



Good Practice Guide No. 104

Fatigue and creep testing of adhesives and thermoplastic joined systems



EXPERT

TECHNICAL LEVEL

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Abstract

Welded thermoplastic and adhesively bonded joints are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the joined system. However, service conditions can often involve exposure to static and/or cyclic fatigue loading conditions. Fatigue damage can be particularly harmful to the structural integrity of bonded joints, shortening the life expectancy of the joined system by considerable margins, and is known to occur at relatively low stress levels, particularly in the presence of hostile environments. Similarly, premature failure can occur in bonded joints and welded thermoplastics under static loading conditions with deformation occurring at relatively low loads. This Good Practice Guide is intended to give guidance on fatigue testing of adhesively bonded joints and creep testing of bonded joints and welded thermoplastics for generating design data and for quality assurance purposes. The document is primarily concerned with structural adhesives and thermoplastics.

NPL has made every effort to ensure all information contained in this Good Practice Guide was correct at the time of publication. NPL is not responsible for any errors, omissions, or obsolescence, and does not accept any liability arising from the use of this Good Practice Guide.

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Glossary of Terms (Based on BSI and ASTM Definitions)

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

Adherend: Body that is or 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.

Ageing: Entirety of all irreversible chemical and physical processes occurring in a material in the course of time.

Amorphous: Non-crystalline or devoid of crystalline structure.

Amorphous regions: Regions within a polymeric material, that, based on X-ray diffraction or other suitable techniques, do not show any evidence of crystalline structure.

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.

Conditioning: A series of operations intended to bring a sample or specimen into a reference state with regard to temperature and humidity.

Coupling agent: Substance (e.g., organosilane) that promotes or establishes stronger bond at the interface of the resin matrix and the reinforcement.

Coupling size: Size designed to obtain a good bond between the fibre surface and surrounding resin matrix.

Crack: Fissure that may or may not penetrate the external surface of the material or its entire thickness, the polymeric material being completely separated between the crack walls.

Craze: Defect at or under the surface of a plastic attributable to apparent cracks, bridged by polymeric material of reduced (apparent) density.

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

Crystallinity: Presence of three-dimensional order at the level of molecular dimensions.

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

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

Cure temperature: Temperature at which an adhesive, an assembly or a polymeric composition achieves a state of cure.

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.

Diffusivity: The proportionality constant that describes the ability of chemical species, such as a liquid, to diffuse through a solid. Diffusivity is generally temperature dependent.

Dimensional stability: Consistency of dimensions of a material part or specimen under environmental conditions.

Discolouration: Colour change that involves lightening or darkening and/or change in hue.

Dry strength: Strength of a polymeric material part or specimen after drying under specified conditions.

Ductile failure: Failure in which the fracture surface clearly exhibits permanent deformation with stretching, elongation, and necking.

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

Elastic limit: Greatest stress, which a material is capable of sustaining without any permanent strain remaining upon complete release of stress.

Elongation: Increase in length of a specimen under tension, usually expressed as a percentage of the original length.

Embedding: Process of encasing completely an article, such as a sensor, in a polymeric material.

EN: European Norm

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

Fibre: Unit of matter of relatively short length, characterised by a high ratio of length to thickness or diameter.

Filler: Relatively inert material (e.g., talc) added to a plastic or resin to modify its strength, fire resistance or other qualities, or to lower costs.

Fillet: Portion of an adhesive that bridges the adherends outside the bond-line.

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.

Hardness: Resistance of a material to indentation or scratching.

Hygroscopic: Material capable of absorbing and retaining environmental moisture.

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.

Lay-up: Assembly of layers of resin-impregnated material for processing.

Ligament area: Cross-sectional area remaining after notching.

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.

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

Plastic deformation: That part of the strain in a stressed plastic, which remains after the applied stress has been removed.

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

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

Porosity: A condition of trapped pockets of air, gas, or vacuum within a solid material. Prepreg: Admixture of resins (with or without fillers), additives, and reinforcements in woven and filamentous form, ready for moulding.

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. Roving: Collection of parallel strands (assembled roving) or parallel filaments (direct roving) assembled without intentional twist.

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

Semi-crystalline: Polymer containing crystalline and amorphous phases.

Service life (N): Number of 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.

Size (coupling size): Material applied to fibres or filaments during the course of their manufacture.

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 the 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.

Swelling: Increase in volume of a test specimen immersed in a liquid or exposed to a vapour.

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

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

Toughness: That property of a material by virtue of which it can absorb energy, generally implying absence of brittleness and relatively high elongation to break.

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

True stress: Stress calculated from the supporting area at the time of measurement rather than from the original area.

Viscoelasticity: Stress response of a material acting as though it were a combination of an elastic-solid and a viscous fluid with flow dependent on time, temperature, load, and rate of loading.

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

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

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

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

Chapter 1

Introduction and scope

- Introduction
- Scope

Introduction

Welded thermoplastic and adhesively bonded joints are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the joined system. However, service conditions can often involve exposure to static and/or cyclic fatigue loading conditions. Fatigue damage can be particularly harmful to the structural integrity of joints, shortening the life expectancy of the joined system by considerable margins, and is known to occur at relatively low stress levels, particularly in the presence of hostile environments. Similarly, premature failure can occur in joined systems under static loading conditions. Deformation occurs at relatively low loads.

Scope

This Good Practice Guide is intended to give guidance to technologists, engineers and designers on fatigue and creep testing of adhesively bonded joints, and creep testing of welded thermoplastics for generating design data and for quality assurance purposes. The document is primarily concerned with structural adhesives and thermoplastics (polypropylene). Consideration is given to the effect of processing parameters, geometric factors, and hostile environments on the long-term performance of these joined systems.

The intention of the guide is to provide designers and users with sufficient information, which when coupled with their own expertise and a suitable accelerated test regime can be used to produce design data and enable screening of materials, surface treatments and processing parameters. The document provides guidance of the use of test methods (i.e., performance testing) that can be used to measure physical changes resulting from fatigue and creep. If the intention is to generate design data, then the guide should be used in conjunction with the appropriate structural design codes and standards.

The guide assumes some basic knowledge of the materials and mechanical engineering and is not intended as a textbook or as a design protocol. There are several published works, which provide a comprehensive coverage of plastics, adhesives, and composites technology, and testing and preliminary design [1-28]. Other NPL Measurement Good Practice Guides (see [29-38]), provide advice on issues relating to the preparation and testing of plastics, adhesives, and adhesive joints. The intention of the guide is to complement these published works.

It is recommended that specialist advice be sought from manufacturers and suppliers on material selection, and use of associated technologies and health and safety requirements. Expert advice should be obtained from the adhesive manufacturer on selection and use of surface treatments for adhesive bonding and welding thermoplastics, and that the detail requirements specified by the manufacturer are completely satisfied.

Where tests are performed to assess adhesive materials or processing parameters for welding thermoplastics then it is recommended that the surface preparation is as good as possible, to minimise premature failure. The surface preparation procedures for test specimens will need to mirror those for the final joined component. Organisations that can provide specialist advice are listed at the back of the guide along with relevant standards and publications.

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Chapter 2

Mechanical testing

- Introduction
- Preparation and assembly of joined systems
- Mechanical testing of joined systems
- Specimen geometry and adherend effects

Introduction

Several factors, not included in written standards, can significantly affect the reliability (i.e., repeatability and reproducibility) of data obtained from testing adhesively bonded and welded joints. Factors, including specimen and machine alignment, processing variables, specimen preparation, storage of materials and surface treatments need to be considered in addition to test parameters (e.g., displacement rate), and specimen dimensions that are generally covered in national and international standards. Careful consideration needs to be given to those factors that significantly influence strength and long-term performance of joined systems. This Chapter examines the key issues relating to the preparation, assembly and testing of adhesive joints and welded thermoplastics (including constituents).

Preparation and assembly of joined systems

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. Surface preparation also has a major influence on the quality of welded thermoplastic joints. It is worth noting that a high percentage of failures can be attributed to poor joint manufacture or a lack of understanding of those factors that influence joint performance. These problems can be minimised or eliminated through proper training and education. This section is concerned with issues relating to the preparation and assembly of adhesive joints and welded thermoplastics prior to curing or welding.

Specimen manufacture: Adherends should be manufactured and/or machined accurately to ensure specimen dimensions meet the specifications of the standard. It is important to ensure that the adherends are free of any edge or surface damage. The surfaces to be joined 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 joint and/or the adherend. It is advisable before preparing the surface to ensure that the adherend sections to be joined 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.

Moisture effects: The presence of moisture at the surface or within the system can adversely affect the properties of adhesives and thermoplastic welds during thermal processing (i.e., cure or fusion), and as a result the joint strength may be compromised. Moisture released from the substrates of bonded joints during cure will enter the adhesive. For example, moisture released from polymer composite adherends 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 [39]. Fibre-reinforced thermoset composites are known to absorb moisture in relatively benign environments. For example, epoxy based composite systems can absorb 0.2 wt.% moisture in a laboratory environment (i.e., 23 °C and 50% relative humidity (RH)) within 2 to 4 weeks.

The strength and fatigue performance of the bonded structure under these circumstances will be compromised. 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 adherends should 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 adherends.

Surface preparation: This process is recognised as the most critical step in the joining process and considerable joint testing is performed to optimise surface treatment. Surface preparation of coupon specimens should match that to be used in the bonded or welded component. 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 and welded joints. In the case of adhesive joints, unsatisfactory surface preparation will result in the joint failing unpredictably at the adhesive/adherent interface.

