquid interfaces (γ_{lv}, γ_{sv} and γ_{sl}, respectively).

$$\gamma_{lv} \cos\theta = \gamma_{sv} - \gamma_{sl} \tag{3}$$

The solid-vapour and liquid-vapour energies are defined with respect to air (the vapour pressure in air is material dependent and can be extremely low). The lower the contact angle, the greater the tendency for the liquid to wet the solid, until complete wetting occurs at an angle θ= 0 (cosθ = 1). The surface tension of the liquid is then equal to the critical surface tension of the substrate. The surface energy of a solid can be determined by using the Young equation and probe liquids with different surface tensions to produce a Zisman plot [6]. However, in most cases the contact angle is used as a relative measure of the surface energy.

Surface energy is sensitive to the chemistry of the surface, the morphology, and the presence of adsorbed materials. The adsorption of chemicals on a surface lowers the surface free energy (wettability). Surfaces with high surface energies will have a strong tendency to adsorb materials (e.g., moisture or dust particles) from the atmosphere, reducing wettability. Therefore, contact angle measurements offer a means of studying the ageing of surfaces.

Simple wetting tests

The water break test [40] is a qualitative (go/no-go) test, involving the specimen (in the form of a flat plate) being either immersed in water or water brushed or sprayed onto the surface. The plate is then checked to determine the distribution of water on the surface (i.e., remains as a continuous film indicating good wettability or forms distinct droplets indicating poor wettability).

The wetting or Dyne pen test is a semi-quantitative test [41, 42] that involves marking the surface with a pen containing ink of a known surface tension and observing whether or not the initial continuous line breaks into distinct droplets (indicating that the surface energy is lower than the surface tension of the ink). 'Dyne solutions' are available in sets of different surface tensions and systematic use can quickly provide an estimate of surface energy. Despite the widespread use of these solutions, the method has notable critics [43]. The shelf life of pens can be limited, particularly if in regular use, as transfer of contamination from surfaces to the ink may occur.

Contact angle determination by sessile drop

Contact angles between liquid drops and surfaces can be measured directly from the angle formed at the contact between the liquid and the flat surface [44-47]. Measurements can be made using a goniometer, an inexpensive instrument. The drop is illuminated from behind and viewed through a lens focussed on the silhouette of the drop. A reference line is manually positioned to read the contact angle. The drop may also be projected onto a screen to view the contact angle. Manual measurements can be prone to operator subjectivity.

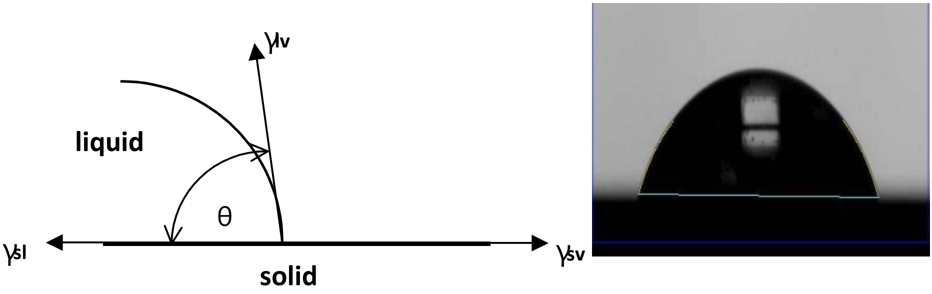

Figure 10. Contact angle measurement

Operator subjectivity can be removed from the process by image analysis of the drop shape and contact using a computer programme. There are many commercially available automated contact angle measurement instruments where the image of the drop is viewed using a video camera and captured on computer. An image analysis programme detects the edge of the drop and the surface. Numeric algorithms are run to establish the ‘shape’ of the drop and the slope of the edge at contact with the surface. The software will also analyse the shape of hanging pendant drops to measure surface tension of liquids. Axisymmetric Drop Shape Analysis (ADSA) is one example of a computer program that uses digital image analysis to detect the edge of a static drop and accurately determine the interfacial tension and contact angle [48]. ADSA is capable, under ideal conditions, of determining contact angles to very high accuracy with uncertainties of less than 0.1°. Through careful control of the position of the syringe plunger it is possible to make the drop advance and recede on the surface. Images of the drop can be captured in real time allowing measurement of contact angle hysteresis through the advancing and receding contact angles.

With automated contact angle measurements, the determination of contact angle should be straightforward provided that:

- The background illumination is of the appropriate intensity to achieve a good contrast between the drop and the background;
- The background illumination level is uniform;
- The camera achieves a sharp focus on the drop and base;
- The surface is flat and level (e.g., checked using spirit levels in two orthogonal directions);
- The plane of the surface is normal to the plane of the camera, i.e., the surface should not slope from left to right or from back to front in the image;
- The surface roughness is not significant (local topography at the junction of the drop and surface can lead to interpretation errors).

During contact angle measurements drops tend to spread on the surfaces with time and the volume of the drop may change due to evaporation or absorbance of moisture. Figure 11 shows a measured contact angle decreasing with time. The procedure for taking measurements needs to specify the time after dispensing the drop at which the measurement should be made, to reduce the measurement variability. A duration of 30s after dispensing the drop is suggested.

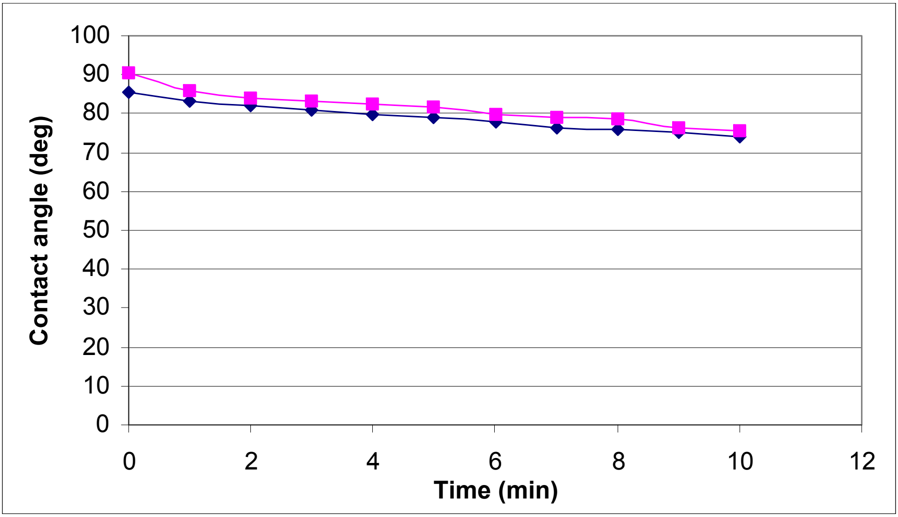

Figure 11. Time dependence of contact angle measurements

Wetting balance

Contact angle can be measured using the Wilhelmy plate technique [38, 39, 49, 50]. This technique measures the forces exerted as the solid-liquid phase boundary is moved along the sample surface as the plate is withdrawn from the liquid (Figure 12). The sample is attached to the balance (with a resolution of 1 µg) and the balance tared to remove the weight of the sample. The liquid (of known surface tension) is raised to meet the solid and the point of contact is determined and recorded as zero immersion depth. The plate continues to be lowered into the liquid to a set depth and the forces on the balance are continuously monitored. The process is then reversed and the solid is raised from the liquid. The contact angle θ may be calculated from the interaction force *F*, sample geometry (perimeter of the edge, *P* = 2 *x* length + 2 *x* thickness) and liquid surface tension (γ_l).

The forces involved are:

$$F = Wetting\ Force - Buoyancy \tag{4}$$

Buoyancy is accounted for by extrapolating the force back to the point of zero immersion depth. The force is given by:

$$F = \gamma_l P \cos\theta \tag{5}$$

When the contact angle is zero (i.e., perfect wetting) the value of *F*/*P* should reduce to the surface tension of the liquid.

Standard methods for wetting balance tests have been produced for determining the efficiency of the wetting of wires by solders [49, 50] but have yet to be standardised for the evaluation of substrates. When carrying out Wilhelmy plate measurements the following points should be considered:

- The edges of the plate immersed should be straight and meet at right angles;
- The plate should be attached vertically so that it meets the surface perpendicularly;
- There should be no bubbles adhering to the surface of the plate when immersed;
- The plate should not float in the liquid.

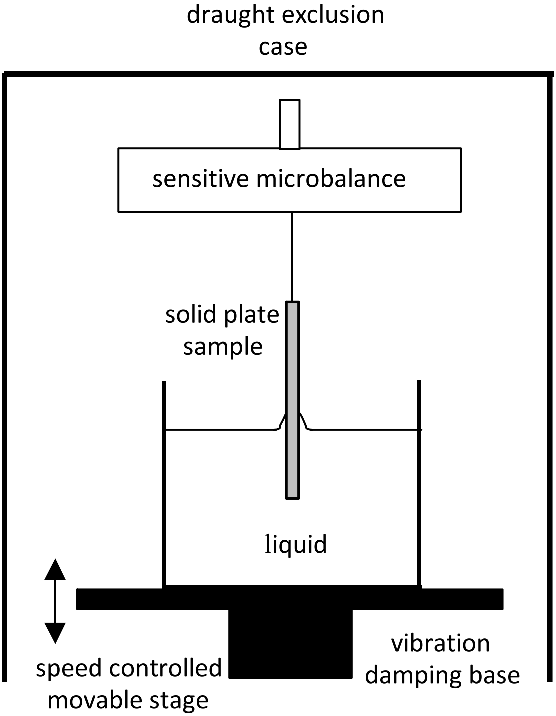

Figure 12. The Wilhelmy plate method

Mechanical properties of surfaces

Strong adhesive bonds require that the surface of the adherend is strongly attached to the rest of the substrate. Weak oxide films, coatings or boundary layers can initiate premature failure. This section describes techniques that can be used to assess the effect of surface treatments or environmental exposure on the mechanical properties and stability of surface layers.

Hardness and indentation

Hardness refers to the ability of a material to resist permanent indentation or deformation (or compressive loads) when in contact with an indenter under load (Figure 13) [51]. Hardness can be assessed on different scales depending on the depth that the indenter penetrates. The basic principle behind hardness testing is to load an indenter into a surface and measure the deformed imprint after retraction of the indenter. The indenter may be spherical (Brinell test [52]), pyramidal (Vickers [53] and Knoop [54] tests) or conical (Rockwell test [55]). The indentation is generally inspected using an optical microscope attached to the hardness test apparatus. Hardness data is defined in terms of the indenter geometry and for visco-elastic/visco-plastic materials the duration of loading. In the Brinell, Vickers and Knoop tests, hardness is the applied load divided by the unit area of the indentation and is expressed in kgf/mm^2. In the Rockwell test, depth of indentation is measured and converted to a hardness number (no associated units), which is inversely related to depth.

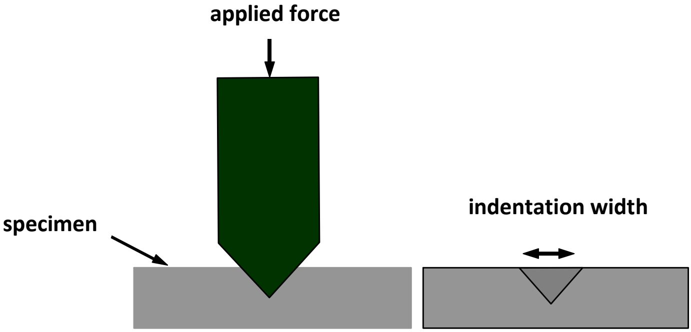

Figure 13. Hardness measurement

For standard hardness measurements, an indentation load of 30 N (or greater) is used and the size of the indentation is approximately 300 µm in size in the plane of the tested surface.

Micro-hardness testing involves smaller indenters and significantly smaller loads (10 mN to 10 N). The corresponding indentation is far smaller and is associated with local properties, whereas standard hardness tests provide bulk hardness measurements and may not distinguish the properties of the surface region. However, it is also more difficult to measure residual imprints for micro-hardness tests, as the residual imprints are relatively small.

Indentation tests instrumented for deflection and force, such as micro- or nano-indentation [56], provide more usable information on the mechanical properties of the near-surface material (including stiffness, plasticity, and fracture). Continuous stiffness measurements during indentation can be used to determine mechanical properties as a function of indentation depth. These techniques can provide very sensitive measurements of the mechanical properties of the surface layer. Analysis of the stiffness data can provide information on fracture and debonding. The combination of continuous stiffness indentation followed by AFM imaging can reveal much information about the surface. However, sensitive instrumented indentation instruments capable of high-resolution measurements are expensive and require specialist operators. Measurements are very sensitive to external vibrations and temperature fluctuations.

Hardness tests can be applied to a wide range of materials and used to characterise the mechanical structure of surface layers (e.g., oxide films or coatings) [56]. Hardness measurements have been shown to be sensitive to surface treatment [27], as shown in Table 4. The associated scatter in hardness measurements can be high, thus differentiation between the levels of treatment may be difficult. There are numerous hardness standards (e.g., [52-55, 57, 58]).

Surface Treatment	Microhardness (Vickers Hardness No.)	Ultra-micro Hardness (N/mm^2)
Aluminium		
As received (untreated)	80-110	1,400
Phosphoric Acid Anodising (PAA)	65-84	2,900
Chromic Acid Anodising (CAA)	79-135	19,000
Titanium		
As received (untreated)	258-352	10,800
Sodium Hydroxide Anodising (SHA)	234-248	800
Chromic Acid Anodising (CAA)	322-369	12,500

Table 4. Hardness measurements for surface treated aluminium and titanium [27]

Scratch testing

The adhesion scratch test is a commonly used method of assessing coating adhesion (see Figure 14) [51, 56]. The test is a variant on a hardness test and is designed to generate stresses at the interface between the coating and the substrate, which exceed the interfacial bond strength of thin, high adhesion coatings or films. A loaded diamond tipped stylus (10 to 60 N) is drawn across the target surface with a steadily increasing load (typically 100 Nmin^{-1}) until some well-defined failure occurs, usually flaking or chipping. The horizontal displacement rate is nominally 10 mm.min^{-1} with sample size being typically 25 mm × 15 mm.

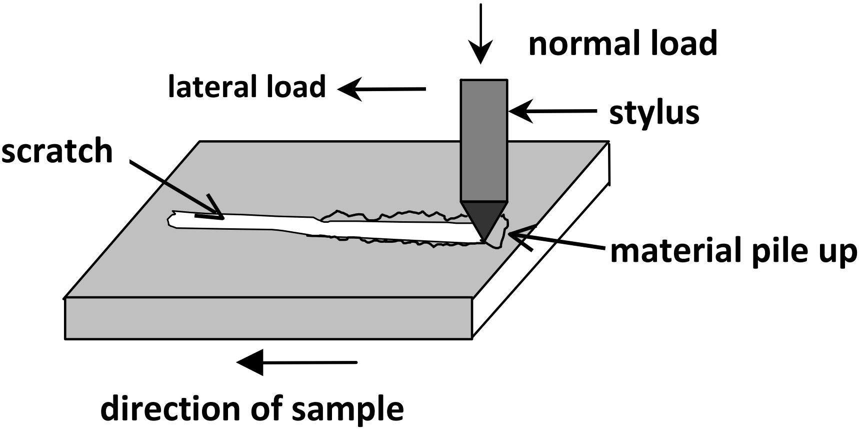

Figure 14. Scratch test method

During the test, the penetration depth and stylus position are recorded. These data can be supplemented with the vertical indenter load, the horizontal force on the indenter and acoustic emission to enable the coefficient of friction and the point of failure to be determined. The scratch can be subsequently analysed with a profilometer, SEM or optical microscopy to ascertain the scratch shape (residual depth, scratch width and pile-up height) and allow the failure mechanism to be identified. Varying the loading rate, the scratch speed and the indenter shape markedly affect the results of the test. Scratch testing equipment is commercially available. Measurements are comparative although there has been recent work to provide a scratch test calibration procedure, draft standards, and reference materials.

Modulus by surface acoustic wave

The propagation velocity of acoustic waves in a medium is proportional to the square root of (elastic modulus divided by density). Surface acoustic waves have very little depth penetration and therefore provide information on the properties of the surface layer rather than the bulk of the material. The laser surface acoustic wave (L-SAW) technique [59] focuses a short laser pulse onto the surface to generate a sound wave that propagates along the material and is detected by a piezoelectric foil. The characteristics of the propagating wave (velocity, wavelength, intensity) can be analysed, using complex functions, to infer the density, Poisson ratio and elastic modulus for both the substrate and the film.

Electrical and optical properties of surfaces

Surface resistivity

The electrical impedance of a surface will change with the surface properties and offers a means of monitoring changes to surface characteristics. A method of converting electrical properties to the surface characteristics is needed to relate measurements to bonding parameters.

Surface resistivity is defined as the electrical resistance of the surface of an insulator and is expressed in ohms/m^2 [60, 61]. An electrical potential is applied between two electrodes on the surface of the test and the resultant current is measured. The surface resistivity σ is determined [60] using:

$$\sigma = K_S \frac{V}{I} \tag{7}$$

where V is the applied voltage, I is the measured current and K_s is the test cell constant for surface resistivity based on cell geometry.

A configuration for measuring surface resistivity is shown in Figure 15. The current is only measured between the bottom two electrodes as shown in the diagram. The top electrode is guarded so that only current flowing between the two lower electrodes is measured (by a picoammeter). The key test parameters are applied voltage, length of electrification time, humidity, and temperature. The longer the voltage is applied, the higher the resistivity. This is because the sample charges exponentially with time. When measuring materials with high surface resistivity, background currents due to charging, static or triboelectric charge, or piezoelectric effects, may cause significant measurement errors.

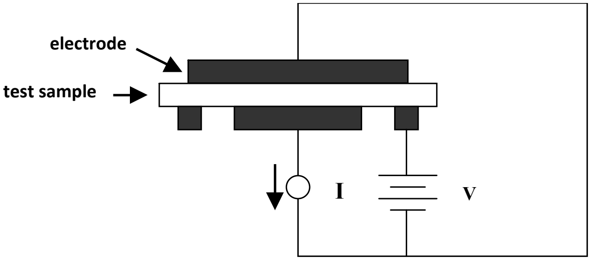

Figure 15. Surface resistivity measurement

The Alternative Current Method is a variation on the surface resistivity method where a bias polarity of positive polarity is applied, and the current is measured after a specified delay time. The polarity is then reversed, and the current is measured again using the same delay time. This process is repeated, and the resistance is calculated from a weighted average of the most recent measurements.

AC impedance techniques have proved successful for characterising oxide films and coatings. Based on an electrochemical method used for corrosion studies to measure oxide growth rates, the test surface is immersed in an electrolyte and an alternating voltage is applied. The resultant current is measured as a function of frequency. Surface impedance is determined from the resistance, capacitance and inductance of the surface and the resistance of the electrolytic solution. It is possible to relate film thickness, film composition and oxidation rate for surface anodisation treatments using impedance measurements. The AC impedance technique can differentiate between various surface treatments and levels of treatment for a given surface treatment.

Electrical techniques are sensitive to variations in the composition and thickness of the oxide film and surface contamination. The electrical and environmental conditions should be kept constant to ensure consistent data (e.g., surface impedance decreases with increasing humidity levels). Poor electrical contact between sample and electrodes will also have an adverse affect on the reliability of surface resistivity measurements. The electrode area must be equal to the contact area since any discrepancies will contribute measurement errors. Although the technique is unsuitable for on-line inspection, there is considerable scope as a developmental tool and for modelling oxidation processes.