Many adhesive joint tests have been developed to assess bonded systems (including surface preparation) – information on surface preparation and relative effectiveness is given in NPL Measurement Good Practice Guide No. 47 [34]. General procedures used for preparing different material surfaces for adhesive bonding are described in “Guide to The Structural Use of Adhesives” produced by The Institution of Structural Engineers [18]. BS 5350-A1 [40], BS 7079 [41], ASTM D2093 [42], ASTM D2651 [43], and BS EN 13887 [44] – see also [45-48].

Note 1: Surface preparation procedures for adhesive joints often require potentially hazardous or environmentally damaging chemicals. All preparation should be carried out to COSHH specifications (see Chapter 5).

Specimen geometry, dimensions, and preparation for welded joints of thermoplastics are given in BS EN 12814-3 [49]. ISO 899-1 [50] recommends that specimen preparation for tensile creep testing on unwelded polymers should be carried out in accordance with ISO 527-1 [51]. Surface contaminants may contribute to failure of the weld. A region of 0.4 mm should be removed from the edges of the sections to be welded before fusing the surfaces.

Cleaning the surfaces with detergent, hot water, or other chemical agents (e.g., solvents) is not recommended. These chemical agents may result in the removal of plasticizers or stabilizers from the surface, which may lead to the breakdown of the surface and compromise the welded joint.

Bonding and welding fixtures

Bonded joints: A bonding fixture is recommended to ensure accurate alignment, correct bond length (in the case of lap joints) and uniform bond-line thickness. Alternatively, large test panels (typically 180 mm wide) capable of providing six specimens can be made and then machined into specimens.

Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e., adherend bending) or that excessive adhesive is forced from the joint due to clamping forces applied to the test specimens. It may be necessary to check the clamping force applied by the fixture to the specimen during the curing process to ensure that clamping force remains constant and has not relaxed through adhesive flow. Mould release agent or thin polytetrafluorene (PTFE) film will need to be used to guarantee easy release of bonded components from the clamping fixture.

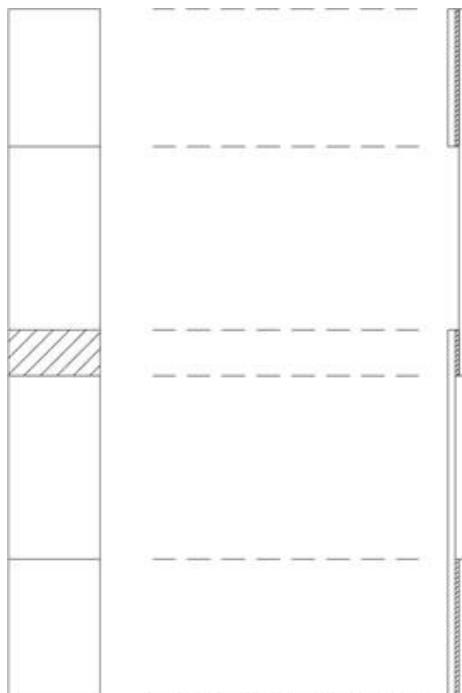


Figure 1. Schematic of a single-lap joint

Note 2: End tabs are introduced to single-lap joints in order to reduce (not eliminate) the eccentricity of the load path that causes out-of-plane bending moments, resulting in high peel stresses and non-uniform shear stresses in the adhesive layer (see Figure 1). BS EN 1465 [52] does not specify the use of end tabs but specifies that the long axis of the specimen coincides with the direction of the applied force through the centre line of the grip assembly. The introduction of end tabs, however, will add to the costs of this quality assurance test.

Welded joints: A similar approach to that employed for bonded joints needs to be adopted when welding thermoplastic sections. A fixture is required that ensures good alignment, and accurate and repeatable bond-lines. The specimen needs to be held securely (i.e., clamped) so there is no lateral or out-of-plane (i.e., buckling) movement. In the case of hot plate welding, the holding fixture needs to be square with respect to the heating plates. It is important to ensure that surface is only melted where required. Figure 2 shows hot plate welding equipment with holding clamps. The hot plate (core) is centrally located (further details on hot plate welding are given in Chapter 10 of [53]). The fixture should be sufficiently robust and capable of withstanding the temperatures required to weld the thermoplastic sections. The recommendation is to weld rectangular sections and then to machine test specimens from the welded panel.



*Figure 2. Hot plate welding rig
(Courtesy of TWI Limited)*

In conventional hot fusion (i.e., hot plate) welding, the cooling time (typically 6-15 minutes) accounts for a considerable portion of the total cycle time and is fixed for the size of the sections or components being welded. It is important, however, not to move the welded section from the holding frame until the welded material is solid and that the weld has sufficient strength to allow removing from the holding frame. Platen surfaces accumulate melted plastic and must be cleaned for high temperature welding materials. It is recommended that the weld bead (see schematic in Figure 3) should be removed before carrying out tests to assess joint performance. A large variation in total bead width, and to a lesser degree height, can be expected for the same welding conditions. The weld bead should be removed before machining individual specimens from the welded panel. Care should be taken to ensure the underlying material within the weld is not damaged due to machining of the specimen. Heating during machining should be minimised. Machined specimens should be visually inspected in accordance with the standard EN 13100-1 [54].

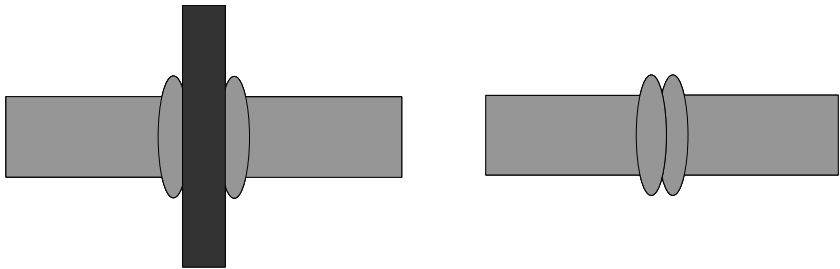


Figure 3. Schematic of hot plate welding

The quality of a weld in thermoplastic polymers (e.g., polypropylene) has a significant affect on the weld strength, and the long-term fatigue and creep rupture behaviour of the welded system. In the case of hot plate welding, the hot plate temperature, fusion pressure, hot soak time and cooling time are key parameters in ensuring the quality of the weld meets specification. Similar parameters operate for other forms of welding. Hot plate temperature seems to have a dominant influence on creep rupture, although the other processing parameters are important and must be considered.

Adhesive fillet

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

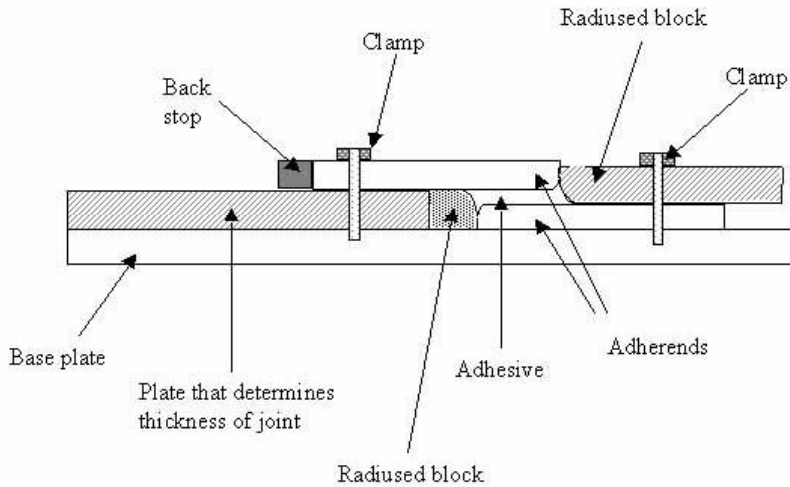


Figure 4. Fixture for controlling single-lap joint fillet

Several points are worth noting:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint (e.g., cylindrical roller). The tool can be held in place using heat resistant tape. Figure 5 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 due to high viscosity.
- 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.

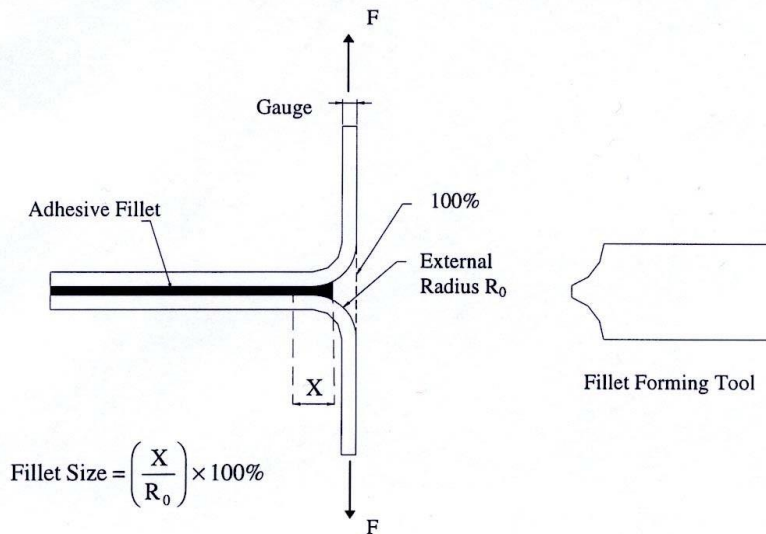


Figure 5. Definition of fillet size in T-peel joint (including fillet forming tool) [55]

Adhesive joint bond-line thickness

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

Control of bond-line thickness can be achieved by mechanical means (i.e., separation of adherends physically controlled by the bonding fixture), using thin wire spacers (e.g., stainless steel) inserted between the adherends or by ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1 weight %). Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness. It is essential that wire spacers used to control bond-line thickness are located well within the bonded area away from the specimen edges and regions of high stress concentrations (i.e., ends of joints). It may be difficult to employ wire spacers where the bond length is relatively short, such as single-lap joints (typically 12.5 mm overlap length).

When using glass beads to control bond-line thickness, the distribution of glass beads in the adhesive must be uniform, and therefore glass beads should be thoroughly mixed into the adhesive. Mixing should take place before applying the adhesive to the adherend surfaces.

Curing adhesive

Several key points should be considered when curing adhesive joint specimens:

- Porosity, in the form of entrapped air and volatiles, is a common cause of premature failure. In many cases it is virtually impossible to produce void free specimens, particularly for materials with a high viscosity. Specimens should be prepared using methods that minimise the inclusion of air in the test specimens. Visual inspection should be carried out to ensure there is no air entrapment.
- The cure state of the adhesive layer in the adhesive joint specimen should be similar to the adhesive in the bonded structure or to bulk adhesive specimens [56-57]. Failure to achieve similar thermal histories can result in significant differences in material properties. Differences between thermal histories will lead to differences in mechanical properties (See [33]).
- 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 in adhesive joints at the end of the cure period can differ significantly.
- For heat curing systems, the temperature of the specimen will lag behind the oven temperature, and it may therefore be necessary to elevate the oven temperature when curing joint specimens.
- Adhesives should be fully cured prior to conditioning and testing otherwise an adhesive will continue to cure, thus invalidating the test data.
- Adhesives have a low thermal conductivity. This may prevent dissipation of heat generated by exothermic cure reactions, thus causing heat damage.
- Residual thermal stresses may be generated as a result of non-uniform (rapid) cooling, resin shrinkage and thermal expansion coefficient mismatch between the adhesive and adherend. As the joint is cooled down from the cure temperature, residual stresses are frozen in the material.
- As mentioned previously, handling adhesives can be hazardous to human health, thus COSHH procedures should be followed to minimise operator exposure. Ovens and work areas should be suitably ventilated, ensuring minimal levels of hazardous vapours/gases in the work area.

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

Real-time monitoring of material property development in adhesives can be achieved using oscillatory rheometry or ultrasonic methods. Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), can provide useful information relating to adhesive composition and final state of cure [58-60].

Note 3: The analysis of material by any technique is complicated by changes in the material during testing. For adhesive materials, there will most likely be further curing or loss of moisture. In some cases, these changes will prevent the accurate determination of T_g . Repeat measurements need to be taken to check completeness of cure. If the difference in T_g is greater than 5 °C between the first and second runs, then a third run is recommended. It is important that the material used for DSC and DMA samples is representative of the adhesive within the bonded structure. The suggested method for preparing samples is to place the adhesive between two flat, smooth, parallel metal adherends (i.e., stainless steel), which have been coated with a thin layer of lubricant or release agent to prevent adhesion. Adhesive samples can be easily removed without sticking from the bonded joint after curing.

Quality assurance

After the adhesive is cured or weld has cooled, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the joined region. 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.

Adhesive joints: 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.

Welded Joints: The size (i.e., width and height) of the weld bead and weld type should be recorded. If the bead has been removed, then a check should be carried out to ensure no damage has occurred to the material within the welded region and adjacent material. Special care needs to be taken when notching material (e.g., full-notch creep test (FNCT) – see ISO 16770 [61]) to ensure the notches are coplanar and the plane of notching is perpendicular to the tensile axis of the test specimen.

The notch radius needs to be less than 10 μm . It is recommended that razor blades be used for this purpose, although a cutting machine with a tool like a broaching device can be used provided the notch tip radius is less than 10 μm . Due care needs to be taken to avoid blunting the notch during this operation. The recommended tolerance on the notch depth is $\pm 0.1\text{ mm}$.

Several techniques are available for the non-destructive inspection of adhesive and welded joints including ultrasonic C-scan, X-ray radiography and thermography.

Note 4: There is no NDE technique that can provide a quantitative assessment of joint strength.

Ultrasonic C-scan technique is particularly suited to the detection of planar type defects (e.g., debonds and delaminations) normal to the incident beam. Voids and porosity in the adhesive and adherends are also detectable. The minimum size of voids and delaminations detectable with C-scan is approximately 2-3 mm. The technique is not suitable for detecting surface contaminants (e.g., oils and grease). Scanning acoustic microscopy (SAM) can detect 0.2-0.3 mm size sub-surface defects in polymeric materials.

X-radiography can be used to inspect for debonds in polymer composite joints and between the outer skins and inner core of bonded composite sandwich structures. The technique is also capable of detecting micro-cracks in polymeric materials. X-radiography is not suitable for inspecting joints with metal adherends. Thin debonds and delaminations are difficult to detect because the presence of these defects has minimal effect on the absorption characteristics of polymeric materials.

The use of penetrant fluids can enhance the imaging process; however, these fluids can adversely affect the short-term properties and fatigue performance of polymeric materials. The fatigue life can be reduced by a factor of 2 or more. Penetrants should not be used in those tests where the test data is to be used for design or quality assurance purposes. Small tensile loads or the use of a vacuum pump can be used to promote fluid penetration.

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

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

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

Specimens should be stored in a desiccator (unless otherwise specified) to prevent moisture ingress, which can degrade the adhesive joint. Adhesive joints have been known to deteriorate under standard laboratory conditions (10% strength loss within 3-6 months). It is recommended that an accurate record be maintained for all batches of adhesive joints. Documentation should include details on date of manufacture, precursor materials (i.e., adhesive and adherends), description of method of joint fabrication and assembly (including processing variables - curing time, temperature, and pressure), surface preparation, and specimen dimensions (including width, bond length, adhesive layer and adherend thickness).

Mechanical testing of joined systems

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 polymers, and adhesive and welded joints. Guidance is provided on the main factors that need to be controlled when carrying out mechanical testing. Appendix 1 in NPL Measurement Good Practice Guide No. 47 [34] provides a summary of commonly used adhesive joint test methods and related standards.

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.

Note 5: Poor alignment of test specimens can result in low strength values and a significant reduction in fatigue life.

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. Alignment specimens can be in the form of a rectangular or circular bar. These specimens need to be accurately machined to ensure errors in parallelism are $< 0.2 \text{ mm/m}$ and in concentricity (lateral offset) of 0.03 mm [62]. 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. A positioning device should be used 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.

Gripping 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 that 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 (torsion) 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

Several contact and non-contact techniques are available for measuring strain and displacement many of which are not suited to fatigue tested (e.g., electronic speckle pattern interferometry). These techniques are described in NPL Measurement Good Practice Guide No. 47 [34] and No. 115 [63].

Contact extensometers are the preferred method for measuring strain and displacement, and hence stiffness of adhesive and welded joints. It is recommended that two extensometers, attached to opposite faces of the specimen, be used to measure displacement. Any bending of the specimen will be apparent from diverging displacement readings. It is also 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. This also applies to welded sections where the measured extension is the average effect of the deformation within the welded region and surrounding material within the gauge section.

Knife-edged tensile extensometers, as described in reference [30], can be used provided the extensometer straddles the bond-line. The deformation of the adherends needs to be accounted for when analysing the data, but where the stiffness of the adherends is very much greater than that of an 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 $\pm 0.5 \mu\text{m}$ 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. Contact extensometers and data acquisition system must also have sufficient response time to cope with the test frequency in cyclic fatigue testing.

Strain gauges: Currently there are no standard tests using strain gauges to monitor strain in an adhesive layer or welded region. However, structural monitoring capabilities, where strain-sensing optical fibre devices are embedded in materials, are the subject of research in many organisations. Care should be taken to ensure that measurements of strain derived from strain gauges mounted on the test specimen are reliable; as strain gauges tend to locally stiffen polymers, such that the strain is less than expected for a given load.

Strain gauges can be attached to adherends and will measure the strain in these adherends. The usefulness of such measurements may be limited except in the cases where changes in joint performance are manifested in measurable changes in the adherend strain. One such application is back-face strain gauging of thin lap-shear joints where crack growth in the adhesive layer can be monitored through strains measured by gauges bonded to the external surface of the adherends at the overlap. Strain gauges are occasionally used for monitoring strain in bonded structures (e.g., four-point bending of sandwich structures).

Strain gauges are generally limited to the measurement of strains less than 10%. Biaxial rosettes are available for measuring longitudinal and lateral strains. Large strain gauges are preferable as alignment and handling is easier, and they average out local strain variations. Local strain variations can cause premature failure of the strain gauges.

Correct alignment of strain gauges is important, as significant errors can be caused by careless application of the strain gauges to the specimen. Errors of 15% can occur from a 2° misalignment.

The adhesive used to bond the strain gauge should be capable of withstanding the test environment for the complete duration of the test. Most adhesives are sensitive to moisture (and other chemicals), which can often preclude bonding prior to specimen conditioning. Moisture attack of an adhesive and strain gauges will occur from the top, edges and in the case of polymeric materials through the substrate beneath the gauge. The situation is exacerbated at elevated temperatures. It is therefore important to ensure that the adhesive selected for bonding the strain gauge and associated electrical wiring is suitably encapsulated. Strain gauge manufacturers can provide information on adhesive selection, surface preparation and procedures for protecting strain gauges, and providing advice on fatigue performance and strain limits of these devices.