Electrical measurements are used as non-destructive testing (NDT) methods for detecting the presence and monitoring the evolution of cracks in metals [62-65]. These techniques may also be suitable for monitoring changes in surfaces. The alternating current potential drop (ACPD) takes advantage of the fact, that when passing through a metal, an alternating current is not distributed uniformly through the depth of the metal. Instead, it is forced to flow mainly in a thin surface layer, an effect often referred to as the “skin effect” Thus, variations in the surface will cause measurable perturbations in the electric field [62-64]. The eddy current (EC) method as a non-destructive tool was developed in the 1940s. The technique has been widely used for crack detection in aerospace, power and oil and gas industries, and has been standardised as an NDT method in many applications.

The principle of eddy current is simple [65]. Eddy current measurements are generated in the sample by exciting an alternating current at a given frequency through a coil, often called a probe coil, located as near as possible to the component to be inspected. As a result of the eddy current in the component, changes take place in the impedance of a pick-up coil. The impedance of the coil is sensitive to the thickness of insulating coatings and is also affected by the presence of defects in the component. Therefore, under a given test condition where the design of the probe and the properties of the component are known or constant, the defect can then be detected and sized by measuring the impedance (phase and amplitude) characteristics in a pick-up coil above the surface.

Ellipsometry

Ellipsometry is an optical technique for probing the properties of thin films (less than the wavelength of the incident light) [27, 66, 67]. A polarised light beam (usually a laser) is directed onto the surface at an oblique angle of incidence. The phase change and intensity (amplitude) of the reflected light are measured. The interference of multiple reflections within surface layers provides information about the layer thickness *t*, optical constant *n* (index of refraction) and extinction coefficient *k* (a measure of absorption) for each layer interacting with the incident beam. The optical properties of isotropic materials can be represented by the complex index of refraction $\tilde{n}$ ($\tilde{n} = n + ik$). Ellipsometry can yield information about the thickness, morphology and chemical composition of layers that are thinner than the wavelength of the light source.

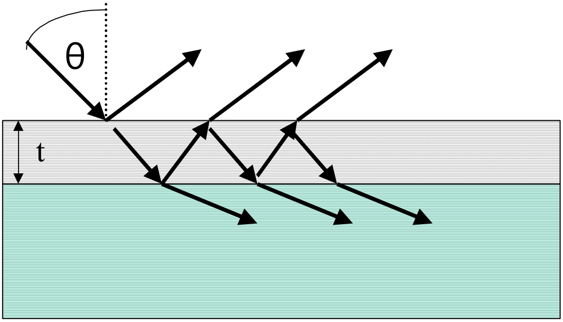

Figure 16. Schematic of multiple reflections

Most ellipsometers use a fixed incidence angle. However, Variable Angle Spectroscopic Ellipsometer (VASE) instruments give increased resolution [66]. VASE ellipsometers can also incorporate infrared spectrometry (IR-VASE) and provide a powerful technique for probing the optical properties, chemistry, and thickness of surface films. The molecular bond absorption bands for different surface treatments are distinguishable through IR ellipsometry data making it possible to fingerprint the surface chemistry (molecular bonds) present in the surface layers. The presence of a barrier layer or contamination will result in a different molecular bond absorption fingerprint.

Ellipsometry analysis requires a baseline set of known properties of the surface (measured on a ‘clean’ fresh surface and the surface film (e.g., Al_2O_3 ($n = 1.766$)) in the case of aluminium. The determination of film thickness is achieved by modelling the optical properties and requires a good knowledge of the optical properties of individual layers. Multi-layered systems can be modelled but this is a complex process and will lack accuracy unless information on each material layer is available. Ellipsometry measurements of the thickness of films [25] produced on aluminium using different levels of anodisation agreed well with transmission electron microscopy (TEM). Ellipsometry also provided evidence of differences in optical properties in the X- and Y-directions resulting from the milling process.

Optically stimulated electron emission

Optically stimulated electron emission (OSEE) is a method for monitoring surface contamination and surface treatments for metallic substrates [25]. It is highly sensitive to very low levels of contamination and has been successfully used for determining surface characteristics in multi-stage processing. OSEE operates by illuminating the area of inspection with ultraviolet (UV) radiation in the presence of a direct current (DC) electric field (see Figure 17). The UV radiation liberates electrons in the area of inspection by the photoelectric effect. The free electrons are collected on the positively charged anode with the magnitude of the resultant current being strongly related to the level of surface contamination. Contaminants absorb the UV radiation, thereby reducing the number of electrons emitted from the substrate, thus the greater the current the cleaner the surface. OSEE is particularly suited to studying contamination build-up following surface treatment and could be used to determine maximum time allowable between treatment and application of an adhesive (i.e., open time).

Six different aluminium surfaces were inspected using a portable OSEE unit. This instrument is suitable for both hand-held and on-line inspection (scan facility) and requires minimal operator training. A total of six measurements were taken for each condition and the results averaged. The units of measurement for the technique are based on a voltage relative to the condition exhibiting the highest output, which will depend on the nature of the substrate, cleanliness, etc., as well as the gain used to amplify the signal.

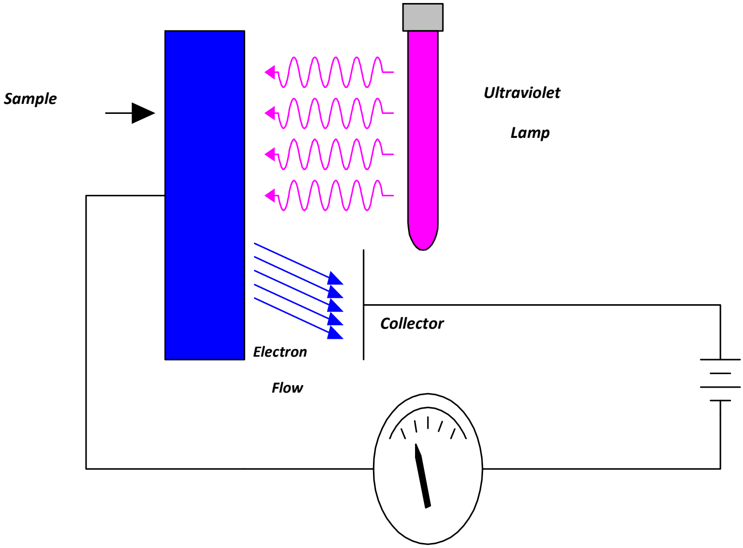

Figure 17. Optically stimulated electron emission (OSEE)

In general, as the cleanliness of the metal increases, the OSEE reading will increase due to a decrease in the attenuation of electrons by organic contaminants. However, as the thickness of the oxide layer increases, the OSEE will decrease due to an increase in electron attenuation by the oxide layer [24]. The results for the anodised treatments examined tend to correlate with these trends, Figure 18. The mill-finished material exhibits the lowest output due to the heavy oxide and organic contaminants on the metal surface. The highest readings were obtained for the lightly cleaned material, which is followed closely by the full electrolytic etch. Once the barrier and/or anodised film are applied, the OSEE output drops dramatically due to the increase in electron absorption by the thin films. There are minimal differences in the readings obtained for full clean + 25 nm barrier film ("optimised" system), light clean + 25 nm barrier and full clean + over-anodised - since the UV source is of a fixed wavelength so a substantial portion of the electrons are absorbed.

Note 1: Contaminants or thin films on the surface, depending on their own photoemission characteristics, can either enhance or attenuate the inherent emission from the surface as well. It also should be mentioned that there could be considerable variability in measurements depending on the uniformity of the surface with respect to cleaning and/or film thickness.

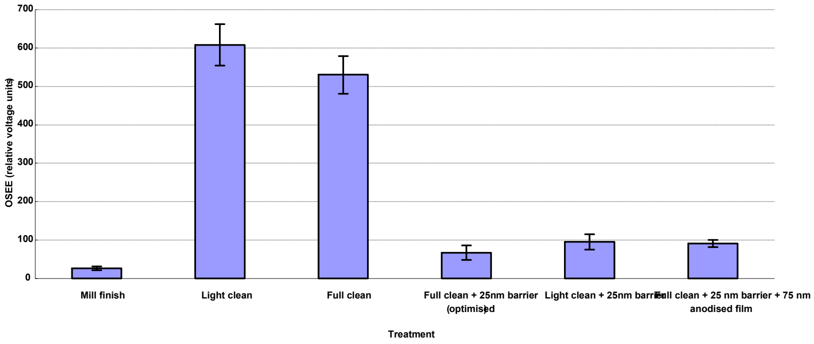

Figure 18. OSSE measurements on anodised aluminium (courtesy of Alcan, Ontario)

In summary, OSEE is suited for on-line inspection to determine the level of cleanliness or degree of treatment but unable to provide absolute measurements of surface activity for different treatments or ageing times. The technique is only able to detect barrier layers/cleaning levels through changes relative to initial substrate surface measurements. Hence, calibration is required for each surface to be assessed. Efficacy is compromised when contaminants fluoresce. OSEE is a non-contact, non-destructive technique that provides quantitative data. It is fast and relatively inexpensive, and straightforward to operate. The technique can be used to assess different methods of cleaning or the effectiveness of different cleaning agents.

Summary of surface inspection methods

Many techniques are useful for determining changes in surface properties for different levels of treatment, although the range of application of these techniques to many materials systems is generally limited. A study of applicability of several methods for a range of surface preparations was undertaken [25] and the results of this study are summarised in Table 5. Additional work was then undertaken to investigate some of these tests for following changes in surfaces due to ageing and accelerated conditioning [32].

Colorimetry is a versatile technique for detecting changes in surface morphology but, while it is possible to quantify changes in reflectivity and relate these changes to levels of treatment or various stages in production of multi-layered systems, the level of uncertainty can limit the ability to distinguish between similar levels of treatment.

Ellipsometry can be used to accurately determine layer thickness of multi-layered systems, provided the analyst has a good knowledge of the technique and optical properties of individual layers within the system.

The sessile drop method appears more reliable than the Wilhelmy plate technique in differentiating between surfaces with good and poor adhesion. The Wilhelmy technique requires considerable care in specimen preparation, whereas there are no special preparation requirements for the sessile drop method. Contact angle appears most useful for assessing bonding to polymers.

The portable units used for measuring surface roughness, surface resistivity and gloss required minimal operator training and were relatively inexpensive. These techniques can distinguish gross differences in treatments (e.g., untreated areas from treated areas) but are not sensitive enough to separate different levels of treatment or conditioning. OSEE is an on-line production tool for inspecting surface quality of metal substrates but is limited in its use for determining the surface activity of aged materials (i.e., off-line).

Corona discharge trials demonstrated the methodology and logic behind planning and conducting a matrix of tests in a systematic manner using statistical design of experiments (DoE) approaches [68]. Combined with surface characterisation techniques, such as colorimetry and the sessile drop method for measuring contact angle, this approach enables production engineers/researchers to assess the effects and non-linearity of key processing factors and possibly optimise process conditions.

Characterisation Technique	**Surface Treatment**				
	Grit Blasted	**CAE**	**Anodised**	**Oil Lubricated**	**Corona* Discharge**
Micro-hardness	No	No	No	No	No
Contact Angle	Yes	Yes	Yes	No	Yes
Profilometry					
Contact	Yes	Yes	Yes	No	No
Optical	Yes	Yes	Yes	No	No
AFM	Yes	Yes	Yes	No	No
Gloss Reflectivity	Yes	Yes	Possibly	No	No
Colorimetry	Yes	Yes	Yes	Yes	No
Ellipsometry	No	Yes	Yes	No	No
OSEE (on-line)	Yes	Yes	Yes	No	No
Surface Resistivity	No	No	No	No	No

Table 5. Suitability of characterisation techniques for different surface treatments
*(*Corona discharge treatment of glass fibre-reinforced polypropylene)*

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Chapter 3

Adhesive joint tests

- Introduction
- Overlap test methods for adhesives (tensile-shear)
- Peel test methods for adhesives
- Fracture test methods (cleavage – thick sections)
- Tensile test methods
- Flexure test methods

Introduction

Chapter 3 of the Guide focuses on (joint) test methods for characterising strength of adhesion between polymeric adhesives and substrates. Adhesive joints tend to be designed on the basis of perfect bonding with the assumption that the locus of failure will be in either the adhesive layer or the adherends. The properties of the interface tend to be ignored in the design, in part because reliable properties for the interface are difficult to obtain. It is common design practice to employ Finite Element (FE) methods to predict structural stiffness and stress distributions. The approach is often to model the continuum behaviour of the adhesive layer in order to predict joint stiffness and strength [4] but fracture methods may also be employed (Appendix 3). To accurately predict the performance of the adhesive joint using Finite Element Analysis (FEA) the following information is required:

- Mechanical properties for the adherends and the adhesive;
- Dimensions of the parts;
- Constraints and loads on the part;
- Failure criteria for the materials employed;
- Failure criteria for the bond at the interface; and
- Material models for adherends and the adhesive.

It is assumed that information on the material properties of the adherends and adhesives, joint dimensions, constraints, and loading are available and can be modelled accurately. Methods suitable for determining material properties (elastic constants, yield functions and ultimate strength) of the adhesive needed for design are described in other Good Practice Guides [1, 4]. Failure can originate in regions of the joint where the stress exceeds the cohesive strength of the adhesive, which depends on many factors such as temperature, strain rate, load history and chemical exposure. However, failure can also start at the interface where the local stress exceeds the interfacial adhesion strength and this needs to be considered. The strength of adhesion throughout the lifetime of the bond will depend on many factors, including:

- Mode of loading;
- Relative stiffness of the adhesive and adherends;
- Attractive forces between the adhesive and the adherend;
- Presence of defects or contamination;
- Degree of intimate contact between the adhesive and surface;
- Surface properties of the substrate;
- Material properties of adhesive and adherend in the interfacial region, which may differ from the bulk and change during service; and
- Degree of chemical or physical degradation of the interface.

The complexity of the interfacial region makes it impossible, except perhaps in very simple model systems, to predict the strength of an ‘adhesive bond’ from first principles. Therefore, to determine adhesion strength parameters, adhesive joint tests will be required.

There are many test methods available but their usefulness for establishing quantitative adhesion strength is variable [69]. Even in simple joint configurations, such as a single-lap shear joint, the resulting stress and strain distributions in the adhesive layer can be extremely complex with a mixture of shear, peel and hydrostatic pressure stresses occurring. Such situations are not easily amenable to closed form analytical methods and FEA may be needed to help interpret results. The preparation of joint test specimens is well recognised as having a critical role in the accuracy and reproducibility of test data and some guidance is reproduced in Appendix 2 [2, 3].

When considering a test method for establishing adhesive strength there are some basic requirements that should be considered. An appropriate test method should:

- Induce failure at the interface under study;
- Provide quantitative test data that are suitable for design calculations;
- Ensure a uniform, predictable stress distribution in the region of failure;
- Involve straightforward specimen preparation and robust test procedures; and
- Be accepted by users (e.g., through standards) and cost effective.

The choice of test method to study adhesion strength will depend significantly on the bending stiffness of the materials to be bonded and the likely loading vectors on the component. It is difficult to relate joint strength data from one configuration to a different configuration. Therefore, it is preferable to use joint tests that approximate the important modes of loading in the structure.

The basic modes of loading of adhesively bonded joints are (see Figure 19).

- Peel loads produced by out-of-plane loads acting on thin/flexiuble 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/thick adherends at the ends of the joints.

Tensile, cleavage or torsion loadings require two thick, rigid adherends. Peel loading requires at least one flexible adherend. Tensile-shear loading is the most versatile.

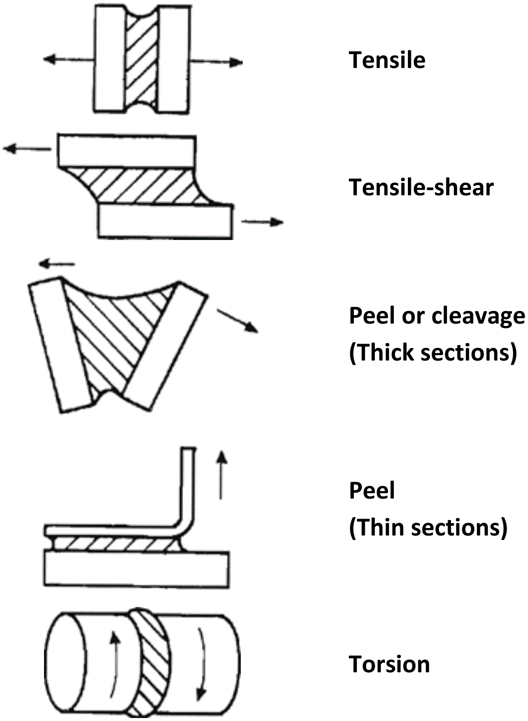


Figure 19. Basic adhesive loading configurations

Overlap test methods for adhesives (tensile-shear)

Shear test methods are widely used to obtain data on bond strength and durability [69]. Many of these tests are standardised and well accepted by the industry. Most shear test methods use specimens with flat adherends, bonded with an overlap, which are pulled in tension with the direction of loading parallel to the length of the overlap.

Single-lap shear test

[The single-lap shear test is ubiquitous in the field of adhesives and adhesion testing. National and international standards [70-75] set out the basic methods. Lap shear test results are extensively quoted on adhesive data sheets, often providing ‘evidence’ for ability to stick to different materials or of bond durability. The test specimen, shown in Figure 20, is loaded in tension until failure. BS EN 1465 [71] 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. It is good practice to use tabs to reduce out-of-plane deformation (i.e., bending).

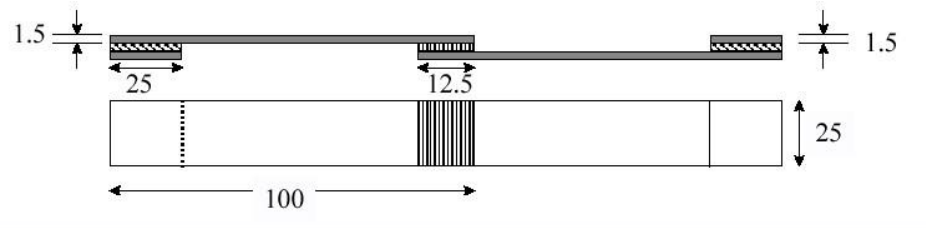


Figure 20. Single-lap shear test

The lap-shear strength τ is given by:

$$\tau = \frac{P}{bl} \tag{8}$$

where *P* is the maximum load, b is the joint width and *L* is the joint overlap length. The analysis assumes the adherends are rigid, and that the adhesive only deforms in shear. In fact, the resultant stress distribution, across and along the bond length is very complex and is dependent on adhesive and adherend properties, and joint geometry [76-80].

Single-lap joint tests are relatively straightforward from the perspective of specimen preparation and testing. However, these tests have recognised limitations for the accurate determination of joint design parameters. The basic mode of loading of the adhesive bond is in shear but, due to the eccentricity of the loading path, the critical end regions of the joint experience complex stress states with peel components. The directions and magnitudes of these stresses depend on numerous factors:

- Applied load and deflection (increasing either leads to an increase in stress);
- Mechanical properties of the adhesive (elastic modulus and yield stress);
- Mechanical stiffness of the adherends (determined by their thickness, elastic modulus and yield stress) - lower stiffness leads to higher peel stresses [4, 81]; and
- Geometry at end of overlap (shape of adherend corners, joint fillet) [76-80].