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

Crosshead displacement: An approximate measurement of strain, and hence stiffness can be obtained from measuring the crosshead displacement of the test frame [30]. The strain is the ratio of crosshead displacement and the initial grip separation. Hence, any slippage within the loading train will produce errors in the strain measurement. The strain values obtained from crosshead measurements will differ from the actual strain in the central region of the specimen. Stiffness measurements directly obtained from the crosshead movement need to be corrected to take into account the stiffness of the loading chain. This can be a difficult task as the specimen size and geometry, and the deformation behaviour of the specimen need to be taken into account. Given the small adhesive layer deflections that occur even at large strains owing to thin bond-lines, the accuracy of strains determined using crosshead displacements must be considered suspect and used only for qualitative purposes.

Linear voltage displacement transducers (LVDTs): LVDTs are recommended in preference to monitoring crosshead movement. These devices provide a direct reading of the moving part and can be attached at any point on the structure as required. LVDTs tend to be used to monitor global rather than localised deformation. Accurate alignment is essential otherwise measurement errors will occur and the movement of the device can be restricted.

It is important to ensure the device can operate effectively in the test environment and that electrical wiring is suitably protected. There is potential problem of friction, which arises from the movement of the core within the barrel, which is normally designed to have limited

rotational freedom [29]. Friction between the core and barrel can be significant resulting in “stick-slip” movement of the device.

Optical fibre-based sensors: Optical fibre-based sensors (e.g., Fibre Bragg grating (FBG)) devices can be used to monitor a wide range of parameters including strain, temperature, pressure, humidity, moisture ingress, in-situ cure kinetics, vibration, pH levels, chemical concentration, and gamma radiation. In addition, these devices can be used to monitor degradation processes and fracture. Most of the evaluation of these devices tends to be laboratory based under ideal conditions. As with other sensors, fibre optic devices can be in obscure locations not readily accessible by conventional NDE methods. These devices are small with low weight and in many cases can be embedded into or bonded to the structure (to produce smart structures). Composite laminates are particularly suited to their use simply by the fibrous nature of the material, and the fact that these devices can be introduced during the manufacturing of composite components. Bonding these devices to surfaces pose similar problems to those for strain gauges. Optical fibre diameters are large compared with carbon and glass fibres and can cause local distortion within composite structures. Optical techniques require sensors to remain fully bonded with the composite throughout the life of the component to ensure effective strain transfer.

Non-contact or optical extensometers: Non-contact extensometers (e.g., video extensometers) avoid the problems of contact damage and use up to failure. There are no temperature or environmental restrictions as video extensometers can be located outside the test chamber (provided that the specimen can be imaged clearly). Standard systems measure the separation of marks in one direction, mimicking contact extensometers, but some systems provide capabilities for dot location measurements, which allows two-dimensional measurements and a limited strain mapping capability. Dot location provides versatility and, potentially, enables measurement for a wide variety of different specimen geometries using a single system.

The technique uses remote cameras and image analysis software to monitor the separation of high contrast marks or lines inscribed on the test specimen. The initial separation of the marks defines the gauge-length and the change in separation of the marks is recorded throughout the test. Gauge marks should not be made on the specimen in any way that may cause damage to the specimen. Accuracy tends to be low for small strain measurements. Developments in resolution, sensitivity and speed of digital imaging and data processing are leading to improved capabilities. One limitation of the technology is that, unless a dual camera system is used, measurement is normally only possible at one side of the specimen so that bending cannot be evaluated.

Digital image correlation (DIC): DIC is a non-contact full-field strain measurement technique. The basic concept of DIC is to compare two images of a component before and after deformation. It uses computer image analysis to track the movement of blocks of applied speckle patterns on the surface of the specimen (see [63]).

Displacements and strains are determined by correlating the position of pixel subsets or blocks in the original and deformed image, normally based upon contrast (i.e., grey intensity levels). In order to identify if there is any movement between the two blocks there must be sufficient detail for it to be considered unique. It may be the case that the specimen or component already has a suitable level of surface features which can be imaged directly, but if not, some form of spray paint or coating or scratches on the surface can be used.

For best results a unique surface finish and a good distribution of intensity values must be obtained. This can be achieved by spraying the surfaces to be inspected with black, grey, and white paint to produce a random specular pattern for the image correlation. The deformations of the specimens are then calculated by correlating the positions and displacements of pixel subsets or blocks in the original and deformed image to produce a deformation vector map. This is then processed further to produce a full-field strain map. Calculation of 3D deformations is possible if two or more cameras are used. Calibration of the image by using a calibration plate is straightforward.

Mechanical testing

The stiffness, strength, creep deformation and fatigue life measured form only part of the useful data that are required when assessing the performance of joined systems. The mode of failure in adhesive joints (i.e., cohesive failure in the adhesive or interfacial failure) and degree of cohesive and adhesive failure should be recorded for each specimen. Similarly, the fracture behaviour should be recorded for creep rupture tests conducted on welded joints noting the degree of brittle and ductile fracture and location of failure. Fracture can occur within either the welded region or in the parent material adjacent to the weld. Optical microscopy or scanning electron microscopy may be required to analyse the fracture morphology, particularly if failure is close to the adhesive/adherend interface or at the interface between the welded region and parent material. Adhesive standards often recommend that results from specimens where failure occurs in the adherend should be discarded and replaced. However, unless the reason for adherend failure can be traced to inherent flaws in the test adherend that are unlikely to be replicated in the bonded component then these tests can still provide some valuable information.

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

Number of test specimens: A minimum number of five specimens should be tested at each stress level. If a greater precision of the mean value is required, then the number of specimens tested should be increased (see ISO 2602 [64]).

Specimen dimensions: Specimen dimensions and crack length need to be accurately measured, as small measurement errors can translate into large variations in strength or fracture toughness, particularly if the calculation includes squares or cubes terms of the measured parameter (see Table 1). The uncertainty in strength or fracture toughness calculation is compounded where there is more than term (i.e., width, thickness, and crack length, etc), each with an associated uncertainty. The bond-line thickness, being a very small dimension, tends to be the dimension where accuracy and precision of dimensional measurement are most critical. Measurements at different locations should be carried out to check the uniformity of bond-line thickness.

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

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

Table 1. Associated uncertainty with measurement error (percentage)

Test speed and frequency: Adhesives and thermoplastics are viscoelastic materials; that is their mechanical properties (strength and stiffness) are sensitive to the rate at which they are loaded (or more accurately the strain rate). Standards relating to testing of adhesive and welded joints infrequently specify the speed/rate or frequency of testing required. 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 quasi-static test trials to failure are recommended to ascertain the equivalent frequency required to match 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.

Specimen geometry and adherend effects

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

Adherend Thickness/Overlap Length	Load/Width (N/mm)
CR1 Mild rolled steel	
1.5 mm thick/12.5 mm overlap	334 ± 11
2.5 mm thick/12.5 mm overlap	354 ± 10
2.5 mm thick/25.0 mm overlap	428 ± 38
2.5 mm thick/50.0 mm overlap	633 ± 63
5251 aluminium alloy	
1.6 mm thick/12.5 mm overlap	191 ± 14
3.0 mm thick/12.5 mm overlap	325 ± 28
6AL-4V titanium alloy	
2.0 mm thick/12.5 mm overlap	457 ± 52
Unidirectional T300/924 carbon/epoxy	
2.0 mm thick/12.5 mm overlap	369 ± 41
Plain woven fabric (Tufnol 10G/40)	
2.5 mm thick/12.5 mm overlap	275 ± 28
2.5 mm thick/25.0 mm overlap	454 ± 27
2.5 mm thick/50.0 mm overlap	511 ± 32
5.1 mm thick/12.5 mm overlap	327 ± 27

Table 2. Failure load per unit width for epoxy bonded single-lap joints

The strength, and hence fatigue resistance of bonded joints is dependent on the adherend material, adherend thickness, bond-line thickness and bond length. Table 2 compares the failure load per unit width (N/mm) for various single-lap joint configurations and materials bonded with an epoxy adhesive [65-67]. Table 3 demonstrates the sensitivity of single-lap strength on bond-line thickness and bond length.

The joints consisted of mild steel sections bonded with an elastomer adhesive. Small variations in bond-line thickness can result in significant changes in bond strength. For comparative studies, careful consideration should therefore be given to ensuring that the stress and strain distributions (i.e., maximum peel and shear stresses at the ends of the joint) for different systems are at least similar.

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

Table 3. Failure load (N) for mild steel/elastomer single-lap joints

Basic design considerations for maximising the static strength and fatigue performance of adhesively bonded joints include:

- Increasing the overlap area results in higher joint strength. The failure load is directly proportional to specimen width. Increasing the joint width by a factor of 2 will double the failure load, however increasing the bond length by a factor of 2 will not necessarily result in a two-fold increase in bond strength (see Table 2).
- Increasing either the adherend stiffness (i.e., elastic modulus) or adherend thickness results in an increase in load-bearing capacity of the single-lap joint. The use of stiff or thick adherends will minimise peak stress levels and yield a more uniform adhesive stress distribution. However, the use of absolutely rigid adherends will not prevent the formation of stress concentrations at the bond-line.
- The total overlap length must be sufficiently long to ensure that the shear stress in the middle of the overlap is low enough to avoid creep. Short overlaps can result in failure through creep-rupture. It is recommended that the overlap length is ~10 times the minimum adherend thickness to ensure a uniform shear distribution.
- Increasing the overlap lengths beyond this value does not result in substantial increases in static and fatigue performance. The low stress region in the middle of a long overlap contributes to joint strength by providing elastic restoring force or reserve. It is recommended to maximise bond area. Longer overlap lengths are highly desirable (provided cost and weight penalties are not too high).
- Minimise shear and peel stress concentrations - shear and peel stress concentrations present at bond-line ends can be reduced by the use of a combination of tapered or bevelled external scarf or radiused adhesive fillets. Significant increases in the joint strength compared with square-ended bond-lines can be achieved. It is recommended that the taper ends of lap joints should have a thickness of 0.76 mm and a slope of 1/10.