The geometry of the joint end can influence the location of the point of initiation of failure. Rounded adherend ends and fillets move the stress concentration from the corner of the joint into the fillet and failure may initiate through cohesive rupture at this region. For a single-lap joint, the maximum adhesive shear stress $\tau_{0,\,max}$ and peel stress, $\sigma_{0,\,max}$ can be calculated, assuming pure elastic behaviour, using the following Equations [10].

$$\tau_{0,max} = \frac{\sigma}{8}(1+3k)\sqrt{\frac{8G_a t}{Et_a}} \tag{9}$$

$$\frac{\sigma_{0,max}}{\sigma}\left(\frac{c}{t}\right)^2 = \lambda^2 \frac{k}{2}\frac{sinh(2\lambda) - sin(2\lambda)}{sinh(2\lambda) + sin(2\lambda)} - \lambda k' \frac{cosh(2\lambda) + cos(2\lambda)}{sinh(2\lambda) + sin(2\lambda)} \tag{10}$$

$$\sigma = \frac{P}{t} \tag{11}$$

$$k = \frac{cosh(u_2 c)\ sinh(u_1 L)}{sinh(u_1 L)\ cosh(u_2 c) + 2\sqrt{2}\, cosh(u_1 L)\ sinh(u_2 c)} \tag{12}$$

$$u_1 = 2\sqrt{2} u_2 \tag{13}$$

$$u_2 = \sqrt{\frac{3\sigma(1 - \nu^2)}{2Et^2}} \tag{14}$$

$$k' = kc\sqrt{\frac{3\sigma(1 - \nu^2)}{Et^2}} \tag{15}$$

$$\lambda = \frac{c}{t}\left(\frac{6E_a t}{E t_a}\right)^{1/4} \tag{16}$$

where:

P = load per unit width	G_a = adhesive shear modulus (initial)
L = length of overlap (bond length)	t_a = adhesive layer thickness
t = adherend thickness	E_a = adhesive tensile modulus
E = adherend modulus	ν = adherend Poisson's ratio

Finite element modelling is also used in the interpretation of stress and strain in the adhesive layer in bond joints. FE models allow the incorporation of non-linear properties (e.g., plastic yield), which are not included in analytical models but often play a critical role in the behaviour of joints, in the interpretation of the stress state. Figure 21 shows the effect of different adherend materials on the predicted peel and shear strain values in the standard lap joint specimen.

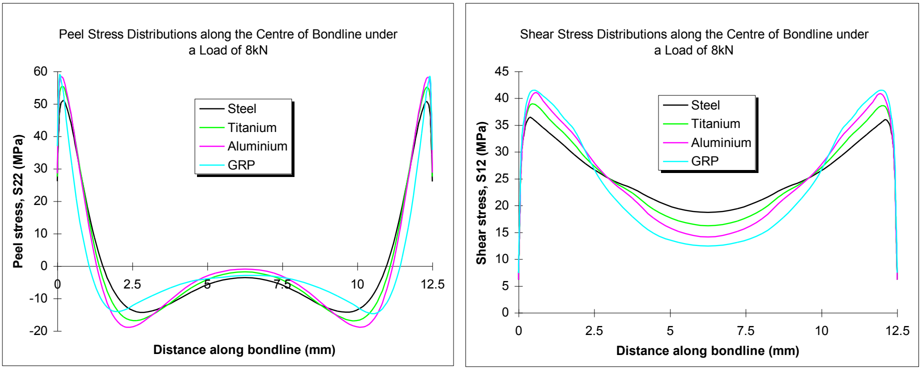


Figure 21. Effect of single lap adherend materials on strain distributions [80]

The single-lap joint provides the user with an “apparent” shear strength value. The apparent shear strength measured with a single-lap specimen cannot be assumed to predict the strength of joints of differing geometry. The thin lap shear test should be used only for the acquisition of qualitative data on adhesive performance.

Thick adherend shear test (TAST)

The thick-adherend shear test (TAST) is probably the most commonly used joint-specimen test for producing material properties under shear [82-85]. The thick and specially profiled adherends in this test produce more uniform stress and strain distributions in the adhesive than are obtained in a lap-shear test [85]. ISO 11003-2 [82] specifies a specimen with an overall length of 110 mm, a width of 25 mm and overlap length of 5 mm. The ISO standard recommends an adherend thickness of 6 mm and a bond-line thickness of 0.5 mm. Slots should be 1.5 mm wide. Load is introduced to the specimen preferably via two 12.7 mm diameter bolt holes. The hole centres are 80 mm apart. Care is needed to ensure that the holes are accurately drilled in the centre of each adherend, since small misalignments can result in unwanted rotation and uneven loading of the joint, thus compromising the test data. The test method is suitable for measuring shear modulus and shear strength of adhesive joints under ambient and hostile environments.

The pre-shaped adherends are more rigid than the bonded plates and reduce the peel stress in the test. However, specimen preparation costs are lower with bonded panels. A miniature TAST specimen, loaded in compression, has been developed at NPL that allows the testing of thinner adherends [86] – see Figure 22.

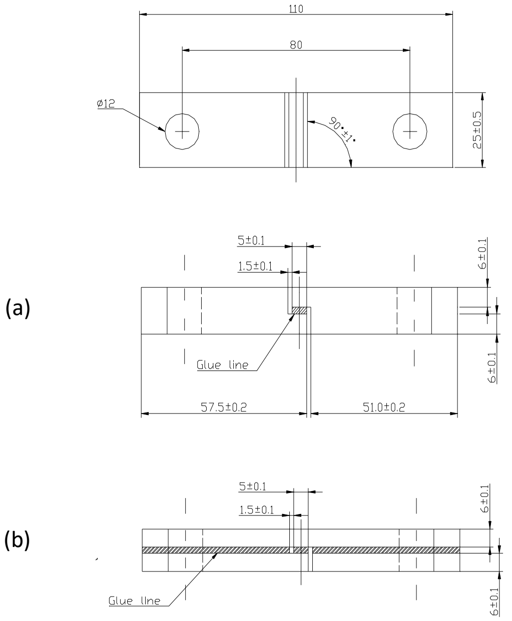


Figure 22. TAST specimen: (a) pre-shaped adherends, (b) bonded adherends (dimensions in mm)

The state of stress is predominantly shear but there are peel stresses at the end of the overlap. Failure occurs at the end of the bond line and is thus sensitive to the geometry of this region. The locus of failure tends to be close to the adherend and, thus, interfacial failure is a possibility. Cracks have been observed to run along the interface [84]. Higher joint extensions can be realised if the corners of the adherends and the spew fillet are profiled to remove stress concentrations. Stresses at the overlap ends are complex and difficult to calculate analytically but FE approaches have been used with some success [84, 85]. The stress state in the miniature specimen will differ from the standard TAST and this should be considered when interpreting results.

Double-lap joint tests

Double-lap joint tests were developed in attempts to eliminate eccentric loading, responsible for bending of the adherends and rotation of the bonded region [87]. Figure 23a shows a schematic representation of the symmetrical double lap joint. The central adherend is twice the thickness of the outer adherends. Although bending is reduced, peel stress at the outer adherends is unavoidable, since the load is applied to the outer adherends, via the adhesive, away from the neutral axis. Tapering the outer adherends can reduce the peel stress. Additional bending can also be introduced when clamping pressure is applied to the double lap end of the specimen, it is recommended that a rigid spacer be used to minimise this effect. The bending moment introduces tensile stresses across the adhesive layer at the free end of the overlap and compressive stresses at the other end. The centre adherend is free from the net bending moment but there is still an element of peel stress at the ends of the overlaps. The stresses at the joint end are sensitive to the mechanical properties of the adherends, the shapes of the joint ends and the geometries of the adhesive fillets.

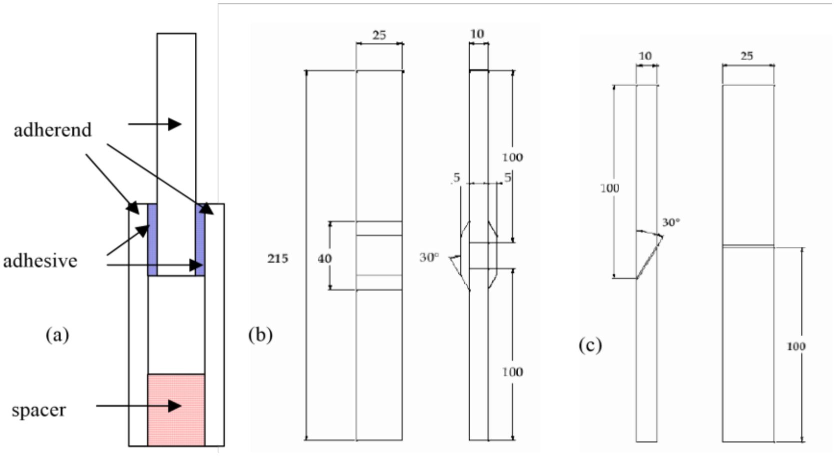


Figure 23. Overlap test geometries (a) double lap, (b) strap joint and (c) scarf

The strap joint consists of two thick adherends butted together. Straps (one for the single strap joint or preferably two for a double strap joint) are then bonded to the faces of the adherends to carry tensile loads in shear (in test specimens the gap between the butt-ends of the central adherends is free of adhesive). This geometry is also described in ASTM D3528 [87] as an alternative specimen to the double lap joint. The strap joint test reduces bending moments since the specimens are loaded through coaxial adherends. The square ended straps generate significant peel forces at the end of the strap. By tapering the straps peel forces at the ends of the adhesive layers can be reduced further.

The tapered strap joint, as shown in Figure 23b, can be used to generate adhesive design data [84, 88]. As with other lap joints, the adhesive in the strap joint is predominantly stressed in shear at the centre of the joint with peel stresses at the end of the overlap. Analyses of the strap joint suggest that a tensile stress concentration is inevitable at the end of the strap. The size and location of the stress concentration depends on the relative stiffness of the adherends and the local geometry at the end of the overlap. The shape of the fillet can determine whether the stress concentration is near the interface with the main adherend or the interface with the strap. This could be used advantageously to characterise the bond strength of an adhesive to either thick sections (central adherends) or thin sections (the straps). The central and strap adherends may be of different materials.

Double lap tests remove some of the disadvantages of the single lap test, but specimens are more expensive to prepare. The inner and outer adherends can be of the different materials and the location of the stress maximum can be controlled through the shape of the outer adherend ends and end fillets. Hence, this specimen has the potential to test the bonding of adhesive systems to materials that are available as either thick or thin adherends. It should be recognised that differences in bending stiffness of adherends could complicate the comparison of data determined for different materials.

Scarf joint tests

The scarf joint, Figure 23c, can be thought of as an ‘angled’ butt joint. When pulled in tension, a mixture of shear and tensile stress is generated in the adhesive layer. The proportions of shear and tension stress can be varied by varying the scarf angle [89]. A scarf angle of 90° to the axis of the specimen is a butt joint where tension dominates. An angle of 0° to the axis of the specimen is a lap joint where shear dominates. Scarf joints with lower scarf angles have higher strengths owing to their greater bonded areas and reduced tensile peel stress. The scarf angle and detailed geometry at the tips of the scarf sections influence the location of the stress maximum, near the interface that initiates failure [89]. The stress state at the stress concentration is complex and it may be difficult to quantify adhesion strength.

Other shear tests

There are many other test methods that impose shear stress on the adhesive layer. The butt and napkin ring torsion tests are known to provide a very uniform shear stress in the adhesive layer with minimal peel stresses [90, 91]. Shear strains to failure determined for adhesives in this test can be much greater than those determined in other tests. Torsion tests require test machines capable of applying loads in torsion and these are less common than tensile test machines. Failures seen in torsion tests appear to be cohesive in the adhesive rather than interfacial.

Pin and collar tests [92, 93] also test adhesives in shear. Specimens are easy to prepare and tests straightforward to perform by pushing the pin from the collar. However, there has been little investigation of the stress distribution in such specimens. A major concern is the possible sensitivity of results to the degree to which the pin is ‘centred’ in the collar.

Joint specimen tests based on bulk specimen shear tests such as the Arcan or Iosipescu tests can also be carried out on adhesives [94]. Failure will tend to initiate at a stress concentration at the end of the bond. The block shear test [95] is another possible method for testing adhesives but has found little favour owing to difficulties in interpreting and correlating results quantitatively.

Peel test methods for adhesives

T-peel tests

The T-peel test [96-98] is commonly used to assess the resistance of adhesive systems to normal force peel loading. The T-peel specimen is shown in Figure 24. The test requires flexible adherends i.e., those that can be bent through 90°. Tests are performed under constant separation speeds in a standard tensile test machine. The measured force-extension curve yields the maximum force and the peeling force (for steady state crack propagation). Peeling force (quoted as load per width) is normally the result quoted. However, work at NPL [98] suggests that the peeling force can be insensitive to environmental degradation and that the maximum force is a much more sensitive indicator of joint performance.

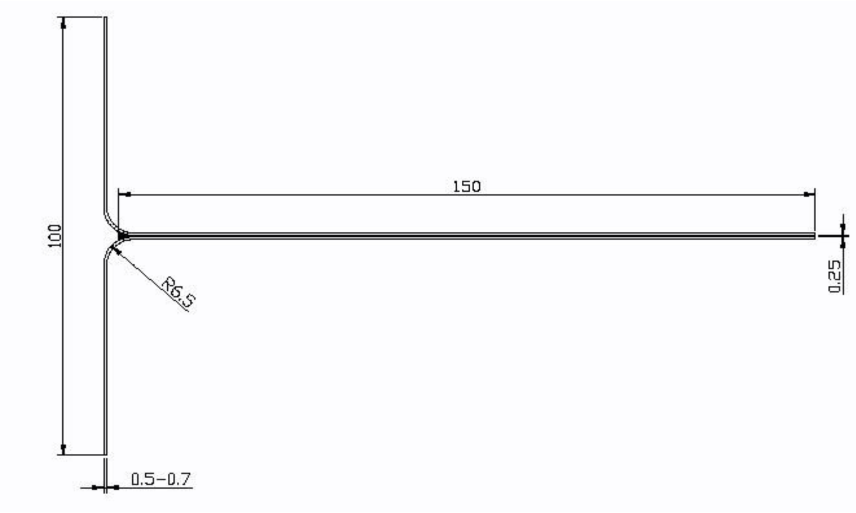


Figure 24. T-peel specimen (dimensions in mm)

The T-peel joint performance depends on joint materials and geometry. Most of the deformation in the test occurs in the adherends. Therefore, the thickness, stiffness, and plastic yield strength of the adherend material have major influences on the test results. The adherends bend during the test, changing the stress distribution. The degree of fillet in the joint influences both the strength and location of initial failure. Fillet size should be carefully controlled during manufacture [3]. The flexibility of the adhesive can alter the location of the stress maximum. With rigid adhesives, such as an epoxy, the stress concentration is at the end of the fillet. In the case of more flexible adhesives, such as a polyurethane [99] or ‘moist’ epoxy [98], the stress concentration is inside the fillet. The locus of failure and propagation route of the crack can vary between the interface and centre of the adhesive layer.

The evidence suggests that the T-peel test is unlikely to provide accurate interfacial strength data for design purposes. The crack propagation may not necessarily be along the interface. The maximum load borne by the joint is sensitive to many factors; in particular the local radius of curvature and peel angle of the adherend near the crack tip, and it is difficult to relate results to a material or interface property.

Flexible to rigid peel tests

There are several peel tests for assessing the bonding of a flexible adherend to a rigid adherend [100-106]. The main differences between the methods are the angles of peel and whether the peel angle remains constant during the test. Most of the energy is dissipated in the adherends rather than the adhesive. Accurate data on the elastic-plastic mechanical properties of both the adhesive and the adherends are important in the analysis of the test data [107-110]. Peel tests tend to be used to a greater extent for characterising flexible adhesives (including pressure sensitive adhesives) than they are for rigid structural adhesives. Peel strength is often quoted on adhesive data sheets. For structural adhesives, the T-peel test is more likely to be quoted than the methods outlined in this section.

The floating roller test method [103-105] determines the peel strength of a flexible-to-rigid bonded assembly. The roller mechanism enables a constant angle of peel to be maintained throughout the test. The test fixture can be adjusted to vary the peel angle. The average load obtained during stable peel is recorded as a characteristic of the bond.

The climbing drum peel test [106] is a standard test used to determine the bonding of sandwich structures. The drum fixture is used to peel the flexible skin from the rigid structure at a constant peel angle. Once again, the average peel load is measured. This method is used mostly in the aerospace industry.

Peel tests, while providing useful comparative data, are unlikely to provide quantitative measures of interface strength. There is no guarantee that the fracture path will be along the interface. Stress distributions depend significantly on local geometrical features such as crack sharpness and peel angle. Other than the aerospace industry, these tests seem to have found little favour in industry for generating data for the design of structural bonds. The conversion between peel load per unit area to stress requires assumptions about the extent of the peel zone that are likely to be inaccurate. Recent developments on the analysis of peel test results concentrate on the determination of fracture toughness properties [111].

Fracture test methods (cleavage – thick sections)

Fracture mechanics tests [13, 83-99] provide information on the growth of a fracture within a material. Stress is intensified at the tip of the crack. The fracture energy is determined from the stress intensity factor, crack length and the mechanical work applied. The quantities determined through fracture mechanics tests are the critical stress intensity factor K_c and the critical strain energy release rate G_c. The stress intensity factor is related to the geometry of the test specimen but K_c and G_c are both material properties. Analysis methods for bulk material fracture tests are well established, particularly for elastic materials. However, techniques for multi-material systems and materials that experience plastic yield are less well established, although theoretical developments are occurring to account for plastic zones [95] and rate/temperature dependence [99].

Fracture mechanics tests have been applied to adhesives. The common test methods are based on the double-cantilevered beam (mode I) and end notch flexure (mode II) tests. Mode I (crack opening) tests impose severe cleavage stresses on bonded joints. Fracture tests require an initial notch or pre-crack. The precise geometry of this notch will influence the results and is a source of uncertainty or variability in the tests. Notch geometry will have more effect on the initiation of crack propagation. Results from the initial part of the test are normally excluded from analyses with G determined from the regions of steady state crack growth.

Fracture tests are commonly performed to assess joint performance in hostile environments and results show sensitivity to the preparation of bonded surfaces. Fracture toughness is recognised as an important adhesive property, contributing to mechanical and impact performance.

Boeing wedge test

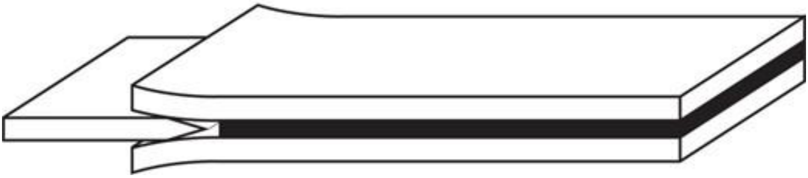


Figure 25. Wedge cleavage test (Boeing Wedge) test

The wedge cleavage test [112, 113], commonly referred to as the Boeing wedge test, uses the introduction of a wedge between two flat surfaces to force the adherends apart and impose cleavage stresses in the region of the crack tip, as shown in Figure 25. The wedge imposes a fixed displacement to the adherends, and the crack opening force is driven by the elastic stored energy in the adherends. Crack length is monitored with time, often using ruled scales attached to the adherends. The wedge test is simple and cheap to perform. No test machine is required since it is self-stressed. The stressed specimen can be exposed to hostile environments and the presence of chemicals at the crack tip can be expected to accelerate degradation much more effectively than in tests where these species must diffuse into the bond layer. Commonly, a crack growth limit is reached within several days making this test attractive for rapidly assessing durability. This test is considered a reliable method for assessing the environmental durability of adherend surface preparations.

Fracture energies *G* can be determined from the crack length *a* wedge displacement *w* adherend modulus *E* and adherend thickness *h*:

$$G = \frac{Ew^2h^3}{16}\left[\frac{3(a+0.6h)^2+h^2}{(a+0.6h)^3+ah^2}\right] \tag{17}$$

The accuracy of the fracture energy determined can be compromised by plastic deformation of the adherends (reducing cleavage forces) and adhesive. This test is not considered particularly accurate for fracture toughness measurements. The driving force depends on wedge insertion distance and the stiffness of the adherends. Although specimens can be manufactured with the initial crack at the interface there is no certainty that the crack will continue to run along the interface. FEA suggests that the regions of maximum strain and stress in the specimen, prior to crack propagation, are close to the interface.

The wedge test has been used to assess the comparative durability of adhesive systems and results suggest that crack growth rates in this simple test can distinguish between different surfaces and surface pre-treatments [69].