- Ensure the joint is loaded in the direction of maximum strength of the adherend. The bonded joint needs not only to be loaded in the direction of maximum strength, but also loads in the weak directions need to be minimised.
- Maintain a uniform bond thickness and wherever possible join identical adherends to minimise skewing of the peak and normal stresses, and to minimise thermal residual stresses and bending stresses due to differences in coefficient of thermal expansion (CTE) values of the adhesive and adherends.
- In the case of fibre-reinforced laminates, avoid interlaminar shear or tensile failures of composite adherends. Also, ensure the laminated adherend is symmetric, thus ensuring the coupling stiffness components of the laminate are zero (i.e., no twisting).
- Mode of loading has a significant affect on joint strength and long-term performance (i.e., compression > shear > tension). The presence of tensile or peel stresses at the end of overlap regions will reduce joint strength.

Additional points to note regarding adhesive joint testing are listed below.

- The stress distribution within the bond-line tends to be non-uniform in a majority of test configurations with stress concentrations existing at the bond-line ends. Premature failure will often occur as a result of these stress concentrations.
- Generally, the time taken for environment effects to become apparent increases with joint size, thus test joints with small bond areas, or with large bond-line perimeters compared with the bonded area are preferred for accelerated testing.
- The accuracy and reliability of displacement measurements are often in question as the magnitude of displacements is often small.

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Chapter 3

Fatigue testing

- Introduction
- Constant amplitude cyclic loading
- Fatigue life assessment
- Welding parametric effects on fatigue performance

Introduction

This Chapter gives guidance on cyclic fatigue testing of adhesively bonded and welded joints, and is primarily concerned with structural adhesives and thermoplastics, and constant amplitude and frequency tension-tension fatigue loading conditions. It is recognised that for many engineering applications, variable amplitude, and frequency loading (i.e., spectral loading) is more realistic of what happens in practice, and that analysis and life prediction for spectral loading is more complicated than constant amplitude and frequency regimes. It should also be recognised that the stiffness of plastic adherends may be considerably less than structural materials, such as metals and fibre-reinforced plastics (FRPs) encountered in bonded structures. This may have relevance when monitoring stiffness changes and strain control during testing.

Constant amplitude cyclic loading

Minimum stress, σ_{MIN}
Maximum stress, σ_{MAX}
Stress range, $\Delta\sigma = \sigma_{MAX} - \sigma_{MIN}$
Stress amplitude, $\sigma_A = \Delta\sigma/2 = (\sigma_{MAX} - \sigma_{MIN})/2$
Mean stress, $\sigma_{MEAN} = (\sigma_{MAX} + \sigma_{MIN})/2$
Stress ratio, $R = \sigma_{MIN}/\sigma_{MAX}$
$0 \leq R \leq 1$ for tension-tension loading
$-\infty \leq R \leq 0$ for tension-compression loading
$1 \leq R \leq +\infty$ for compression-compression

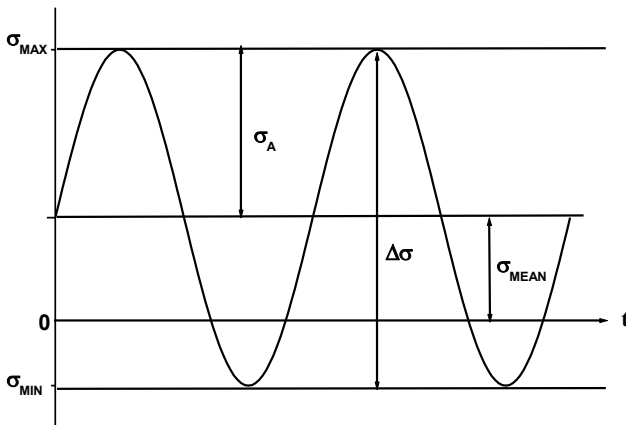


Figure 6. Nomenclature for stress parameters for constant amplitude cyclic loading

The principle of the method, as described in ISO 13003 [68], is that a continuously alternating mechanical load or displacement is applied at a constant frequency to the specimen under test until the specimen either fails or reaches a certain number of fatigue cycles (fatigue life). This load is applied in combination with a specified mean load (which may be zero) - see Figure 6 for the nomenclature for stress parameters for constant amplitude cyclic loading. By testing specimens at each of several percentage levels of the ultimate stress or strain, a plot of the stress-strain versus number of fatigue cycles can be constructed. This plot provides information on the fatigue life of the material or the number of fatigue cycles the material can sustain at a certain stress-strain level before failure occurs.

The test may be carried out at a constant stress (load) amplitude, constant strain amplitude or constant displacement amplitude. The test method, specimen geometry, dimensions and calculations are the same as those used in the equivalent test mode under static (monotonic) loading conditions. The fatigue properties of adhesively bonded joints are a function of the joint geometry and adhesive, and therefore cannot be determined from the intrinsic properties of the adhesive. It is therefore necessary to conduct cyclic fatigue tests on representative joints to those to be used in service.

Fatigue regimes

Fatigue regimes that exist range from compression-compression ($1 \leq R \leq +\infty$), tension-compression ($-\infty \leq R \leq 0$) to tension-tension ($0 \leq R \leq 1$) – see Figure 2 in ISO 13003. The results presented in this guide relate to a sinusoidal cyclic waveform only (Figure 6), but it is noted here that other waveform shapes are commonly used (i.e., triangular, square, sawtooth, etc.) and that proprietary test machine control software allows the user to input bespoke waveforms. The selection of the most appropriate fatigue regime to use is directly linked to the material application requirements and anticipated stress field.

Test frequency

Fatigue data are normally obtained at the highest frequency possible to minimise the duration of tests. Restrictions on test frequency can arise from test equipment limitations (response time), time dependent processes and hysteretic (self-generated) heating. Hysteretic heating, which increases with increasing load and frequency, can result in thermal softening of the polymer (and FRP adherends), adversely affecting the fatigue performance of the material and causing erroneous stiffness and life expectancy measurements. The amount of heat generated in a material under fatigue loading will depend on several factors, including stress/strain amplitude, specimen displacement range (stroke), test frequency and the ability of the material or component to dissipate heat.

The temperature rise of the specimen surface is normally limited to 10 °C, but this depends on the sensitivity of material properties to rises in temperature. It is recommended that even where prior knowledge of test frequency versus heat rise is known for a material, a thermocouple should be attached to the surface of initial test specimens to monitor the degree of hysteretic heating.

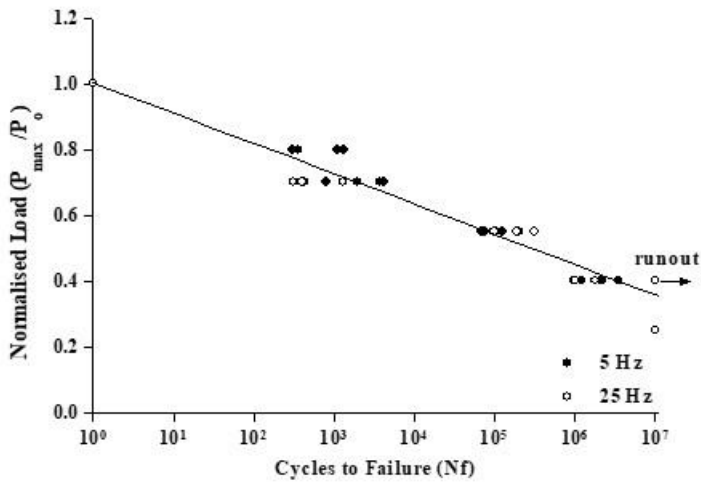


Figure 7 Normalised S-N curve for mild steel/epoxy single-lap joints

It is recommended that the temperature rise of the material surface be kept to a minimum. It may be necessary to stop testing to allow the specimen to cool. Alternatively, the test could be carried out in an environmental cabinet with a thermocouple attached to the specimen surface for monitoring and controlling the temperature of the test specimen, although the cooling rate may be too slow to be practical. Thermal imaging equipment can be used to monitor surface temperature, although the latter is beyond the budget of most industrial facilities. The temperature resolution is ~1 °C for the two methods. Tests should be carried out at each stress level to ensure the temperature rise is kept to less than 10 °C, and if necessary, the test frequency should be reduced to prevent over-heating. For temperature sensitive materials, it may be necessary to set a lower limit on the temperature rise. These comments do not apply to any rapid temperature rise associated with final failure.

Reliable data can be obtained at high frequencies provided the stress levels are low. Test frequencies of the order of 10 to 30 Hz can result in substantial heating, particularly in the grip regions. The upper frequency limit will be dependent upon the thermal conductivity of the joined system, mode of loading and specimen size. Trials may be necessary to determine the upper frequency limit. The results presented in Figure 7 for single-lap joints show no evidence of a self-heating effect, and hence the merging of fatigue results for 5 Hz and 25 Hz.

It is unlikely that test frequencies of the order of 5 Hz (except for low amplitude vibration), or higher will be experienced in service. Fatigue resistance decreases with a reduction in frequency; the decrease is attributable to creep deformation. At a test frequency of 5 Hz, 10^7 loading cycles is equivalent to 23 days testing. Increasing the test frequency to 25 Hz reduces the test duration to less than 5 days.

Determination of ultimate material properties

To determine the relative stress/strain levels to use in a fatigue test the ultimate properties (i.e., strength, strain to failure, etc.) should first be measured at a loading rate equivalent to fatigue testing conditions (i.e., test frequency). When undertaking measurements of ultimate properties, it is important to recognize that stiffness and strength of polymeric materials are rate dependent. The fatigue test rate is defined as that resulting in failure in a time equivalent to $0.5 \times$ the cycle time. Mean values for ultimate properties should be determined from tests undertaken on at least 6 specimens.

Number of test specimens

For the determination of the lifetime diagram, five specimens should be tested at a minimum of four stress levels, or preferably five stress levels (80%, 70%, 55%, 40% and 25% of the fatigue rate ultimate strength). A minimum of 6 fatigue tests should be conducted at each stress level (unless otherwise specified). It is advisable to increase the number of tests per level when carrying out statistical analysis for generating design data.

Specimen preparation and test conditions

Specimen preparation, specimen geometry, loading arrangement and environmental test conditions should be the same as those employed for the monotonic tests (see chapter 2). The applied conditions (i.e., peak load, strain and/or displacement data) when stabilized should be recorded throughout the duration of test.

Analysis of fatigue data

Cyclic fatigue data is generally presented in the form of a stress-cycle (S - N) diagram (i.e., a plot of the fatigue life (number of cycles to failure N_f at various levels of fluctuating stress). For inter-comparative purposes, fatigue strength data (see Figure 7) are normalised with respect to the ultimate static strength P_{ULT} (or P_0) of identically conditioned specimens measured at an equivalent loading rate to the test frequency. The uncertainty in life expectancy at any stress level is large (typically an order of magnitude).

Joint stiffness: In addition to recording the number of cycles to failure, the stiffness properties of specimens can be monitored throughout the test at each logged cycle. This is carried out to see whether changes in stiffness occur due to growth of the damage throughout the fatigue tests, and if there are changes in stiffness, where in relation to the failure cycle these changes

occur. The simplest way of calculating the modulus is to use the minimum and maximum stresses and corresponding strain levels derived from the recorded actuator positions. Whilst this method is relatively simple, the fact that actuator positions are used to calculate strains can lead to errors due to the compliance of the loading train. Using devices such as clip gauge extensometers, LVDTs and strain gauges, can eliminate compliance errors but ensuring that these types of devices do not detach or de-bond from specimens during the fatigue test is crucial (see Chapter 2).

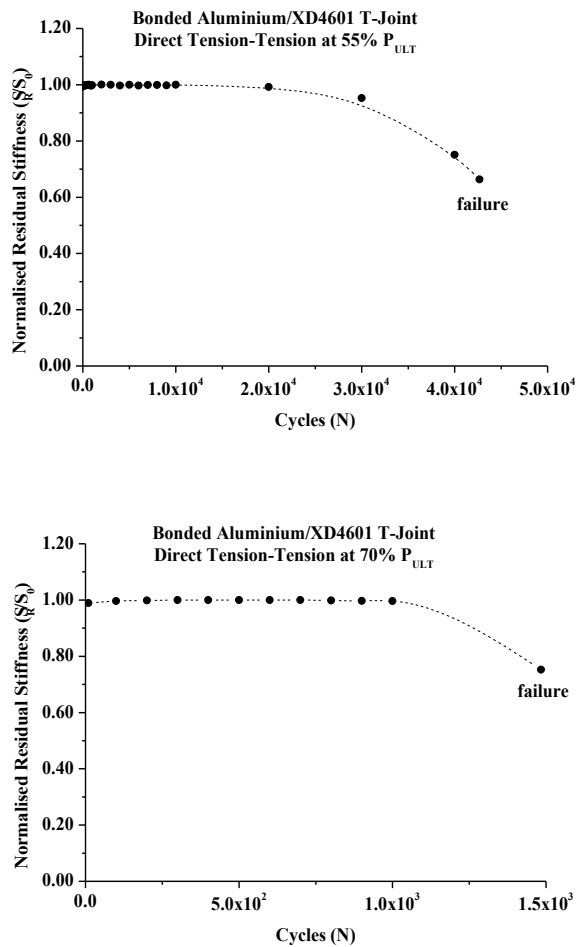


Figure 8. Normalised residual stiffness for fatigue loading of aluminium T-joints

The joint stiffness (i.e., dynamic compliance) will often decrease with the onset of damage within the joint (Figure 8). Ultimate failure is marked by a rapid reduction in joint stiffness and an increase in the loss or damping factor ($\tan \delta$). The loss factor $\tan \delta$ is the ratio between the storage modulus E' and the loss modulus E'' (i.e., $\tan \delta = E'/E''$) where δ is the phase angle between dynamic load/stress and the dynamic displacement/strain (see Figure 9) [59, 68]. The storage modulus is proportional to the maximum energy stored during a loading cycle and represents the stiffness of the joint. The loss modulus is proportional to the energy dissipated (lost) during one loading cycle.

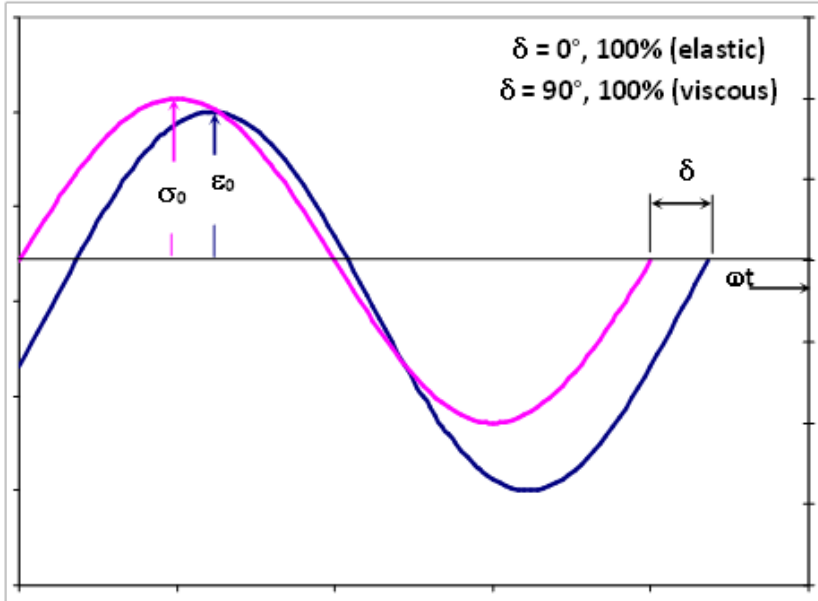


Figure 9. Out-of-phase stress strain response for viscoelastic material, $\delta = 20^\circ$ [68]

The stress σ and strain ε are given by the following relationships (see Figure 9):

$$\sigma = \sigma_0 \sin(\omega t + \delta); \quad \varepsilon = \varepsilon_0 \sin(\omega t) \quad (1)$$

Where ω is the period of strain oscillation, t is time and δ is the phase angle in radians.

The storage modulus E' and the loss modulus E'' are given by:

$$E' = \frac{\sigma_0}{\varepsilon_0} \sin \delta; \quad E'' = \frac{\sigma_0}{\varepsilon_0} \cos \delta \quad (2)$$

For a purely elastic material, the mechanical energy stored during loading is returned completely when the specimen is unloaded. This generates load/displacement or stress/strain curves for the material, which are completely in phase with each other (i.e., $\delta = 0^\circ$), producing no hysteresis (see Figure 10). At the other extreme a purely viscous material (i.e., $\delta = 90^\circ$), which exhibits no elasticity only damping, results in all the energy being dissipated and the load/displacement or stress/strain curves being 90° out of phase with each other. Materials that fall between these two categories exhibit a phase lag (Figure 9) and are classified as viscoelastic.

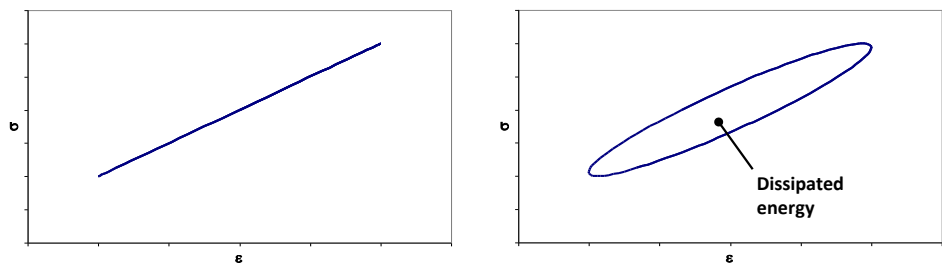


Figure 10. Hysteresis (stored energy): (left) elastic and (right) viscoelastic response [68]

When viscoelastic materials are subjected to fatigue or cyclic motion a proportion of the mechanical energy per cycle is converted to thermal energy (heat) with the rest of the energy being stored. A hysteresis loop develops whose area is equal to the dissipated energy per cycle (Figure 10). The stiffness and hysteresis can be derived mathematically – see [68].