Compact tension test

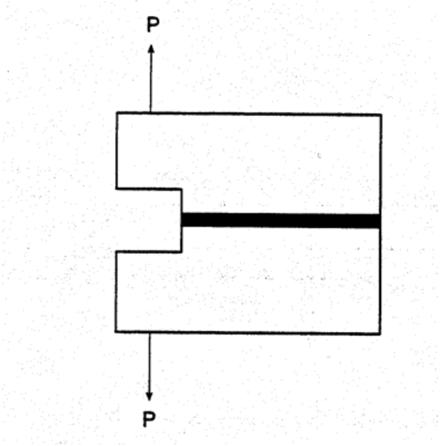


Figure 26. Compact tension test

Compact tension test [114] specimens are manufactured by bonding shaped adherends with the same geometry as the solid compact tension specimen, Figure 26. Specimens are 25 mm wide with a 25 mm long bond line. The depth of adherend is typically 12 mm at either side of the bond line. The specimen is loaded at one end of the bond producing a cleavage force. The test is run at a constant loading rate or crosshead speed until the joint is completely failed. The maximum load is recorded.

Double cantilever beam tests

The double cantilever beam (DCB) test [115, 116], Figure 27, is used to measure the initiation and propagation energy of a Mode I crack. The critical strain energy release rate G_{Ic} depends on crack length and is calculated using:

$$G_{Ic} = \frac{4P^2(3a^2 + h^2)}{Eb^2h^3} \tag{18}$$

where *P* is the applied load, *E* is the Young's modulus of the adherend, *b* is the specimen width, *a* is the crack length and *h* is the adherend thickness. Specimen preparation is straightforward. Thick adherend parts can be bonded and tested directly. Where the beams are formed from thin sheet material end tabs need to be attached to allow the beams to be gripped. The dependence of specimen compliance and strain energy release on crack length adds to the complexity of conducting and analysing this test.

The specimen compliance in the tapered double-cantilevered beam (TDCB) test, Figure 28, is independent of crack length [115, 116]. Compliance *C* is directly related to load *P*, width *b*, adherend modulus *E* and bending moment *m*, and G_{Ic}, proportional to the rate of change of compliance with crack length *a*, is given by:

$$G_{Ic} = \frac{4P^2m}{Eb^2} \tag{19}$$

The taper height is chosen such that *m* is constant with crack length a from the relationship:

$$m = \frac{3a^2}{h^3} + \frac{1}{h} \tag{20}$$

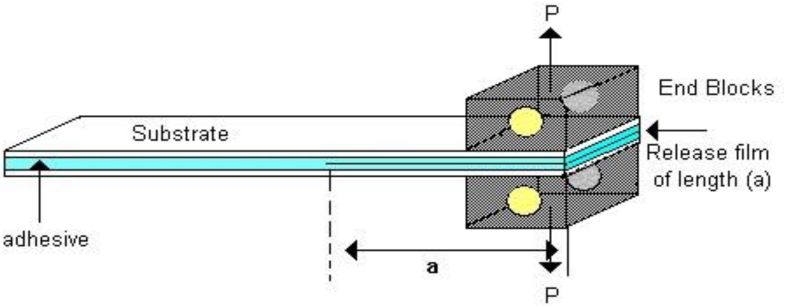


Figure 27. Double cantilever beam (DCB) specimen

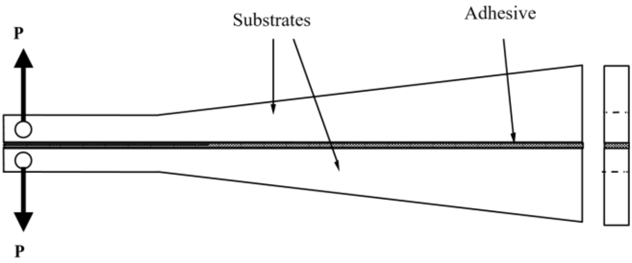


Figure 28. Tapered double cantilever beam (TDCB) specimen

Although TDCB specimen fabrication is more expensive than the DCB specimen the testing is much more routine and, provided that the adherend materials have high yield stress, the adherends should be reusable. Thin adherends can be evaluated using the TDCB test provided they are bonded to rigid components tapered to produce the necessary adherend dimensions. TDCB tests have been used to assess environmental durability. As with all fracture tests, information on the strength of the interface can only be obtained if the crack propagates along a surface.

End notch flexure test

The end notch flexure (ENF) specimen [96] is essentially the double-cantilevered beam specimen loaded in flexure (Figure 29). This test characterises Mode II (in-plane shear) fracture toughness. For small displacements (and negligible transverse shear deformation), strain energy release is calculated using:

$$G_{IIc} = \frac{9P^2a}{16Eb^2h^3} \tag{21}$$

ENF test is being developed as a standard method for Mode II fracture toughness for loading situations where Mode I do not dominate.

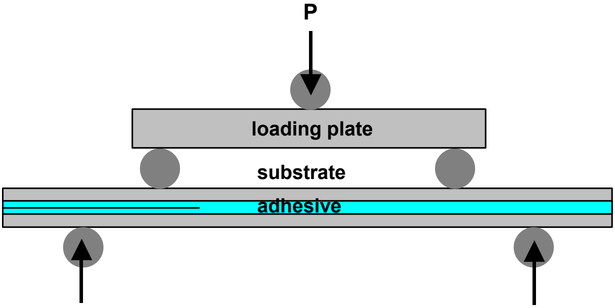


Figure 29. End notched flexure (ENF) test

Tensile test methods

Pull-off tests

Resistance to peel failure can be assessed from tensile pull-off tests. These [69, 118-120] are used widely to evaluate the adhesion of coatings to substrates. This type of test is used to assess adhesives, for example in the construction industry where it is used to determine the development of bond strengths, since it is simple to perform and can be carried out in-situ (either vertically or horizontally) on virtually any type of surface. The pull-off test is attractive as it is quick and simple to perform, requires low cost, commercially available equipment and produces a quantified measure of the adhesive strength from the maximum force applied to the sample. The test method and sample are illustrated in Figure 30.

A test pull stud is bonded to the surface under investigation. The stud is clamped in the test instrument. In some commercial instruments pneumatic pressure is applied, pulling the substrate against a restraining surface, until the stud is pulled off the surface and the maximum pressure is recorded. A calibration look-up table is used to determine the failure stress. Conventional tensile test machines can also be used to perform pull-off tests. The pull off test takes place in a short period of time; maximum load is reached in less than 1 s. The dynamic response of the transducer may introduce measurement errors.

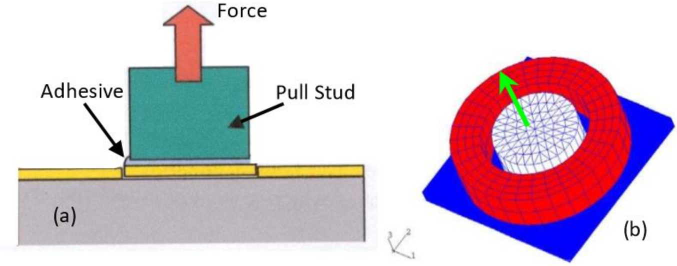


Figure 30. Pull-off test: (a) schematic of substrate; interface and pull stud, (b) FE model showing substrate, stud and 'restraining ring

Sample preparation is straightforward. Adhesive can be applied to either the substrate or the pull stud. These are then pressed together with the pull stud perpendicular to the sample and held in position whilst the samples are cured.

- It is important that the sample should not fail at the interface between the pull stud (or aluminium dolly) and the adhesive. The adhesive and pull stud need to be compatible and the surface of the pull stud prepared to achieve sufficient bond strength. It has been found that gritblasted aluminium provides a suitable surface for epoxy adhesives.

- The surfaces of the pull stud and the substrate should be flat and bonded so that they are parallel and the bond-line thickness is constant throughput the sample. Nonuniform bond thickness will induce cleavage stress in the sample leading to premature failure (see Table 6).
- Stress analysis indicates that the thickness of the adhesive layer has little significant effect on the stress distribution within the adhesive layer [120]. There is therefore little need to control the bond line thickness of the sample beyond ensuring that it is suitable for the type of adhesive being tested. The bond-line thickness can be controlled by mixing a small quantity of glass beads into the adhesive.The adhesive layer thickness should be constant in all regions of the sample.
- A spew fillet is likely to form around the pull stud due to displacement of adhesive from the bond region when pressure is applied. It is recommended that the fillet is not removed as this may damage the bond-line.

The test provides a value of pull-off strength as a ‘stress’ in MPa. This is the applied force divided by the bonded area (calculated from the radius of the pull stud). However, it is recognised that the stress will vary within the bond-line as the substrate will deform during the test. FE stress analysis, Figure 31, shows that the stress in the centre is very low and increases along the radius with the maximum being achieved at the edge of the bond (in the fillet should one be present).

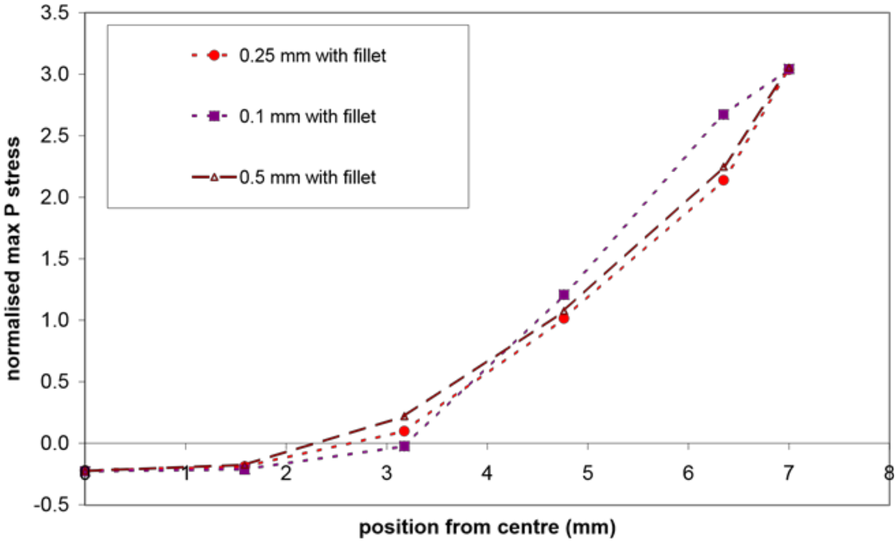


Figure 31. Analysis of maximum principal stress distribution normalised by the average stress in pull-off specimens with different bond-line thickness

The stress distribution in the bond-line depends on the bending stiffness of the substrate. The stress distribution becomes more uniform as the stiffness of the substrate increases. Under equal loads, adhesives bonded to substrates with low bending stiffness will experience higher stresses than they would if bonded to substrates with high bending stiffness. Therefore, care must be taken when comparing the bonding performance of an adhesive on different materials. In situations where the substrate is significantly more flexible than the pull stud (most thinner gauge materials), then the maximum stress is predicted to occur at the interface between the fillet and the adherend. However, as Figure 32 shows, failure is often cohesive within the bond-line, leaving the fillets firmly attached to the substrate.

Figure 32. Pull-off test specimen

Figure 32 also indicates that adherends can be deformed to a significant extent in the pull-off test. A significant amount of cleavage stress will be generated around the rim of the bond-line. Experience of using the pull-off tester suggests that in many cases the sample fails through cohesive failure of the adhesive – the results of the tests therefore provide a minimum value for the adhesion strength (see Table 6). Adhesion failures were associated with poor surface treatments.

Table 6 shows some test results for the pull-off test. The level repeatability in the measurements varies between 10% and 30%. The test shows significant differences between 'good' and 'poor' quality surfaces but is less able to distinguish between similar surfaces. The results show the importance of the care with which adherends are machined on the results – guillotining produces slightly bent adherends that lead to premature failures. Calibration drift may be an issue with this test. It is recommended that a stable and repeatable adhesive-adherend system (identified through testing of multiple batches) is identified and used periodically to check the performance of the test unit.

Surface and Treatment	Adhesive A and Aluminium, Pull-Off Strength (MPa) (Failure Mode: I = interfacial Adhesion, C = Cohesive)	
Aluminium (see Table 3)	**Guillotine/Sawn**	**Spark Eroded**
Mill finish	10.7 ± 0.8 (I)	12.1 ± 4.0 (I)
Light clean	13.0 ± 0.5 (C)	21.6 ± 3.8 (C)
Light clean + anodise	14.0 ± 0.5 (C)	19.6 ± 0.8 (C)
Full clean	14.8 ± 0.4 (C)	18.1 ± 5.4 (C)
Full clean + anodise	14.3 ± 0.7 (C)	24.0 ± 4.8 (C)
Full clean + over anodise	13.3 ± 1.3 (C)	23.6 ± 4.4 (C)
Steel Surface and Treatment	**Adhesive B + Galvanised Steel Pull-Off Strength, MPa**	
Mill finish	13.8 ± 0.9 (C)	
Light clean	5.0 ± 0.9 (I)	
Light clean + anodise	3.4 ± 1.1 (I)	

Table 6. Pull-off test results

Butt joint tests

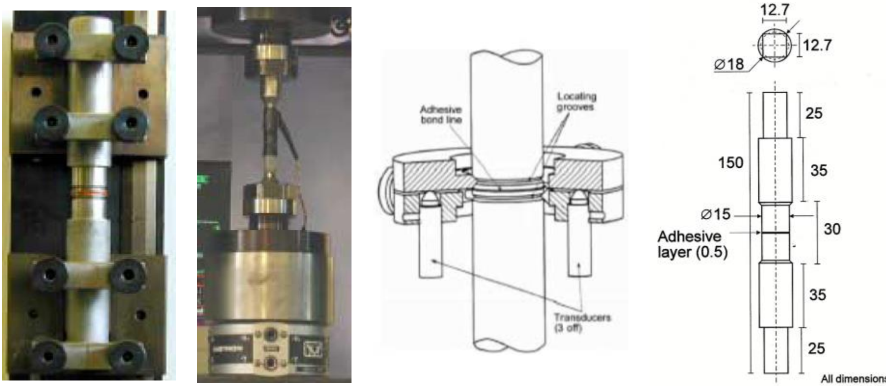
The butt joint test [121-124] is a severe test of an adhesive; specimens are prepared by bonding two rods or bars of equal cross-section together end-on. The joint is pulled in tension to obtain the butt tension strength. Tensile tests on butt-joint specimens have been used with suitable instrumentation to determine elastic properties and plastic property results that serve to check the validity of elastic-plastic models [4]. One use of this test has been to investigate adhesive yield strengths since the adhesive layer experiences high levels of tensile and hydrostatic stress but low shear stresses due to the constraints imposed by the rigid adherends [121]. This severe stress state also applies at the interface, thus providing information on the interface performance. There are several standards for butt joint tests but, as small misalignments can introduce high cleavage stress, it is difficult to obtain reproducible and accurate data unless considerable care is taken.

Accurate alignment of test specimen and loading assembly is critical in this test. The specimen is loaded through collet grips [124] that are rigidly mounted in the test assembly to minimise any lateral deformation of the specimen during loading. Without this constraint to lateral movement, the specimen is observed to bend during plastic deformation causing a non-uniform strain in the adhesive. Alignment is critical at all stages:

- During preparation of the test specimen where the accurately machined cylindrical adherends should be aligned carefully so that they meet precisely to form a straight rod with parallel bonded ends. The axial alignment of the butt joint specimen is closely controlled during manufacture using a precision manufactured V-block assembly jig, Figure 33, to ensure that the sample is bonded end-on. The adherend ends are precisely machined as 20 mm diameter cylinders from 25 mm rods. Slip gauges are used to position adherends and control their separation to obtain the desired bond line thickness (typically 0.5 mm). The specimen is clamped tightly during cure to prevent movement during this process.

- The grips of the test machine should be carefully positioned so that the direction of force application is precisely through the centre of the long axis of the test specimen. An alignment fixture is used to position one grip and enable the specimen to be mounted precisely in the test assembly. Figure 33 shows the fixture and specimen that are used to position the grips. The dummy specimen is strain gauged around the circumference (at equal angular separations) and the strain readings monitored when load is applied. The alignment fixture is adjusted until all the strain readings become equal to within a tight tolerance indicating that the test machine is aligned. Once aligned, the grips are locked rigidly in place and a final check is made of the dummy sample. If this is successful then the dummy sample is removed and test samples can be clamped.
- During tests, the extension at different locations around the circumference of the sample should be monitored to confirm that the loading remains axial. A suitable specimen geometry together with extensometers for the measurement of strain in the adhesive are shown in Figure 33. The specimen consists of two 25 mm diameter, cylindrical adherends that are bonded together at end faces. The v-shape groove in each adherend near the adhesive layer serves to locate an extensometer consisting of two rings with knife-edges on their inner surfaces. Each ring locates in one of the grooves in the test specimen. One of the rings supports three precision displacement transducers that are equally spaced around the ring. The core of each transducer contacts the surface of the second ring. The three individual displacement transducers provide a check on the axiality of the loading during the test. The readings will diverge should the specimen be misaligned. A value for the nominal axial strain in the adhesive under tension is determined from the average displacement divided by the adhesive layer thickness. A small correction can be applied for the contribution from the elastic deformation of the adherends to the total displacement recorded by the extensometer.

The standard butt joint sample consists of cylindrical adherends with flat faces. The sharp, square corners at the rim of the adherends leads to stress concentrations in the outer rim of the adhesive layer where failure will initiate. The sharp rim can be rounded to reduce but not eliminate the stress concentration. FE analyses show the peak stress in the bond-line to be approximately 10% higher than the average stress obtained by dividing the applied force by the bonded area [120]. This stress concentration, although lower in significance than found in many other joint tests, can cause problems with interpretation of results.



Bonding Fixture **Alignment Fixture** **Extensometry** **Butt Joint Specimen**

Figure 33. Tensile butt joint testing

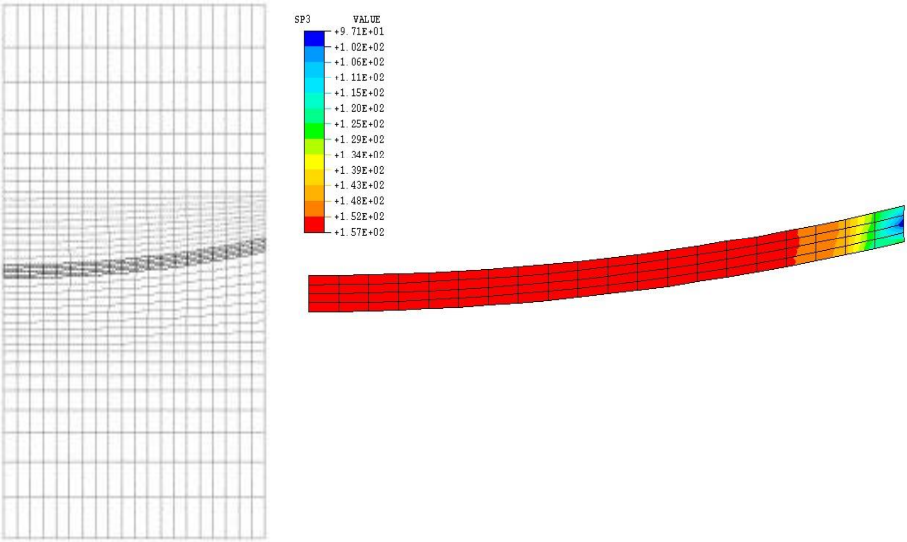


Figure 34. Profiled butt joint bond-line: (left) FE Mesh (rotational symmetry), (right) Maximum principal stress contour plot of the adhesive

Other profiling options, to reduce the stress concentration problem, were investigated with FEA. The most promising profile of those considered was when the butted adherend ends were machined as interlocking concave and convex spherical surfaces with 50 mm radii of curvature – producing a bowl-shaped adhesive layer with constant thickness, Figure 34. The radial distribution of tensile stress distributions predicted in the profiled butt joint specimen is compared the stress in a square ended butt joint in Figure 35. The region of highest stress in the profiled sample extends from the centre of the specimen over most of the adhesive layer. Stress values near the rim are lower. The highest stress predicted is close to the average stress (calculated as 51 MPa from the force divided by the bonded area).

Tensile butt joint tests performed on toughened adhesives using the profiled specimen showed considerable deformation after the adhesive yielded plastically, provided that the test system was correctly aligned (no deviations between individual extensometer readings). If the test system was not perfectly aligned, then the extensometer readings deviated, and the sample failed prematurely through cleavage. The well-aligned tests tended to fail cohesively indicating that the bonding strength exceeded the strength of the adhesive. The maximum load obtainable in these tests was limited by the yielding behaviour of the adhesive rather than failure.

The tensile butt joint test offers, provided sufficient care is taken, many advantages for studying adhesion strengths. However, there are many issues that will prevent widespread use of this technique:

- Tests are expensive to perform as to achieve the good levels of accuracy much care must be taken with sample preparation (including machining) and the setting of the test machine.
- The adherend dimensions must be large enough to produce samples of sufficient length and diameters, which excludes many materials from consideration by this test.

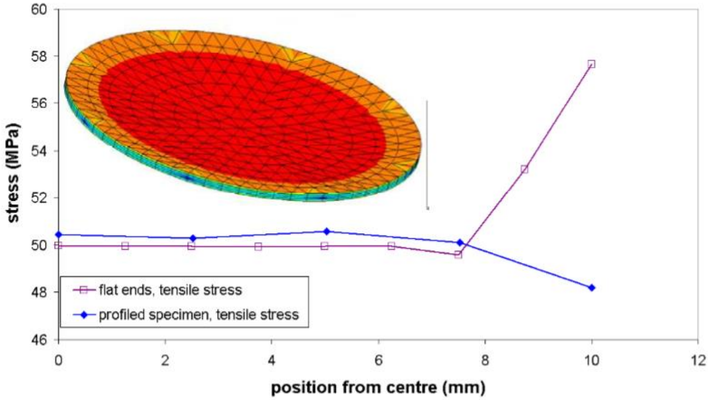


Figure 35. FE stress predictions in the butt joint test

Tensile pull-out tests

In fibre-reinforced polymer composites, the adhesion of the fibres to the resin is a key determinant of the strength of the system. Debonding of fibres from the matrix leads to failure of the composite. Pull-out tests were developed to assess the fibre-matrix adhesion to optimise fibre properties [125, 126]. Micro-pull tests determine the force required to pull embedded fibres from the resin. In order to provide improved interpretation of the data, analytical routines, based on elastic shear lag analyses, were developed to calculate stresses along the interfaces [126]. At first inspection, this test seems promising for adhesives. Specimens would be easier to prepare, and testing would be straightforward with a simple fixture. The largest stresses are at the interface, biasing the test towards interfacial failure and the initiation of failure would be easily identified from the sharp fall in load.

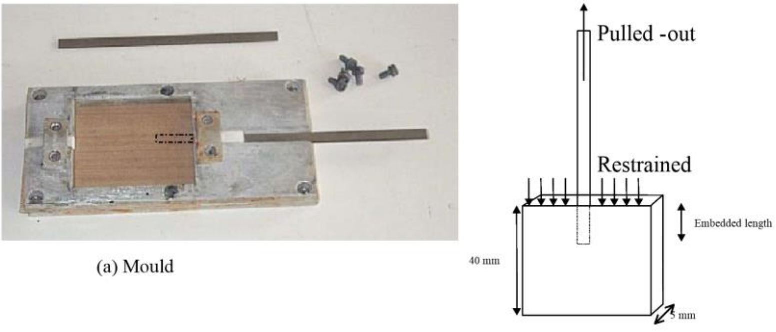
Most of the theoretical work addresses cylindrical fibres but practical adherends are rarely cylindrical. Therefore, an expression for calculating interfacial shear stress (IFSS) values, τ_i, along the surfaces of thin rectangular section shim adherends was developed, based on shear lag calculations for cylindrical fibres [120, 126].

$$\tau_i = n\frac{F}{wh}\frac{cosh[n\,(L_e - x)/r]}{2sinh(nL_e/r)} \tag{23}$$

$$n^2 = \frac{E_a}{E_s(1 + \nu_a)ln\left(\frac{R}{r}\right)} \tag{24}$$

L_e is embedded length, E tensile modulus, ν Poisson's ratio, x distance along shim, w the shim width and h the shim thickness. The subscripts *s* and *a* represent shim and adhesive, respectively. *R* and *r* are equivalent radius values for the adhesive block and shim, respectively. These equivalent radius values were calculated from the equivalent circumferences of the cross-section surfaces.

Pull-out test specimens were prepared in a mould (Figure 36a) where rectangular blocks to the dimensions indicated in Figure 36b could be cast. The channel in the mould keeps the adherend shim perpendicular to the top face of the adhesive block. Sliding the shim to different positions in the channel controls the embedded length. The clamps lock the shim in place once the embedded length has been set. Release film prevents the adhesive bonding to the mould. The sample is held within a clamping jig and the shim pulled from the block in a tensile test machine, Figure 36c. The force-extension curve is recorded, and the point of failure inferred from the point where the load drops, Figure 37.



(a) Mould

(b) Specimen dimensions

(c) Test arrangement

Figure 36. Pull-out specimens

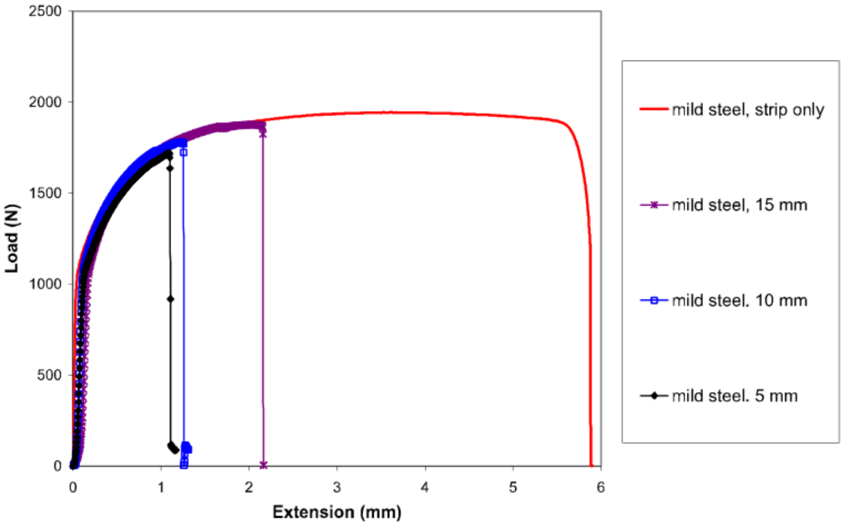


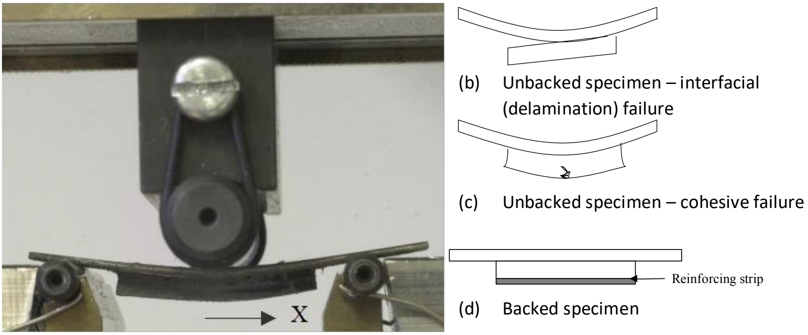
Figure 37. Typical pull-out test force-extension curves

Tests were conducted on a variety of shims and embedded lengths, supported by FEA [120].

- Tensile stresses in the shim sample are large; most of the response in the measured force extension curve shown in Figure 36 is due to plastic yielding of the adherend. In many cases, particularly with longer embedded lengths, the shim failed before the bond.
- The analytical expression is based on a linear elastic analysis and becomes invalid when the shim starts yielding. Hence, the simple closed form analysis of the test results is inaccurate and more complex analysis is required.
  - Failure loads are not directly proportional to embedded length.
  - IFSS values at failure (calculated from failure loads and Equations 23 and 24) are not consistent for different embedded lengths.
  - The IFSS values predicted from Equations 23 and 24 are far lower than those predicted by FEA.
  - FEA suggests similar peak stress levels at failure in each case.
- In some samples the sample failed by cohesive failure of the adhesive block and not failure at the interface.
- Repeatability in the load at failure was poor.
- Manufacturing adhesive blocks of the required thickness (5 mm) can be difficult owing to overheating during exothermic cure.
- The test is limited to adherends that can be prepared as thin samples (e.g., 1 mm thick strips.

Therefore, for the many reasons outlined above, it is very difficult to recommend the pull-out test for determining interfacial adhesion strength.

Flexure test methods



(b) Unbacked specimen – interfacial (delamination) failure

(c) Unbacked specimen – cohesive failure

(d) Backed specimen

(a) Three-point bend test

Figure 38. Three-point bend test, ISO 14679 [127]

When a laminate is flexed, stresses are imposed at the interfaces between different layers and delamination will occur if the laminate strength is exceeded. This principle can be used to assess adhesive joints and coatings. Bend tests are well-established techniques for assessing adhesion of coatings and paints to surfaces [56]. The four-point bend test is often used for thin coatings with the point of failure being detected by visual observation of cracks forming on the coating surface or by acoustic emission techniques. However, four-point bend tests may not produce failure with well-bonded, flexible coatings as peel stresses may not be sufficient to rupture the bond.

The three-point bend test, ISO 14679 [127] has been developed for adhesives. Figure 38a shows a three-point bend specimen and loading jig. A thick rib of adhesive is applied to the middle of the adherend. The adherends used are rectangular with a length of 50 mm and width of 10 mm. These adherends should be thin in comparison with the adhesive rib so that they bend easily and so that the rib of adhesive imparts a reasonable degree of stiffening to the structure. The rib of adhesive applied to the adherend is 25 mm long by 5 mm wide; the adhesive thickness should be approximately 4 mm and uniform, Figure 39.

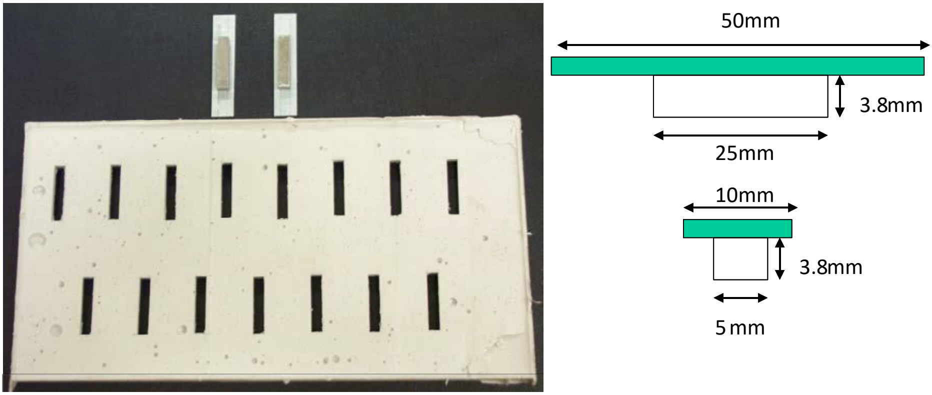


Figure 39. Example of a mould for preparing test specimens

The two lower rollers support the adherend on either side of the rib. The upper roller pushes on the top of the sample. As the sample is depressed, the load increases. The sample bends and stress are generated at the interface between the adhesive and adherend. Delamination should initiate at the end of the bond where the stress is a maximum. The delamination leads to a reduction in force, as shown in Figure 40. In weakly bonded systems or when very low modulus adhesives are tested the drops in load may be very small and difficult to identify.

The test sample should fail by delamination of the adhesive rib from the adherend, Figure 38b, and does so in most cases. However, in some cases, (e.g., flexible adhesives that are well bonded to the adherends) the forces at the interface are insufficient to delaminate and the samples eventually fail through cohesive rupture at the base of the rib (Figure 38c).

A modification to the test, bonding a stiffener strip to the outer edge of the adhesive rib, Figure 38d, can be made to increase the peel stresses at the end on the rib and thus bias failure towards the interface. In these modified samples, failure was observed to initiate at the adherend-rib interface as desired.

Consistent samples can be prepared by using shaped moulds. Moulds can be prepared by casting silicone rubber plaques around solid inserts corresponding to the required dimensions of the sample (plastic beams cut to the required rib dimensions can be glued to spare adherends to produce the inserts). The mould shown in Figure 39 produced samples with a 3.8 mm thick adhesive rib. When preparing the modified specimen thin 25 mm by 5 mm steel plates were inserted into the mould before the adhesive was dispensed. The thickness of the plates was 0.9 mm, and the resulting thickness of the adhesive 'sandwich' layer was 2.9 mm. Adhesive is dispensed into the mould before the 50 mm adherends are placed in position and pressed down using a flat plate. The samples are then cured according to normal cure schedules before they are released from the moulds. Excess adhesive is removed after the samples are cured. One drawback with this method is that the adhesive layer needed is relatively thick and it may be virtually impossible to make representative samples this thick with some types of adhesives, for example anaerobics. Furthermore, if the adhesive cures rapidly large temperature rises may occur in such thick adhesive samples, damaging the material.

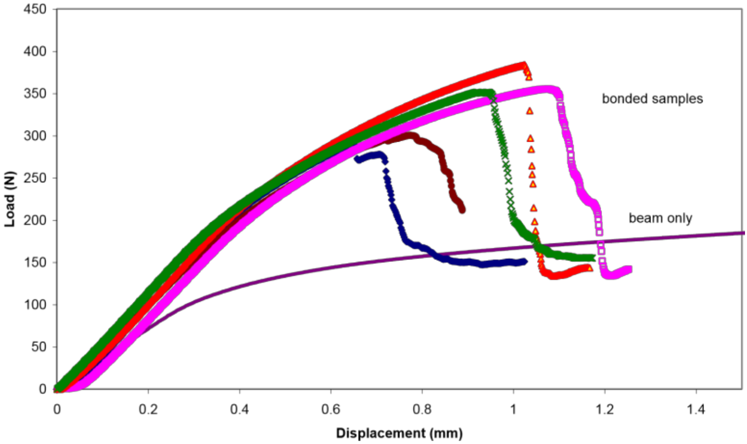


Figure 40. Measured force-extension response

Figure 41 shows typical test results from the 3-point bend test. The maximum force has been determined from the load at the drop in the force extension curve. Equation 25 was used to calculate interfacial shear strength from the maximum load. The results are an average of five specimens per surface treatment/condition; the error bars indicate the experimental scatter, which masks any differences between the treatments. The conclusion is that all the treatments perform similarly and resist degradation under the conditioning used.

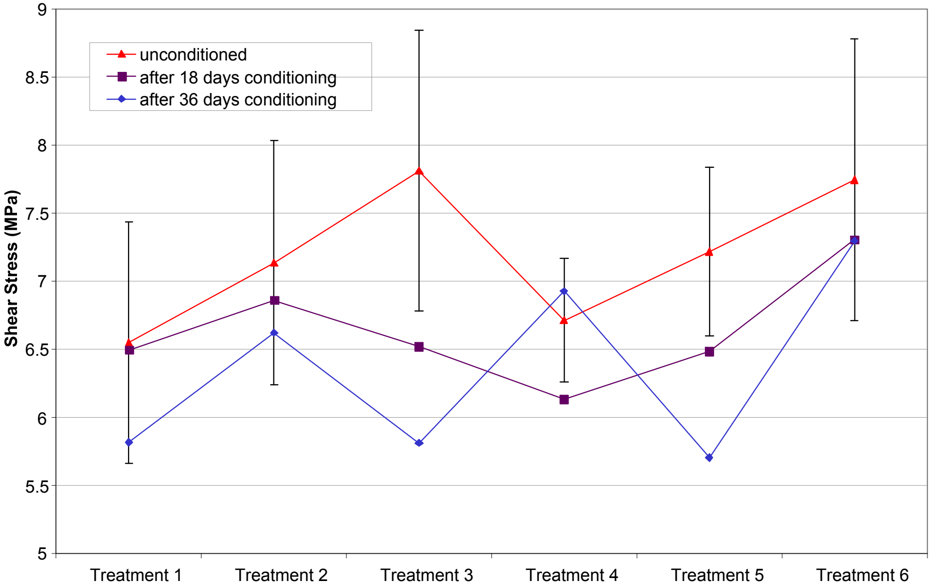


Figure 41. Three-point bend results for aluminium samples (see Table 3 for treatments)

The standard specimen, consisting of adherend and adhesive rib, can be modelled as a two-layer beam. Analytical equations [128] have been developed to calculate the shear stress τ at the interface. These are based on the bending moments and elastic properties of the beam and rib:

$$\tau = \frac{FE_2 b(h-h_1)}{2b_1\left(G_2 - \frac{G_1^2}{G_0}\right)}\left[\frac{h+h_1}{2} - \frac{G_1}{G_0}\right] \tag{25}$$

where

$$G_0 = E_1 b_1 h_1 + E_2 b(h-h_1)$$

$$G_1 = \frac{E_1 b_1 h_1^2}{2} + \frac{E_2 b(h^2 - h_1^2)}{2}$$

$$G_2 = \frac{E_1 b_1 h_1^3}{3} + \frac{E_2 b(h^3 - h_1^3)}{3}$$

The modified specimen, with metal stiffening strip added to the base of the adhesive rib, can be modelled as a 3-layer beam. The analytical calculation for shear stress (Equation 25), modified to account for the presence of the stiffener [120], is:

$$\tau = \frac{FE_2b(h-h_1)}{2b_1\left(G_2-\frac{G_1^2}{G_0}\right)}\left[\frac{h+h_1}{2}-\frac{G_1}{G_0}\right] \tag{26}$$

where

$$G_0 = E_0b_1h_0 + E_1b(h_1-h_0) + E_2b(h-h_1)$$

$$G_1 = \frac{E_0b_1h_0^2}{2} + \frac{E_1b_1(h_1^2-h_0^2)}{2} + \frac{E_2b(h^2-h_1^2)}{2}$$

$$G_2 = \frac{E_0b_1h_0^3}{3} + \frac{E_1b_1(h_1^3-h_0^3)}{3} + \frac{E_2b(h^3-h_1^3)}{3}$$

F = applied force
b = width of the adherend
b_1 = width of the rib (and is equal to the width of the stiffener)
h = total depth of the adherend plus rib (or the + rib + stiffener)
h_0 = depth of the stiffener
h_1 = depth of the rib (or the rib + stiffener)
E_0 = modulus of stiffener
E_1= modulus of the adhesive h total depth of the adherend plus rib (or adherend + rib + stiffener)
E_2 = tensile modulus of the adherend

FE analyses were performed to determine the effects on the test data that would occur through varying various aspects of the test (e.g., rib thickness, fillet shape). These showed that the presence of a curved fillet was to be avoided, as this would move the area of peak stress in the sample from the interface (at the end of the bond-line) into the outer edge of the fillet to produce a cohesive failure. The peak stress in specimens with square ends (both the standard and the backed version) occurs at the end of the bond at the interface and specimens failed through delamination as required. Rib thickness affects the magnitude of stress at the interface for comparable deflections and the relationship is linear over the range of manufacturing variation in the rib thickness [120].

The interfacial shear stress predictions from the FEA results were compared with those experimental results in Figure 41, calculated from Equation 23. The analytical calculations predict significantly less strain the stress at the interface than the FEA calculations. The level of shear stress determined in Figure 41 (ranging from 6 MPa to 8 MPa) is much lower than obtained from lap shear measurements of adhesive on aluminium which are much closer to the FEA prediction of shear stress at failure > 25 MPa. The large underestimate of stress from the analytical calculations is partly due to them calculating the shear stress at the interface in the centre of the beam rather than at the ends of the bond-line. The analytical formulations are based on linear elastic assumptions that are unlikely to be valid at failure.