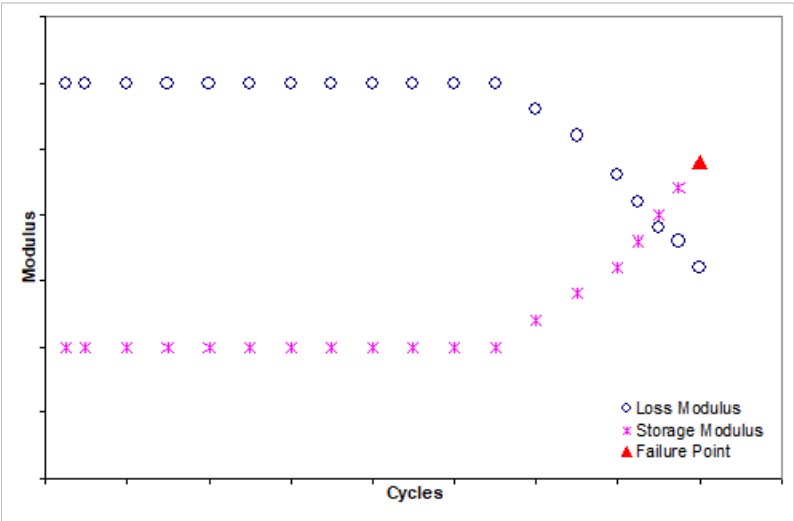


Figure 11. Storage and loss modulus changes [68]

Analysis of data on specimens that have been fatigued to failure should show that over time the stiffness of the material decreases towards failure with the energy loss of the material becoming greater as damage accumulates and friction causes material heating (Figure 11). As damage accumulates within the material, the slope of the ellipse will decrease and the area within the ellipse will increase. For a considerable proportion of the fatigue life (50-60%), stiffness and hysteresis will remain reasonably constant (see Figure 8). There will be a rapid change in stiffness and hysteresis within the last few hundred cycles. Although measurement of global stiffness is relatively straightforward using a linear voltage displacement transducer (LVDT) or strain gauges, it is often difficult to obtain an accurate measurement of the hysteresis. Localised deformation measurements can be obtained using strain gauges bonded to the specimen surface. These gauges if strategically located at critical stress regions, may indicate the onset of localised damage.

Fatigue (endurance) limit is the maximum fluctuating stress a material can endure for an infinite number of cycles (Figure 12). Under constant amplitude loading conditions, most materials or structures seem to exhibit a plateau in the stress-cycle curve, which typically occurs at $N > 10^6$ cycles. The plateau level corresponds to the fatigue or endurance limit. Below this limit, the material or structure can in principle be cycled indefinitely without causing failure. Recent work conducted at NPL on unidirectional glass/fibre-reinforced epoxy suggests that given sufficient time, fatigue failure would occur at stress levels less than 25% of the ultimate strength. This may also apply to adhesive and welded joints.

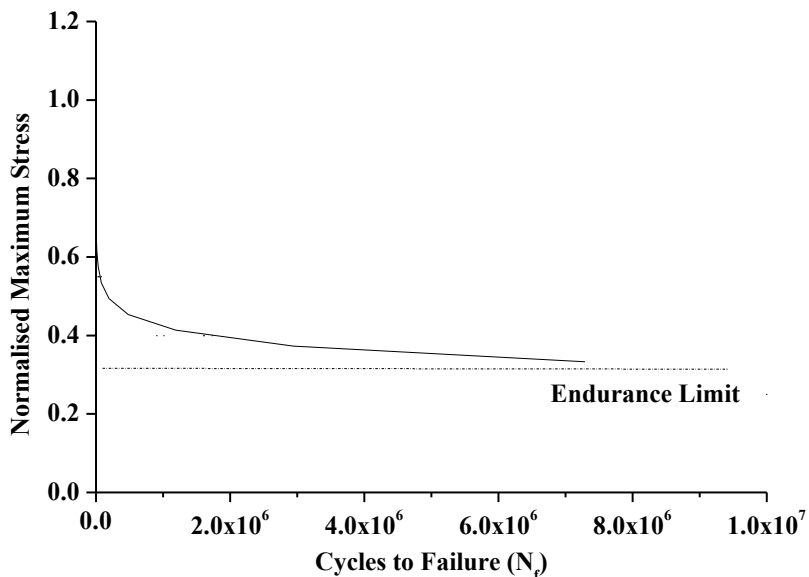


Figure 12. Typical S-N curve with fatigue or endurance limit

Fatigue life assessment

In order to compare fatigue performance, fatigue strength data are often normalised with respect to the ultimate static strength P_{ULT} of identically conditioned specimens measured at an equivalent loading rate to the test frequency. Normalised $S-N$ curves for bulk adhesives and bonded joints (see Figures 7 and 13) can be approximated by the following relationship:

$$P_{MAX}/P_{ULT} = 1 - k \log N_f \tag{3}$$

P_{MAX} is the maximum load applied to the specimen, P_{ULT} is the ultimate strength of the joint and N_f is the number of cycles to failure.

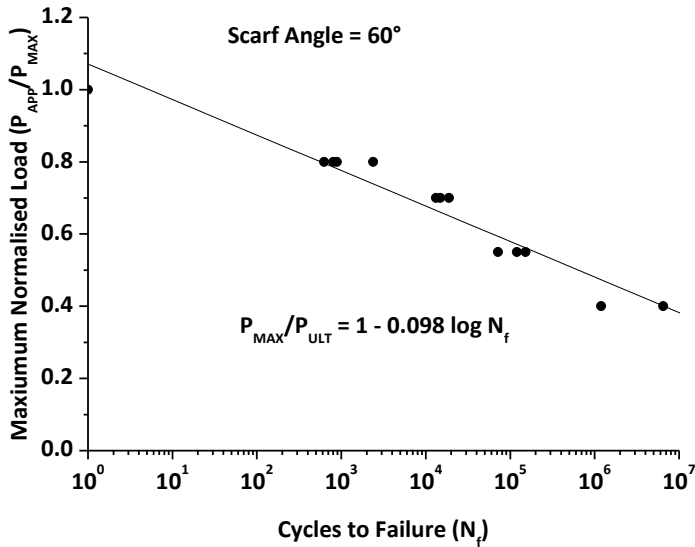


Figure 13. Normalised $S-N$ curve for a mild steel/epoxy scarf joint ($R = 0.1$, $f = 5$ Hz)

Fatigue performance of an adhesive joint can be approximated by (rule of thumb) Equation 3 where k the fractional loss in strength per decade of cycles is a measure of fatigue resistance of the joint. The lower the k value the better the fatigue performance. The value of k is dependent on joint geometry and stress ratio. Table 4 shows typical k values for several metal and composite joints bonded with epoxy adhesives. A scarf joint with an angle of 15° with respect to the horizontal where failure is dominated by shear stresses has a far better fatigue performance than tests where peel or hydrostatic tensile stresses are the major cause of failure (e.g., T-peel joints and 60° scarf joints (see Figure 13)). Cyclic fatigue performance is highly sensitive to bond-line thickness.

Joint Type	k
Scarf (steel adherends with 15° angle)	0.063
Thick adherend shear test (steel adherends)	0.063
Double-lap (titanium adherends)	0.075
Double strap (aluminium adherends)	0.088
Single-lap (mild steel adherends)	0.093
Double-lap (woven fabric)	0.097
Scarf (steel adherends with 60° angle)	0.098
T-joint (aluminium adherends)	0.104
T-peel (mild steel adherends)	0.130

Table 4. Typical k Values for bonded joints ($R = 0.1$ and $f = 5$ Hz)

A bond-line thickness of the order of 0.2-0.3 mm tends to have far superior fatigue resistance than thicker bond-lines (> 0.5 mm); probably because of lower porosity within the adhesive. An increase in temperature or moisture ingress will compromise the fatigue performance of polymers and adhesive joints (k can be expected to increase).

Constant-life diagrams are often used to represent the effects of mean stress and stress amplitude on fatigue performance of adhesive joints. Different combinations of normalised stress amplitude, $\Delta P/P_{ULT}$, and the normalised mean stress, P_{MEAN}/P_{ULT} , can be plotted to give constant fatigue life curves. Figure 14 shows normalised stress-amplitude plots for different mean stress values. The stress values are normalised with respect to the ultimate tensile strength, σ_{UTS} , of the material. In principle, the curves should converge to the static strength of the material on the mean stress axis (i.e., when the mean load is increased to the static strength then no amplitude is required to cause failure).

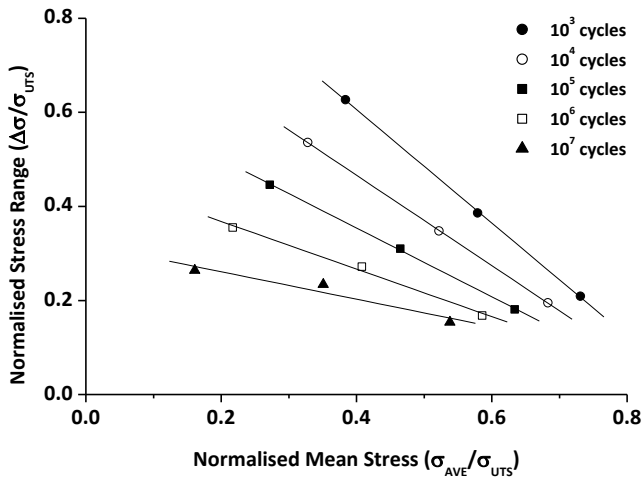


Figure 14. Stress amplitude-life plots for different mean stress values

Several models have been suggested for determining stress amplitude-life plots of polymeric materials [69]. A Goodman type curve has been shown to be a valid method of representing the effect of mean stress and stress amplitude on fatigue performance of single-lap and T-peel joints [70]. The Goodman relation is given below [69]:

$$P_A = P_{FS} \left(1 - \frac{P_{MEAN}}{P_{ULT}} \right) \quad (4)$$

P_A is the stress amplitude (for a non-zero mean stress), P_{FS} is the fatigue strength (for a fixed life), P_{MEAN} is the mean stress and P_{ULT} is the ultimate strength of the material.