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Chapter 4

Interpretation of test results

- Mode of failure
- Influence of material properties
- Residual stress
- Detection of failure
- Curing adhesive
- Quality assurance

Mode of failure

The failure of an adhesive bond may occur due to:

- Interfacial failure between the adhesive and the adherend surface, called ‘adhesive failure’;
- Cohesive failure of the adhesive;
- Cohesive failure of the adherend;
- Failure of the surface layer of the adherend.

Inspection of the fracture surface can indicate which mode of failure predominates. Samples should be examined after testing to establish the mode of failure. The absence of adhesive on large regions of the surface indicates adhesive failure. The presence of adhesive on both the surfaces indicates cohesive failure. Both halves of the sample should be examined; the surface of the adhesive can indicate failure of the substrate through the presence of bonded material or help confirm adhesive/cohesive failure from the topography. It should be noted that in instances where the adhesive fails cohesively very close to the adherend surface then the thin adhering layer might not be readily visible without more detailed examination (e.g., by microscopy or spectroscopy).

The value of examining both failure surfaces is demonstrated in Figure 42, which shows fracture surfaces for a flexure test sample and a pull-off test sample. Both substrates are galvanised steel and have had the same surface preparation. Specimens were manufactured and cured on the same day bonded using the same epoxy adhesive. Each specimen was conditioned for 44 days in a hot/wet environment (70 °C/85% relative humidity). The specimens were tested on the same day.

The flexure test sample shows a region of adhesion failure (upper left end) where fracture initiates. The mode of failure then changes to cohesive failure very close to the interface. The location of the change from adhesive to cohesive failure is most obvious on the surface of the adhesive rib. In the centre of the sample is a region where the steel has corroded. There is rust on the corresponding face of the adhesive indicating a weak surface layer that failed rather than the adhesive. In contrast, the pull-off sample shows cohesive failure. There are regions on the substrate where there is adhesive that is obviously still adhering (black patches). The surface of the adhesive is rough in appearance, suggesting that there are few areas where interfacial failure has occurred.

Further information may be obtained by combining imaging and chemical analysis measurements. For example, SEM and EDX measurements made on the flexure sample are shown in Figures 43 and 44. The EDX element maps show regions of enhanced iron and depleted zinc corresponding to the observed locations of the corrosion. The carbon content is low except at the edge of the bonded region, indicating the absence of cohesive failure of the adhesive both in the corroded region and the area of the sample next to the corrosion.

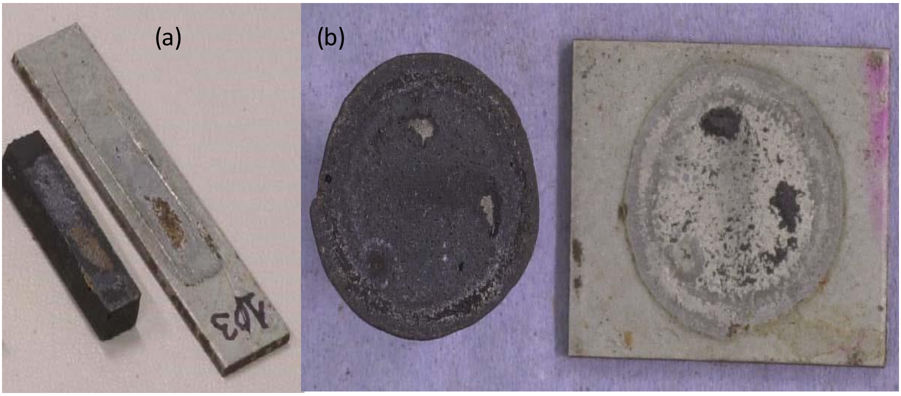

Figure 42. Fracture surfaces of galvanised steel/epoxy test samples (a) three point bend and (b) pull-off

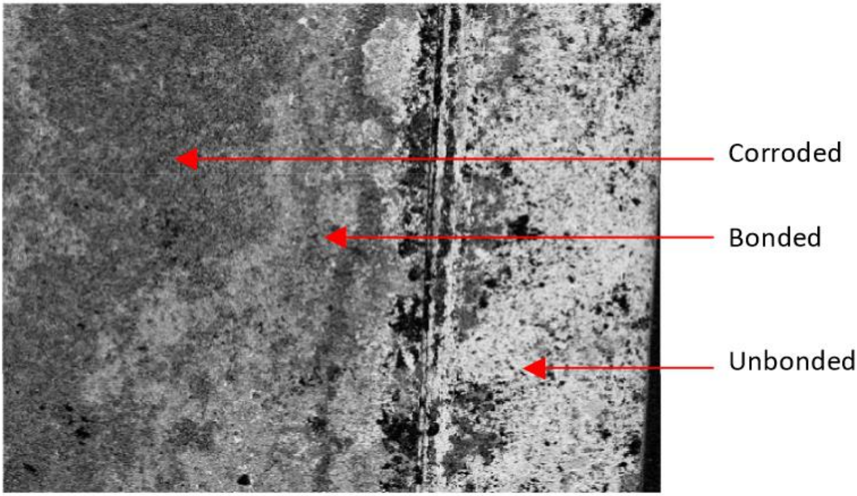

Figure 43. SEM image of sample from Figure 42

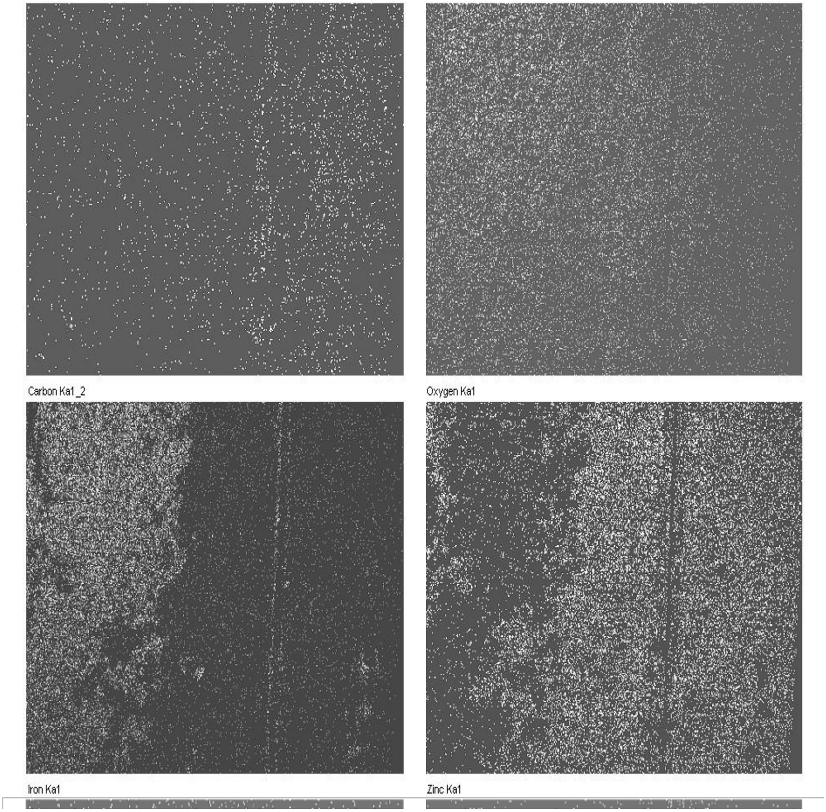

Figure 44. EDX element intensity maps of region shown in Figure 43

Influence of material properties

Plastic yielding

The results of various joint tests can be analysed, using functions derived from linear elastic mechanics, to determine local stress or strain values at the point of failure. However, in strong structural joints very often the plastic yield stress of either the adhesive or the adherend will be exceeded in localised regions before the joint fails. Adherend yielding is most likely to occur with joint tests using thin adherends (e.g., three-point bend, single lap shear or peel tests), thick sections are less likely to yield. When yielding occurs, strain at the location of yield will increase very rapidly compared to strain elsewhere until failure occurs. The linear elastic assumptions behind many of the equations used to analyse the test results will no longer be valid and the true stress or strain at failure will be underestimated as stress localisation at yield cannot be accounted for by linear models.

Degradation of material properties

- Static loads (creep);
- Intermittent loads (fatigue);
- Exposure to chemicals (including moisture);
- Thermal cycling.

The purpose of most adhesive joint testing programmes is to establish the service performance of the bond. Specimens can either be exposed to conditions until they fail (e.g., creep rupture tests) or tested after specific durations to establish the degradation in bond strength. Often conditions harsher than the service conditions (e.g., higher loads, temperatures or chemical concentrations) will be used to accelerate the ageing of the joint. Degradation may occur in the strength of the interface and/or the mechanical properties (modulus, yield stress, cohesive strength) of the adhesive and/or the adherend. Results of durability tests may need to separate these effects in order to provide reliable design data.

Exposure of adhesives to moisture is common. Adhesives will absorb moisture and the absorbed moisture may affect the properties of the adhesive and the interface. Moisture will plasticise epoxy adhesives – reducing the modulus, yield stress and ultimate tensile strength as shown in Figure 45. A fully saturated adhesive joint will have a very muchreduced joint strength due to the reduction in the cohesive strength of the adhesive even if the interface strength is unaffected.

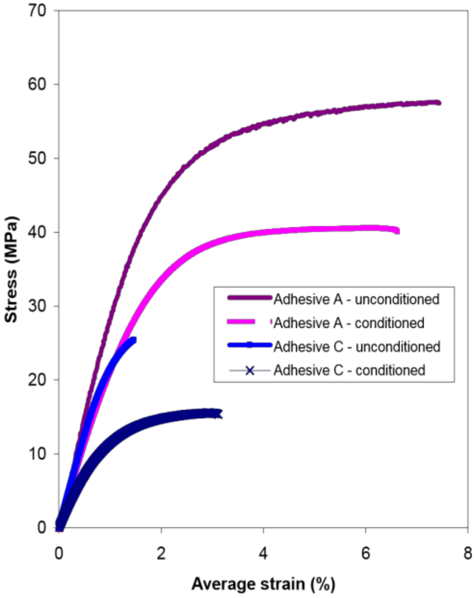

Figure 45. Effect of conditioning on the mechanical properties of the adhesive

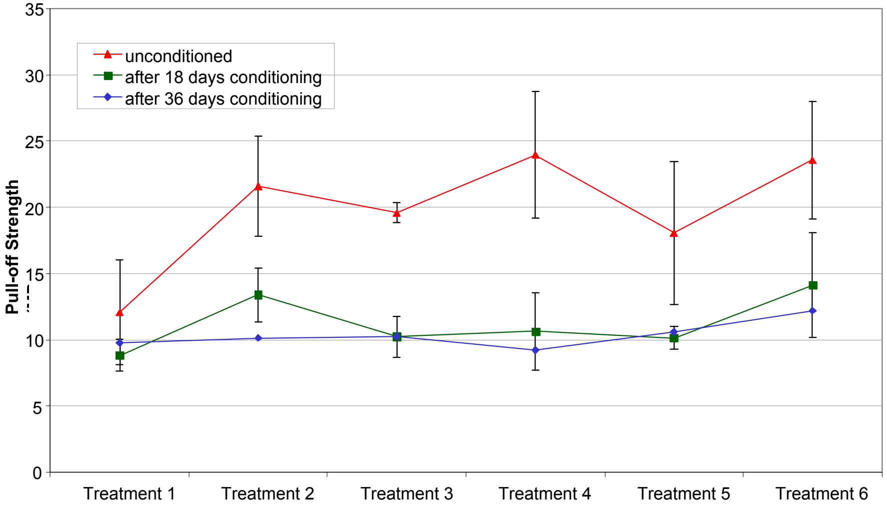

Figure 46. Pull-off test results for aluminium/adhesive A samples (see Table 3 for treatments)

As an example of the effect of degraded material properties on bond strength results, Figure 46 shows pull-off test results for aluminium test samples corresponding to the same surface treatments/conditioning combinations as the results from the three-point bend test presented in Figure 41. The adhesive in each case was adhesive A in Figure 44. In Figure 41, there was little difference between the results of the test combinations. However, the results in Figure 46 show:

- The unconditioned, untreated sample (Teatment 1) has significantly lower bond strength than the unconditioned treated samples (Treatments 2-6), which are comparable. Treatment 1 fails adhesively and the others cohesively (Table 6);
- The strength of the untreated sample is unaffected by conditioning; and
- Conditioning reduces the strengths of Treatments 2-6 until they are comparable with the untreated samples.

Normally, it would be expected that the treated samples would be more environmentally resistant than the untreated samples. FE analyses were made of the conditioned and unconditioned systems (using the appropriate materials properties shown Figure 45). These showed that, at the typical failure loads for the unconditioned (22 MPa) and conditioned (12 MPa) samples, the peak stress values in the adhesive layer were 66 MPa and 41 MPa, respectively – comparable to the tensile strength of the adhesive and suggesting that the loads measured are at the limit of the cohesive strength of the adhesive.

Residual stress

Adhesive joint tests assume that all stress in the adhesive layer is generated by the application of load through the adherends. However, residual stress in the adhesive layer may already be present following manufacture of the specimens. Residual stress may be generated in an adhesive layer through:

- Shrinkage of adhesive during cure; or
- Differences in the residual thermal expansion of the adhesive and adherends generating strain when the joint is cooled from the cure (or post-cure) temperature.

The extent of shrinkage may be determined through dilatometry or internal strain measurements of the adhesive during cure and cooling. These measurements will provide data on both the cure shrinkage and the coefficient of thermal expansion (CTE). Shrinkage whilst the adhesive is still a viscous liquid/paste will have little effect on internal stress generation and, therefore, the technique for monitoring cure shrinkage only needs to be capable of measuring the behaviour following the gel point of the adhesive, where it changes state from viscous liquid to visco-elastic solid.

Optical Fibre Bragg Grating (FBG) strain sensors, Figure 47, can be embedded within adhesives during cure. The strain transfer between the adhesive and the optical fibre alters the spacing of the diffraction grating printed on the fibre. The change in grating spacing changes the wavelength of light reflected through constructive interference. The shift in wavelength is proportional to strain. The strain signal needs to be corrected to account for thermal expansion of the fibre and, therefore, simultaneous temperature measurement is needed.

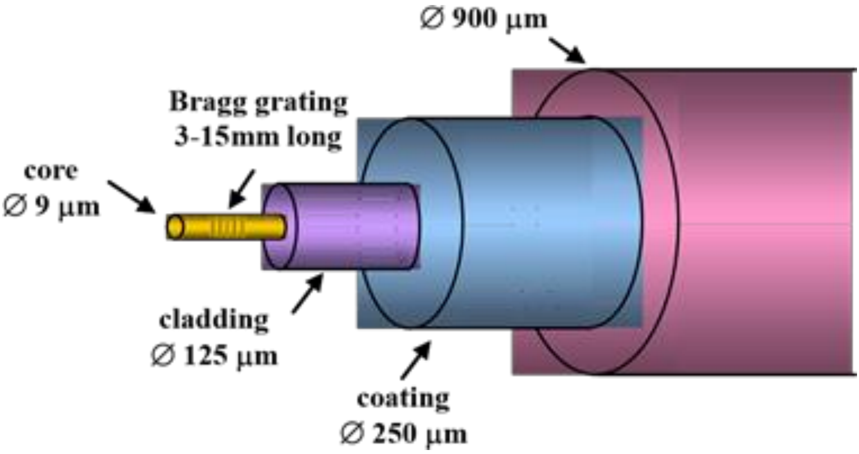

Figure 47. Fibre Bragg grating

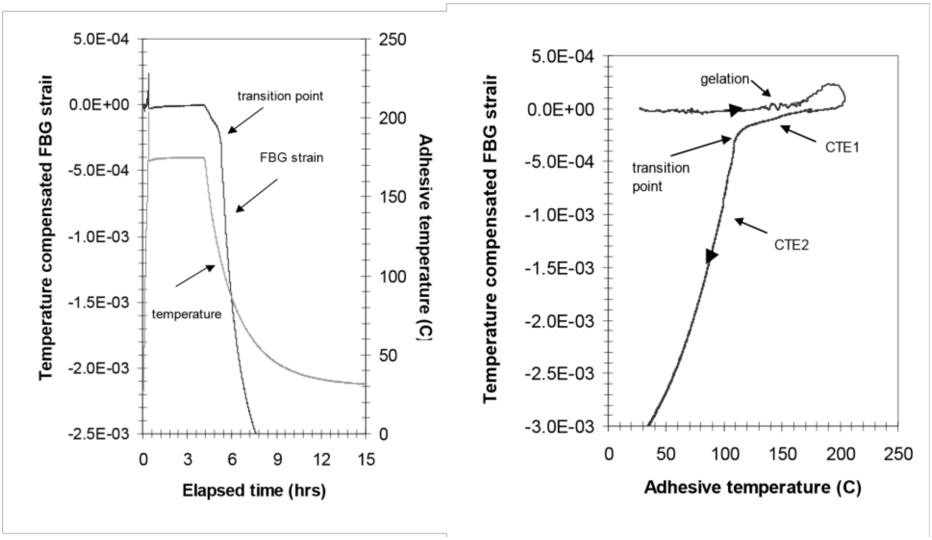

Figure 48. FBG strain measurements of an epoxy adhesive during cure
(a) plotted against time and (b) plotted against temperature

The cured sample was subsequently cycled between room temperature and the cure temperature. On the first cycle a significant reduction was observed in the strain on heating beyond the glass transition temperature. The cooling cycle and subsequent heating/cooling cycle did not exhibit this feature, which was attributed to the relief of internal stress by annealing.

Figure 48 shows FBG measurements on a one-part epoxy adhesive cured at high temperature (180 °C). The temperature profile shows the adhesive heating towards the cure temperature, accelerating as the exothermic cure reaction occurs, settling to the stable cure temperature and then falling as the sample is cooled. The temperature compensated strain is virtually constant, except around the exotherm peak where the temperature compensation may be lagging the actual fibre temperature and then decreases as the adhesive shrinks during cooling. The ‘knee’ in the strain curve occurs at a temperature of ca. 110 °C, consistent with the glass transition temperature determined using dynamic mechanical thermal analysis (DMTA). The lack of any change in strain during the curing period indicates a very low cure shrinkage, which is expected for a filled thermoplastic. The CTE of the adhesive is higher than most metals and, therefore, on cooling the adherends will shrink less than the adhesive. This will lead to tensile stress in the adhesive, as the adherends will generally be stiffer than the adhesive and constrain it from full shrinkage.

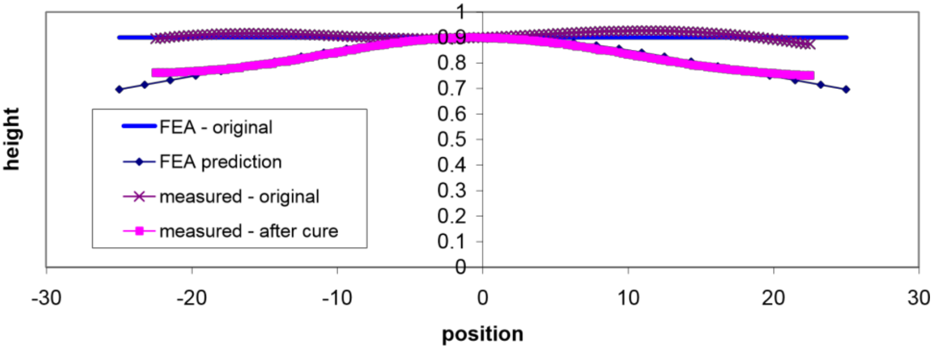

Figure 49. Residual stress induced deformation of a thin three-point bend adherend

Figure 49 illustrates the effect that residual stress can have on test samples. The profile of an adherend for the three-point bend test was measured before and after the adhesive was cured. Additionally, an FE analysis was run to predict the effect of thermal shrinkage on the sample deflection and the internal stress. The deflection of the thin adherend is measurable and agrees well with the FE prediction. The stress analyses predict that the peak residual stress in the sample is approximately 20 MPa, which is a significant proportion of the 60 MPa tensile strength of the adhesive. The residual stress is greatest in the centre of this sample and, hence, does not contribute significantly to the region of maximum stress at the end of the bond where failure initiates.

It is not only residual stress in the adhesive that may affect the performance of the adhesive joint. Stress may be induced at the surface of the adherend, which could damage any surface layer or coating and weaken the bond. For example, the large residual stress transferred to the surface of the sample may explain the location of the corrosion observed in Figure 42a. The tensile stress may have ‘opened’ the zinc surface, creating higher porosity where water could reach the steel, and damaged the protective capability of the zinc layer.

Detection of failure

An important output of an investigation into the adhesion strength of an adhesive joint is the identification of the point where failure initiates. Work on testing adhesive joints shows that, in many cases, joint failure initiates before the ultimate extension or load of the joint is reached [89, 99]. Designing for a long-term service life requires that joints never be loaded to the extent that these failures can initiate (whether through cohesive rupture or adhesive failure). Therefore, identification of the failure initiation point is a critical part of any joint test. By knowing this point, analyses can be performed to calculate stress and strain distributions immediately prior to the initiation of the crack. At the more easily identified point of maximum load in the test, a fracture will be present in the bond that will introduce inaccuracies in any analyses.

Shape of the force-extension curve

The formation of a crack will result in a redistribution of load and, in principle, failure could be detected from a change in the shape of the measured force-extension curve at the point of crack initiation. The slope of the loading curve should fall as a crack forms because the load bearing area of the bond is reduced. In many tests, such as the three-point bend, the initiation of fracture seems to be readily apparent (e.g., Figure 39). However, in other tests the joint stiffness reduces as load is applied, for example as a toughened adhesive yields plastically. In these situations, particularly if crack growth is slow, it may be very difficult to distinguish the effects due to crack formation from those due to yield. Finite element predictions may be used to establish the load at which the measured force-extension response of a joint deviates from the predicted behaviour. However, for these analyses to have any reliability, the models of the material properties and boundary conditions need to be extremely accurate.

Visual inspection

It should to be possible to detect a crack forming and growing by viewing the bond. Obviously this method is restricted to the edge of the bond and cracks forming in the interior of the bond-line will not be visible until the crack has grown to the edge of the joint. High magnification of the region of the joint where failure is expected would be required to view the beginning of a crack. Photography or video recordings could be used to record the formation and growth of the crack [89, 99]. These would need to be referenced to the force-extension or stress-strain curves for identification of the conditions generating the fracture. Synchronisation of the time index of the test data and the photographs is one mechanism through which the data could be analysed.

There are can be difficulties with visual detection of cracks particularly where heterogeneity and surface structures at the joint edge can mask the presence of a crack until it has grown somewhat. The edges of the adhesive bond could be polished to reduce these effects and a penetrant dye used to improve the contrast between the crack and the surface. Tests need to be performed at slow strain rates in order to capture the crack growth meaningfully, unless high-speed photography facilities are available. However, a fast propagating crack is likely to be associated with sudden, catastrophic failure of the joint – the situation where the maximum load is most likely to correspond to the load for the initiation of failure.

Acoustic emission of fracture energy

The fracture of an elastic material or bond is accompanied by a release of energy. A portion of this energy will be sound energy. Thus, fracture can be heard, a loud crack occurs as a brittle adhesive bond fails due to the large amount of acoustic energy emitted by the fast propagating crack. The sound intensity emitted by the initiation of a slow propagating crack in a tough adhesive is low and may be difficult to detect aurally above any background noise. Sensitive acoustic transducers placed near the bond line should detect acoustic emission from a crack forming. Analysis of the acoustic signal may yield additional information on the crack location and growth rate. Acoustic emission is a technique being applied to many systems (e.g., composites, coatings) as a research and structural monitoring tool. The technique relies on the operator having sufficient experience to be able to identify particular defect types from the AE data. The technique is essentially a laboratory tool, however, AE is used for proof and qualification tests of pressure vessels prior to installation and routine inservice inspection.

Strain mapping techniques

The initiation of a crack will alter the distribution of strain in its immediate vicinity. Local surface strain measurements should give an indication of the initiation of a fracture, even if the fracture originates within the bulk of the adhesive.

Strain mapping can be carried out using a variety of techniques. Modern fast computing technology allows for strain mapping through techniques involving image correlation of surface features, such as speckle interference, fringe patterns bonded to the surface (e.g., Moiré [129]) or natural patterns on the surface of interest. The absolute accuracy of strain maps in small regions of critical stress may be questionable but the initiation of a crack should have sufficient effects on the strain map to be readily apparent.

Internal strains can be characterised through Raman microscopy provided that the materials are transparent to the wavelengths used. Stretching a molecular bond alters its characteristic vibrational properties. With a suitable calibration curve, Raman spectroscopy can be used to determine strains in local regions [126]. X-ray diffraction can characterise strain in metal adherends close to the adhesive interface. These strains can be used to calculate stress in the adherend close the interface and, by inference, provide information on the interface [130]. Photoelasticity measurements can also be applied where one of the adherends is transparent [131].

The formation of a crack at the adhesive-adherend interface will relieve stress locally and thus reduce the strain in the adherend. These changes in strain can be detected using the strain mapping methods outlined above. However, where the adherends are rigid, strain gauges can be attached to the adherends to monitor strain. This technique is known as ‘back face strain gauging’ and can be a cost effective monitoring method. Strain gauges will also enable the identification of the point of plastic yielding of the adherends, which is a likely precursor to failure of the joint.

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Chapter 5

Concluding remarks

Potential measurement methods for characterising and quantifying the interfacial adhesion strength properties that are key to the strength of materials systems have been discussed in this review. There is, at present, no consensus as to best practice in considering the interface in a design or quantifying the adhesion strength in a structure. Industry tends to assume a perfect interface when designing adhesive bonds. This is due to both the complexity of models needed to include the interface and the lack of reliable data.

Tests for adhesive joints, fracture toughness and engineering coatings that can provide information on the interface strength have been described. Although there is a plethora of methods available, few of them are capable of providing good quality quantitative data that are readily usable in design calculations. Analysis of test results presents problems as there are difficulties in defining the state of stress, strain or fracture energy close to the interface due to the complex stress distributions that are difficult to model accurately (particularly when material responses are non-linear) and the possibility of material properties close to the interface being different to those of the bulk material. In many situations it may be sufficient to perform qualitative ranking studies to determine the most effective adhesive/adherend/surface treatment system within a specified set of loading and service condition variables.

There are few joint tests that are applicable to both thick and thin adherends. Obviously, thick adherends can be machined to the dimensions required for tests but this increases the cost of testing. The results from lap joint tests are difficult to interpret owing to the complex distribution of peel and shear stresses near the end of the overlap. Joints with higher stiffness (due to thicker adherends) such as the thick adherend shear, double lap or strap joint ought to give more reliable results. The tapered strap joint offers the possibility of inducing failure at either the thick, central adherend or the thin straps depending on the joint geometries chosen. This could enable the evaluation of both thin and thick section bonds using the same test configuration.

Peel tests can provide information on bond strengths but these are difficult to relate to a general design situation as converting load per unit width to stress requires making assumptions about the size of the peeling zone. Often fracture in peel tests occurs in the adhesive layer rather than at the interface. Fracture toughness tests also offer the capability of characterising the interface. The wedge test is simple and cheap to perform but, whilst well suited for comparing the durability performance of different surface treatments, does not provide accurate data on surface fracture energies. The double-cantilevered beam tests (DCB and TDCB) provide more accurate data. A possible limitation is that fracture may run into the adhesive rather than along the interface.

The pull-off and three-point bend tests are simple tests, suitable for screening purposes, which appear to maximise stresses at the interface between the adhesive and substrate. These stress distributions are complex and although analysis techniques have been developed to interpret the results the accuracy of these is open to question. The butt tension test can provide good quality quantitative data but great care needs to be taken to produce reliable results.

The conditions causing initiation of failure are critical in the interpretation of test results and for the safe design of bonded structures. Techniques for detecting the start of a fracture need to be utilised when testing the material systems in order to maximise the usefulness of the data obtained. Inspection of the fracture surfaces to determine the mode of failure, even if only by visual inspection, needs to be done to aid the understanding of results. The cohesive strength of the adhesive and adherends needs to be considered in the interpretation of test results. It is possible that what appears to be a degradation of interface performance on conditioning may be a consequence of reduction in the mechanical properties of the materials involved.

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Appendix 1

Adhesion

Mechanisms of adhesion

The term adhesive is used to describe a polymeric material that bonds two surfaces or components together to form a joint, particularly one that will bear load. A true adhesive bond to a surface is formed through inter-molecular attraction between molecules in intimate contact. Bonds form due to (in order of increasing bond strength):

- Van der Waals attractive forces
- Polar interactions and hydrogen bonding
- Covalent, chemical bonding

The primary mechanisms involved in the development of an efficient bond are:

Mechanical adhesion

Mechanical adhesion is related to the degree of roughness and as a consequence friction of the adherend surface. A certain amount of bonding can be expected purely from the mechanical interlocking of the two contacting surfaces, increasing the total surface area available for chemical bonding and creating a convoluted failure path where the adhesive penetrates crevices on the adherend surface. Although the tensile strength of the bond can depend on the re-entrant angles on the adherend surface, shear strength increases significantly with increased roughness.

Wetting

Wetting out refers to the ability of an adhesive to flow and cover a surface to maximise the contact area and the attractive forces between the adhesive and bonding surface. If sufficiently intimate contact is achieved between the adherend and adhesive a physical interaction develops between the atoms of the two surfaces, which results in wetting. Formation of a physical bond results from highly localised intermolecular forces. Adhesives that have surface energies less than that of the adherend will readily wet the surface and yield good bonds. Wetting may be due to acid-base interactions, weak hydrogen bonding or Van der Waals forces (dipole-dipole and dispersion forces). The extent of wetting depends on the differences in surface free energies of the solid, liquid and subsequent interface.

Chemical bonding

Chemical bonding is the formation of a stable linkage by the reaction between a functional grouping on the adherend surface and a compatible group in the adhesive. Adherend surfaces are usually given surface treatments (e.g., chemical etching, corona discharge and plasma treatments) to create compatible groups. These treatments serve to increase the concentration of oxygen and nitrogen containing functional groups on the adherend surface since these species are considered to enhance adhesion by encouraging the formation of strong covalent or hydrogen bonds.

In thermodynamic terms, to break an adhesive bond energy must be applied to the system such that the input energy (ΔE) is greater than the free energy of the two new surfaces created less the interfacial energy.

$$\Delta E \geq W_A + W_S - W_{AS}$$

where W_A is the surface energy of the adhesive, W_S is the surface energy of the substrate, W_{AS} is the interaction energy.

In theory, all bond strengths could be calculated from these surface energy properties. Wettability (and contact angle) measurement techniques enable determination of surface energies resolved into dispersive and polar terms [132-135] and with the correct choice of probe liquid acidic and basic properties of the surfaces can be quantified. Unfortunately, the surface energy approach has only limited application to predicting the strength of a joint since, as mentioned above, true interfacial failure is unlikely. Further complications arise since the contact area, due to roughness at a molecular scale, is difficult to determine although adsorption techniques have been used [136]. Furthermore, the surface energy approach ignores any direct chemical interaction between the adhesive and adherend and also fails to consider any changes in the composition of the surface layers due to the presence of the second layer. Wettability is therefore a useful method for assessing suitability to bond but would be difficult to relate directly to bond strength. Phenomena such as mechanical keying often provide enhanced bond strengths to the physico-chemical bonds.

An alternative technique for characterising energies of interaction between surfaces is the determination of detachment forces and contact radius using sensitive surface force apparatus [137]. Highly sensitive force measurement systems enable the measurement of extremely small adhesion forces. The measurements require that the surfaces are molecularly smooth. This is generally achieved through the use of cleaved mica sheet substrates. Monolayers of polymer or surfactant can be deposited on the mica surface and interactions measured directly. This type of measurement can yield accurate adhesion strengths but is limited in the types of materials that can be studied through the requirement of molecularly smooth surfaces.

Workers in the adhesives field, such as Sharpe [138] and Bikerman [132], have argued that the statistical improbability of a fracture propagating solely along a molecularly rough surface means that true interfacial failure never occurs. Thus, the phenomenon of ‘interfacial failure’ is more accurately described as near surface failure. However, other authors [133] have argued that failure at the interface can be thermodynamically favoured and that the surface energies can be correlated with bond strength with polar components of the surface energies playing a critical role. Whatever the terminology employed, understanding the behaviour of interfaces throws up many challenges.

Stresses in the region of the interface are difficult to determine accurately owing to the difficulty in modelling load transfer between dissimilar materials. The properties of surfaces may differ both chemically and physically from those of the bulk material (e.g., oxide layers on metals). Furthermore, there is some evidence that the proximity of another surface may further modify the properties of interface layers of the adhesive through mechanisms such as molecular re-orientation, preferential diffusion/adsorption of components or altered chemical reactions (e.g., through catalysis or differences in the thermal history during cure). The concept of an interphase region has been used to describe material characteristics near an interface [139, 140]. The properties of the adhesive in this region can be significantly different to the bulk adhesive [139-142]. The differences between bulk and interface properties depend on the adhesive, surface material and surface preparation. Substantially higher modulus values have been reported for the near surface material, attributed to the transfer of materials from the surface oxide layers into the adhesive [139] as have decreased modulus values near the interface [140-142], thought to be due to incomplete cure and residual stresses in the near interface region. The extents of the interfacial zones in the adhesive layer have been estimated as relatively thick (greater than 0.1 mm) in comparison to bond thickness [132, 139-142]. The keying of adhesive into surface roughness features can also be considered as forming an interphase region whose properties affect the mechanical performance of the joint [143]. In this Guide the term ‘interface’ is used to encompass the true interface, the interphase and the near surface area.

Appendix 2

Joint preparation and testing

Introduction

This Appendix summarises good practice in adhesive joint preparation and testing which was originally presented in Good Practice Guide No. 47 [3].

Handling and storage of adhesives and adherends

A number of issues relating to the handling and storage of adhesives and adherends need to be considered as many of these factors can affect the performance of the material. Issues to be considered include:

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

Handling materials (particularly 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 of adhesives

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 humidities above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [8]. 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 in regard to storage 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 following points should be considered for the storage (and transport of adhesives):

- 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.
- Repeated exposure of the adhesive is a frequent cause of adhesive ageing and deterioration.
- 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). Containers that have been opened must be sealed immediately after use.

- Adhesives should not be stored below freezing unless recommended by the manufacturer. In some systems components of the adhesive may phase separate at sub-zero temperatures, degrading the adhesive performance.
- Exposure to repeated freeze/thaw cycling could limit the shelf life of an adhesive stored at low temperature.
- 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.
- A refrigerated adhesive may be used several times during the shelf life of the product. It is advisable to take samples of the product under the storage conditions. However, to handle the adhesive, this may not always be possible. When the adhesive is taken from a sub-ambient (often sub-zero) temperature and allowed to warm, the amount of time at ambient for these adhesives should be kept to a minimum (advice on the length of time can be obtained from the adhesive manufacturer). 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.
- 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.

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. Temperature should be monitored to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. 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.

Handling and storage of adherends

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 humidities above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [8]. The overall effect is to reduce the adhesive performance and induce premature failure of adhesively bonded joints.

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.

Preparation of adherends

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 section 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. Adherend surfaces, unless specified otherwise, should be prepared according to ISO 4588 [144] for metals and ISO 2818 [145] for polymer matrix composites (PMCs). Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e. adherend bending)

- 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.

Many substrates (e.g., fibre-reinforced polymer composites) or surfaces (e.g., porous anodised films) will absorb moisture from the atmosphere. The presence of moisture can interfere with bond formation and may adversely affect the properties of the adhesive during the cure process. As a result, the joint strength may be compromised. Moisture released from the substrates 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 G_c of a rubber toughened epoxy adhesive by a factor of 10 [146]. 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.

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. There are many procedures available – further information can be found in “Guide to The Structural Use of Adhesives” produced by The Institution of Structural Engineers [13] and specific treatments are defined in various standards, e.g., BS 7079 [14], BS EN 12768 [15], ASTM D 2651 [16], ASTM D 2093 [17] and BS EN 1840 [18] – see also [19-22]. 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.

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 fixtures

A bonding fixture is recommended to ensure correct bond length (e.g., 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 Figure A2.1 [147]. This 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 six 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.

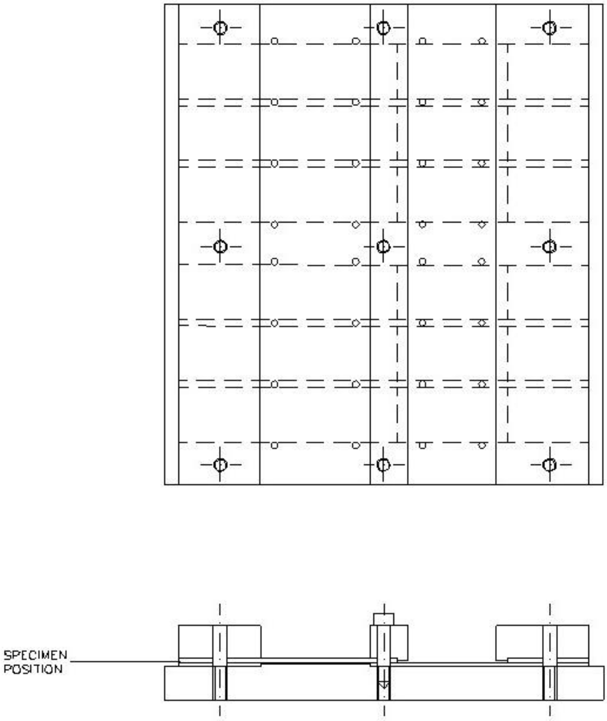

Figure A2.1. Single-lap joint bonding fixture (accommodates six specimens)

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. Care must be taken to avoid contaminating bonding surfaces with mould release agent. Where possible solid films should be used to eliminate the risk of contamination.

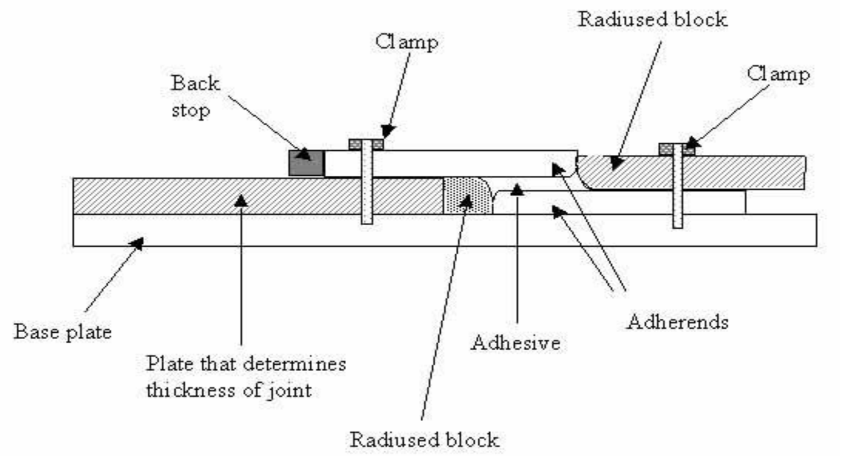

Figure A2.2. Fixture for controlling single-lap joint adhesive fillet

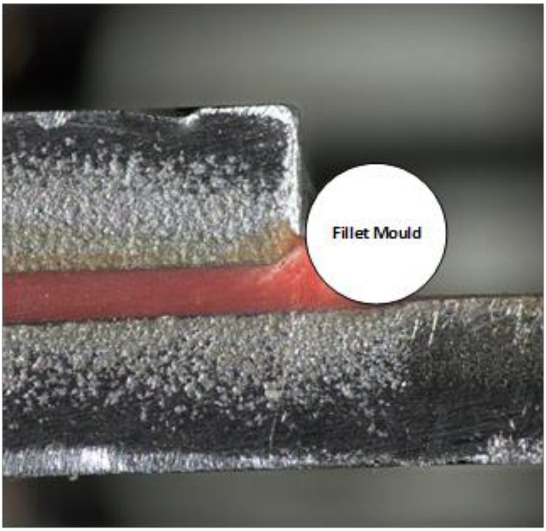

Figure A2.3. Single-lap joint with a concave fillet

Adhesive fillets

Adhesive fillets can have a significant effect on the performance of an adhesive joint. The size and shape of these fillets should be controlled carefully to ensure repeatable results from tests. Fillets tend to reduce stress concentrations formed at the bond-line ends and therefore increase the “apparent” shear strength of joints, compared with square-ended bond-lines. 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.

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 A2.2 or a special tool shaped to fit within the bonded joint; as shown in Figure A2.3. The tool can be held in place using heat resistant tape. Figure A2.4 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., film adhesives). The high viscosity associated with highly filled 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 2 mm, 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. These additional features will add considerably to the costs of specimen manufacture.

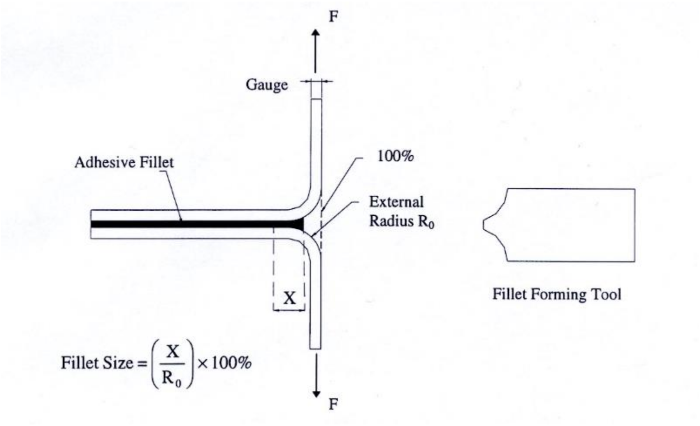

Figure A2.4. Tool for controlling fillet size in T-peel specimens

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),
- Thin wire spacers (e.g., stainless steel) inserted between the adherends 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), or
- Ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1 wt. % by mass). 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.
- Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness.

Controlling the bond-line thickness of very viscous adhesives is difficult due to the lack of flow. The preferred method is to use thin wire spacers. However, this method of bond-line control is not always reliable.

Dispensing of adhesives

The adhesive should be carefully dispensed to ensure that the correct volume is used to avoid underfilling the joint (leaving gaps that weaken the joint) or overfilling the joint (and having to remove the excess).

Multi-part adhesives should be thoroughly mixed in the correct ratio before being applied to the adhesive joint. Often material is supplied in twin pack cartridges and dispensed through mixing nozzles, which provides good control over mixing. However, if the parts require mixing by hand, then the two parts should be weighed out before mixing to ensure control of the mix ratio. The two parts of the adhesive are normally different colours and complete mixing is usually indicated by a uniform colour with no streaks. The period of mixing should be kept within the working life of the material to ensure that the adhesive does not start to cure and therefore flows correctly when it is applied and when the bond is closed. To ensure consistency when making batches of samples, it is important to ensure that the samples are made with as little difference as possible in the times between mixing and making the first and last joints. The whole operation should be completed well within the working life of the adhesive.

The procedure for manufacturing the joint should minimise any inclusion of air in the adhesive that could lead to voids in the joint. It is best to apply the adhesive, whether through a nozzle or by spatula, in a single pass to avoid folding the adhesive over on itself.

It may be necessary to remove excess air from the adhesive prior to manufacturing specimens. This can be done by vacuum out gassing if there are no volatile components in the adhesive system. The pressure should be reduced slowly to avoid ‘foaming’ of the adhesive. Stirring while under vacuum improves the effectiveness of air removal. Adhesive can also be centrifuged to remove air, but this runs the risk of components in the adhesive with different densities, such as fillers, becoming separated by the process.

Curing of adhesives

There are a number of key points that should be considered when curing adhesive joint specimens [1, 3, 5]:

- 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 test coupon should be similar to that in the final bonded structure. The same is also true of bulk adhesive specimens used to generate mechanical property data [1]. 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 [5].
- 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, different types of joint specimens may heat at different rates and therefore the final temperatures of the adhesive joint at the end of the cure period can be significantly different.
- 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. This is a particular problem with thick bond-lines.
- 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 previously, handling adhesives can be hazardous to human health, thus 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 [148]. 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 (see also NPL Measurement Good Practice Guide No. 32 [149]).

Quality and assurance documentation

Quality assurance tests

After the adhesive is cured, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the bonded area. The trained eye can detect specimen misalignment (i.e., twist and non-parallel edges), unfilled areas and voids.

Joints containing adhesive depleted regions around the edges or ends of the bonded area should not be tested. Thick, thin, or uneven bond-line can also be detected visually or by using a micrometer or optical microscope.

Checks should also be made on the adhesive fillet to ensure that the fillet complies with test specification. It is also advisable to check that the adhesive is fully cured and has bonded to the surface of the adherend. The texture and hardness can be a clue as to the effectiveness of the curing process. Several techniques are available for the non-destructive inspection of adhesive joints:

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

Documentation (record keeping)

It is recommended that an accurate record of the adhesive and adherends be maintained. It is good practice to perform routine checks each month to ensure that the stored materials are within the time limits recommended by the adhesive manufacturer. The storage record of the adhesives and adherends should include details on:

- The material source, manufacturer's code number, batch or lot number,
- Form and packaging (i.e., for adhesives one-or two-part, or film, etc.),
- Shelf life and recommended storage conditions,
- Observations of the condition of the material on receipt (e.g., surface conditions of the adherends, including corrosion and surface defects such as scratches),
- Actual storage conditions (including temperature),
- Handling history during the shelf life (i.e., dates of use and length of time at room temperature if refrigerated),
- Manufacturer's data sheets (material certification), and
- The records should also include test data from acceptance tests and routine quality checks carried out to assess material performance.

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.

The adhesive joint manufacture records should include details of:

- Date of manufacture;
- Person doing the manufacture;
- Adherends used;
- Adhesive used (including batch number and details of previous use);
- Surface preparation of the adherends and details of preconditioning; and
- Manufacturing details
    - Assembly method (jigging)
    - Fillet control
    - Bond-line thickness control measures
    - Curing conditions (i.e., temperature and humidity)
    - Manufacturing conditions
    - Post manufacture quality checks

Test records should include the information for the test specimen recorded previously as well as:

- Details of test machine and instrumentation employed (including calibration records)
- Test conditions
    - Load or strain rate
    - temperature

Mechanical testing of adhesive joints

This section considers the affect of test parameters (i.e., test machine alignment, load train stiffness, methods of gripping test specimens, 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. The guidance considers monotonic (static) loading but is relevant for other modes (i.e., cyclic fatigue and creep) and environmental conditions (i.e., elevated humidity and temperature).

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). 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.

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 [150, 151]. Alignment specimens can be in the form of a rectangular or circular bar (see Figure 51). 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 [150]. 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 51).

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

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 [1]. 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. 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.

An approximate measurement of strain and hence stiffness can be obtained from measuring the crosshead displacement of the test frame. The strain is the ratio of crosshead displacement and the bond-line thickness, and 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 to take into account the stiffness of the loading chain. 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.

Speed of testing

Polymeric adhesives are visco-elastic materials; that is their mechanical properties 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.

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Appendix 3

Finite element analysis

Introduction

Finite element analysis (FEA) is a useful tool for interpreting the mechanical behaviour of adhesive joints. Force-extension responses can be predicted, and maps of the stress distribution produced to identify critical regions where the local stress or strain may exceed a failure criterion for the material. The behaviour of bonded adhesives has been modelled through different approaches using finite element (FE) analysis. In many cases the adhesive and adherends are modelled using continuum elements, assuming the adhesive is perfectly bonded to the adherends [69]. For example, Cotton et al. [126] determined the interfacial shear strength of a system by assuming the two components were perfectly bonded and that the substrate maintained contact without any slippage. The assumption of a perfect bond means that the finite element analysis takes no account of the adhesion properties of the interface.

There does not appear to be an obvious way of predicting interface strength, as often the input data needed requires knowledge of the interface strength, or the failure stress/strain of the adhesive. But there are ways of accounting for adhesion in an FE analysis, by modelling the way in which the adhesive fails. This Appendix highlights some of the options available. There are two main approaches for modelling the failure of an adhesive interface. One approach is to model the growth of a crack (fracture mechanics energy-based approach). A second method is to model adhesive de-bonding (stress or strain-based approach).

Fracture approach

In the fracture mechanics approach, a sharp crack is assumed to exist within the material and failure occurs through propagation of this crack. In an adhesive joint, de-bonding may initiate in regions of stress concentration, then grow and cause joint failure. A de-bonded area can therefore be classed as a crack. This approach does not predict the crack initiation stress or energy. The general approach is to relate the crack growth rate (*da/dt*) through the joint to the applied strain energy release rate *G* or the stress intensity factor *K*. Within the literature, authors have considered measured and/or predicted values of *G* [152-154] and *K* [155, 156]. Calculation of the energy release rate *G* is based on linear elastic fracture mechanics (LEFM). This is valid when the plastic zone around the crack tip is very small. LEFM is not necessarily always applicable to adhesives. Toughened adhesives are relatively ductile and therefore do not always exhibit sharp crack behaviour. For non-linear situations, *G* is replaced by *J*, the non-linear energy release rate [152, 156]. *J* is applicable to any stress-strain relationship and is equal to *G* when the material is linear elastic. *J* can be written as a path-independent contour line integral giving rise to the name, the J-integral. The J-integral takes account of plastic zone size and is therefore more appropriate than *G* when the plastic zone size is large. The determination of *G* for linear elastic materials is relatively straightforward, while determination of the J-integral for non-linear materials is more complex. With FE methods, stresses, strains, and displacements along a path can be calculated using the J-integral [152, 156]. As the J-integral is path-independent it can be calculated away from the crack tip and is therefore not so affected by approximations in modelling stress and strain fields at the crack tip.

Simulations of crack propagation advance the crack front when the local energy release rate rises to a critical value. To apply such methods, one must introduce an initial flaw. In FE packages, such as ABAQUS [157], crack propagation can only be modelled along a predetermined surface. Potential crack surfaces must be modelled using contact surface definitions. Surfaces may be partially bonded initially and then may de-bond during crack propagation. The three de-bonding criteria are: crack opening displacement; critical stress criterion at a critical distance ahead of the crack tip; crack length as a function of time. After de-bonding, the interface behaviour reverts to standard contact, including any frictional effects.

Guild et al. [158] used the virtual crack growth method to model crack propagation. Cracks were introduced into the mesh by renumbering nodes of adjacent elements. Crack tip elements were not used, as point values of stress at the crack tip are not meaningful. The preferred direction of crack growth because Mode I opening could be deduced from examination of the vectors of maximum principal stress. The virtual crack growth method for assessing energy release rate is based on the concept that the energy released in growing the crack is equal to the energy which would be required to close the crack. Forces and displacements at the crack tip are found from FE simulations and calculations of energy can be made subsequently. The shape of the growing crack can be used as a further indication of the likely direction of further crack growth.

Interface elements can also be used to predict crack growth. In the work of Chen et al. [159] interface elements, defined via a user subroutine, were located along the de-bonding interface. This method is based on the indirect use of fracture mechanics in which a softened decohesion material model is provided with traction/relative displacement relationships that are constructed so that the enclosed area is equated to the critical fracture energy. Initial flaws are not required, with initiation being governed by a strength criterion. Two material properties were required for interface elements: G_c, the total energy from experiments and S_t, the assumed interfacial material strength. Both a linear and a cubic decohesion material model were used. In the linear model there is a triangular relationship between traction and the relative displacement (see Figure A3.1). The triangular area is the critical interlaminar fracture energy, G_c. The stress/strain relationship is defined by the interfacial tensile strength, σ_t, the cracking strain ε0, and the maximum strain, ε_c. In the FE simulation, the strain can exceed ε_c but the equivalent stress is zero and the interface is de-bonded. The elements where softening takes place are in the region $\varepsilon_0 < \varepsilon < \varepsilon_c$. The cubic relationship for the decohesion model is shown in Figure A3.1.

Cohesive zone models can be used to analyse the failure of an interface. These have been employed in the study of fracture in many ways (e.g., as phenomenological element breaking rules for fracture, and as models for the process zone ahead of a crack tip). Jagota et al. [160] describe cohesive zone models for polymer interfacial fracture, which have been implemented as cohesive elements for use with FE codes. The authors suggest that when incorporated between element edges (or faces in 3D) cohesive elements extend conventional FE in a way that allows independent specification of interfacial fracture and bulk constitutive behaviour. The cohesive elements describe the deformation and failure of the interface between two bulk finite elements by specifying the tractions that resist relative motion. Bennison et al. [161] used the cohesive zone model with FEA to analyse a compression shear strength test to determine adhesion. Cohesive elements were placed along the interface to capture intrinsic and near crack dissipation associated with interface de-bonding. The cohesive elements define the tractions acting across this interface in terms of opening and sliding displacements. The authors concluded that analysis of the compression shear strength test shows that cohesive elements can be used to treat adhesion/fracture problems where large inelastic deformations characterise the polymer response to loading.

Liechti and Wu [162] used the cohesive zone modelling approach to model rubber/metal debonding. The authors found that failing ligaments clearly provided some bridging between crack faces. The failing material in this study was represented by non-linear Kelvin units (non-linear springs and dashpots in parallel). The rate-dependent cohesive force was expressed as the sum of the non-dissipative force (springs) and the dissipative force (dashpot). At a particular ligament extension damage starts to occur at the interface and the stiffness of the interface decreases. At a critical extension, the ligament breaks, and the cohesive force vanishes. In the FE model part of the bond line was not connected to the substrate to account for the initial crack length and traction-free boundary conditions. The remainder of the bond line was replaced with an array of equally spaced non-linear Kelvin units to enable crack growth to occur along the interface. Calibration of the cohesive zone model was accomplished via numerical simulations of the observed de-bonding experiments. The values of the parameters of the cohesive zone model were chosen such that the solution for the crack extension history corresponded to the measured de-bonding history at one loading rate.

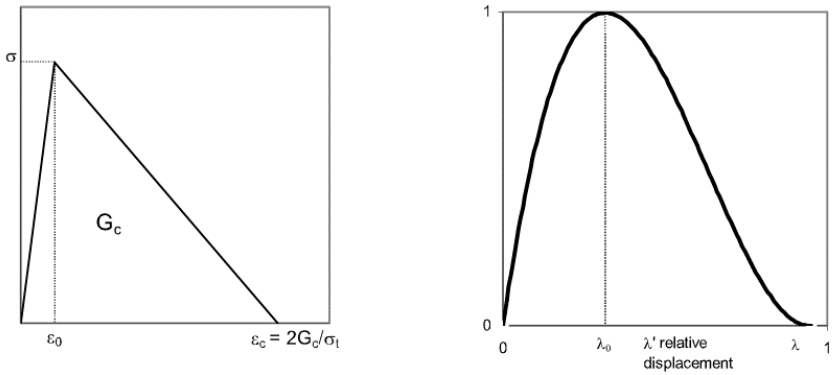

Figure A3.1: Decohesion models: linear model (left) and cubic model (right)

Coupling elements have also been used to study adhesion. Sebastian [163] modelled FRP plates de-bonding from concrete beams. A non-linear FE analysis used a layered bending membrane hybrid element for the beam and the plate, along with a coupling element for the adhesive connection. The coupling element was programmed to rupture and to disallow further interaction between the hybrid elements on either side once a predetermined stress level corresponding to the shear bond strength of the plate to concrete connection had been activated. The program can incorporate non-linear constitutive models for the materials present. The author suggests that the FE program can be improved: the analysis used was a stress-based failure criterion for connection failure, but the author suggests that a fracture mechanics-based criterion may be more appropriate.

An alternative de-bonding method can be employed in ABAQUS using the *de-bond option [157] to specify that de-bonding is to be considered. An amplitude-time function (see Figure A3.2) is used to give the relative magnitude of force to be transmitted between the surfaces at time t_0 + t_i (t_0 being the time when de-bonding begins). When the fracture criterion is met at a node, the force at that node is ramped down according to the de-bonding data. The force at the node must have a value of 1.0 at zero time and must end with a magnitude of zero at the final time (i.e., node de-bonded).

Damage modelling is another method for modelling failure of an adhesive. Feih et al. [164] modelled a bending test. The adhesive was modelled with isotropic elasticity and von Mises plasticity. Failure of the adhesive was assumed to occur when the maximum tensile plastic strain was reached under tension. Cracking onset of the adhesive was modelled by reducing the transferable stress to 10%. This simplified damage method simulates the stress redistribution inside the adhesive and was employed as a user subroutine within an FE model. Stabilised integration point degradation was used to degrade the element properties at the integration point once failure was recorded at that point (one element might have different properties at its integration points over a certain period). In order to avoid numerical problems due to a singular stiffness matrix, full degradation is modelled using a very small degradation factor of 0.01 rather than zero.

ABAQUS/Explicit also offers two damage models: a shear failure model driven by plastic yielding and a tensile failure model driven by tensile loading. These failure models provide simple failure criteria that are designed to allow stable removal of elements from the mesh because of tearing, ripping or tensile spalling of the structure. These models can be used in conjunction with the Mises or Johnson-Cook plasticity models. Each model provides several failure choices including the removal of elements from the mesh.

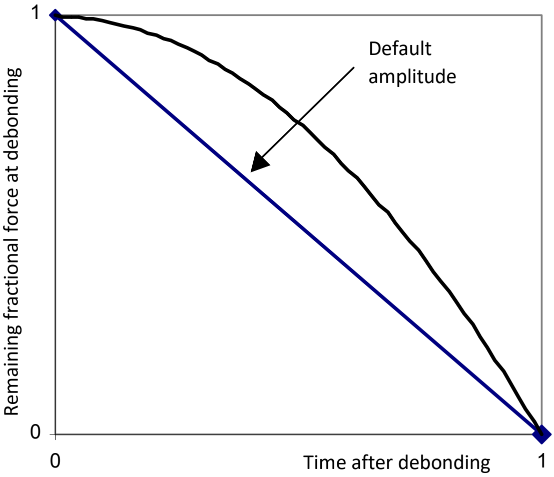

Figure A3.2. Amplitude-time function used to provide the relative magnitude of force to be transmitted

In ABAQUS it is also possible to define breakable bonds that connect the surface of contact pairs from two contact surfaces. Either a time to failure or a damage failure model is used to simulate the post failure response of the bonds. These breakable bonds are design for modelling spot-welds and a spot weld failure criterion is used to model failure. This is a function of the force required to cause failure in tension (Mode I loading) and force required to cause failure in pure shear (mode II loading). This method could possibly be used to model de-bonding of the adhesive/adherend interface. Other possibilities for modelling the adhesive interface include using non-linear spring elements to connect the adhesive elements to the adherend elements or including a narrow row of elements at the adherend/adhesive interface, with different properties to the bulk adhesive to simulate the interface. This would probably require a very refined mesh of elements and knowledge of how the interface should behave. It might also be possible to use contact surfaces with softened behaviour, i.e., as the surfaces move apart the contact pressure between them decreases until it reaches zero (separation).

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