An alternative approach for estimating fatigue life is to apply a model originally developed for fibre-reinforced plastics known as the normalised life prediction model [71]:

$$\frac{(1 - R)\sigma_{MAX}^*}{2 - (1 + R)\sigma_{MAX}^*} = \left(\frac{1}{2N_f} \right)^{\frac{1}{n^*}} \quad (5)$$

The fatigue strength is normalised with respect to the ultimate (static) strength in the loading direction:

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{ULT}} \quad (6)$$

For tension-tension and compression-compression fatigue, respectively:

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{UTS}} \quad (7)$$

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{UCS}} \quad (8)$$

σ_{UTS} and σ_{UCS} are the ultimate tensile and compressive strengths, respectively.

The value of n^* is derived from fatigue tests carried out at three different stress ratios R (e.g., 0.1, 0.5 and 0.75) and at different values of maximum stress (i.e., 80%, 70%, 55%, 40% and 25%).

For tension-tension fatigue ($|R| \leq 1$), the value of n^* has been determined for unidirectional glass/epoxy and bulk epoxy adhesive DP460 and found to be approximately 10 and 12, respectively.

The value of n^* for steel scarf joints bonded with DP460 epoxy adhesive with a 60° scarf angle with respect to the horizontal was observed to be ~10. The higher the value of n^* the better the overall fatigue resistance of the polymeric system.

Welding parametric effects on fatigue performance

Cyclic fatigue behaviour seems to be sensitive to welding conditions with fatigue resistance at elevated stresses decreasing with an increase in welding temperature. Table 5 compares the average fatigue life obtained from tension-tension fatigue tests conducted at a stress level of 70% of the ultimate tensile strength. The stress ratio was 0.1 and the test frequency 5 Hz. Five test specimens were tested per batch. Fatigue performance is highly sensitive to the presence of the weld as shown by the large decrease in fatigue performance. The uncertainty in fatigue life data was typically $\pm 25\%$ for both the parent and welded materials.

Material	Average Fatigue Life (Cycles)
Parent	45,581
Welded	
198 °C	8,141
216 °C	5,637
232 °C	5,519
250 °C	4,000

Table 5. Average fatigue life of welded and unwelded polypropylene

Table 6 indicates that the differences between the tensile strength obtained for polypropylene for different weld temperatures is minimal. The difference in tensile strength between parent material and welded polypropylene is ~12% at 23 °C and 6% at 50 °C.

Material	Tensile Strength (MPa)
Parent	27.0 \pm 3.0
Welded	
198 °C	23.7 \pm 0.3
216 °C	23.6 \pm 0.4
232 °C	23.1 \pm 0.3
250 °C	23.0 \pm 0.4

Table 6. Tensile strength of welded and unwelded polypropylene

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Chapter 4

Creep testing

- Introduction
- Creep rupture
- Creep testing
- Creep behaviour
- Procedure for modelling creep in polymers

Introduction

Creep is the increase in strain or deformation of a material with time when the material is subjected to a constant load for an extended period of time (i.e., time-dependent deformation). The change of strain at any time increases with load (stress), stress state, temperature and relative humidity. Viscoelastic materials, such as thermoplastics and adhesives, can undergo creep deformation at relatively low stress levels (well below the ultimate strength of the material) and low temperatures (i.e., room temperature - referred to as cold flow) compared with many structural materials (metals, glass and concrete). This can lead to considerable reduction in life expectancy of the component. Strain (or deformation) increases with load, temperature, relative humidity and time.

It is therefore important to be able to predict the effects of long-term loading on deformation and failure behaviour. Confidence in predictions requires the use of models and test methods that accurately characterises the deformation behaviour of the polymeric materials in particular joined systems. This sChapter provides guidance on creep testing of polymers, and adhesive and welded joints. Many of the aspects of specimen preparation and testing have been covered in Section 2.

Creep rupture

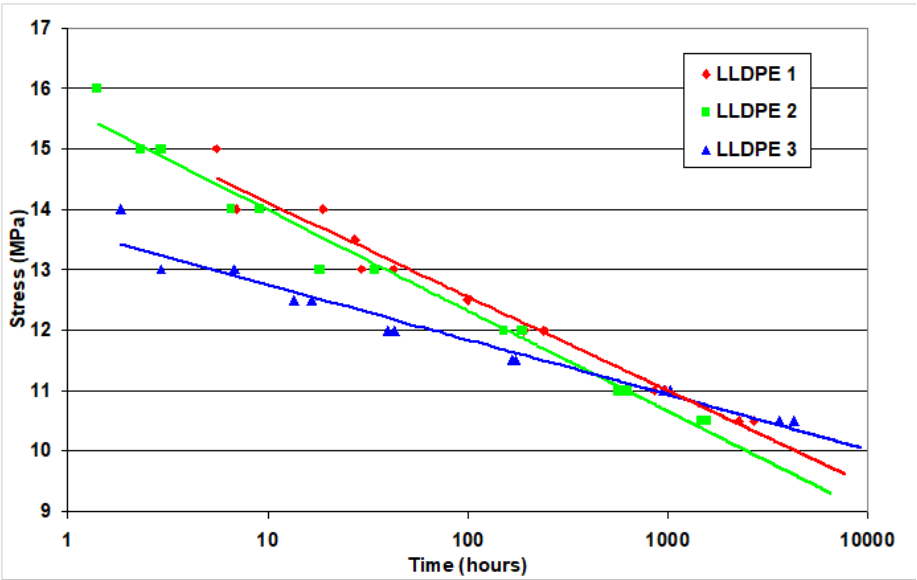


Figure 15. Time to failure of three grades of LLDPE full notch creep test (FNCT) specimens [38]

Creep rupture (or static fatigue), which refers to time-dependent failure of the material when subjected to constant load, is used as a measure for gauging the relative resistance of the material to chemical environments. It can also be used to assess the quality of welds. Tests are conducted at several stress levels (i.e., 80%, 60%, 40% and 20% of the ultimate strength) and the deformation and time-to-failure are recorded. A plot is produced of applied stress versus time-to-failure (Figure 15). It is recommended that six specimens be tested per stress level. The less resistant a material is to a particular environment, the more rapid the drop in the rupture stress time curve. For creep rupture tests, the duration of the test is taken from the moment the test load is reached until the specimen fractures.

For a given applied load, the stress rupture time decreases with increasing humidity and temperature, or chemical concentration. The Full Notch Creep Test (FCNT) [61] shows differences in the ESC resistance, but can be misleading if creep tests are not conducted for a sufficient length of time (up to 1 year). Figure 15 compares the time to failure of three grades of linear low-density polyethylene (LLDPE) in which there is a crossover point at approximately 1,000 hrs – see NPL Measurement Good Practice Guide No. 103 [38].

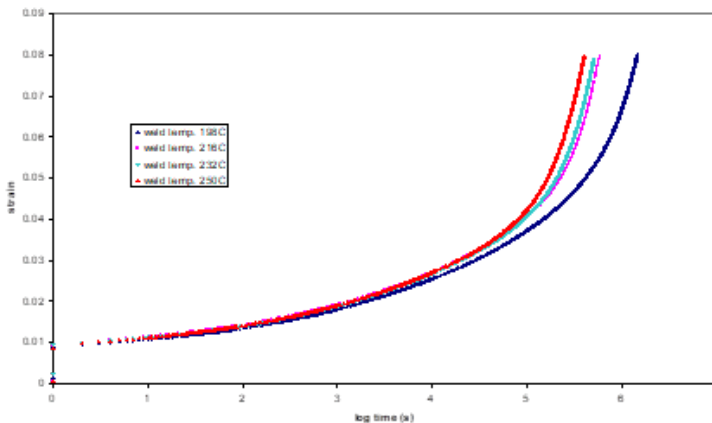


Figure 16. Tensile creep of welded PP for different hot plate temperatures (applied stress of 13.6 MPa)

In thermoplastic systems that have been well fused (bonded), there will be minimal difference in creep behaviour at moderate stresses (≤ 9 MPa for polypropylene). At higher stresses, however, differences in creep behaviour of hot plate welded polypropylene becomes apparent with the creep rate increasing with increasing weld temperature. Figure 16 compares the tensile creep behaviour of hot plate welded polypropylene tested at 13.6 MPa and 23 °C. The hot plate temperature was varied (198 °C, 216 °C, 232 °C and 250 °C) whilst fusion pressure, hot soak time and cooling time were kept constant. Fusion pressure was 0.1 Nmm⁻², hot soak time 135 seconds and cooling time 6 minutes.

Creep testing

The information given below is intended to complement BS EN 12814-3 [49], ISO 899-1 [50], NPL Measurement Good Practice Guide No. 2 [29] and Chapter 2.

Test apparatus and test conditions

Loading system and gripping device: The creep rig should have a rigid frame (no deformation of the test frame is allowed within the applied loading range), which allows the load to be applied smoothly, without causing transient loading, and be capable of maintaining the load to within $\pm 1\%$ of the required load. Dead weight cantilever loading frames are recommended with the construction of the link between the lever arm and the test specimen designed so that the lever arm is independent of the arm – see NPL Measurement Good Practice Guide No. 2 [29] and BS 1610 [72].

The load train should be as short and as stiff as possible (i.e., no universal joints included). Steps should be taken to ensure good axial alignment, and to avoid bending and torsion forces acting on the specimen. Good alignment is critical, particularly for bonded systems (see Chapter 2). The gripping mechanism should be sufficient to prevent slippage within the grips, but without damaging the material in the grip zone that could cause premature failure at high stresses. Wedge action grips are preferred. Self-locking grips that allow the specimen to move as the load increases are unsuitable for creep tests. It is important when handling and testing adhesively bonded and welded specimens that care is taken to avoid any lateral loads being placed on the specimens, and that overloading is also avoided. There are no recommended loads, however ISO 11403-1 [73] recommends 20%, 40%, 60% and 80% of the ultimate load [23].

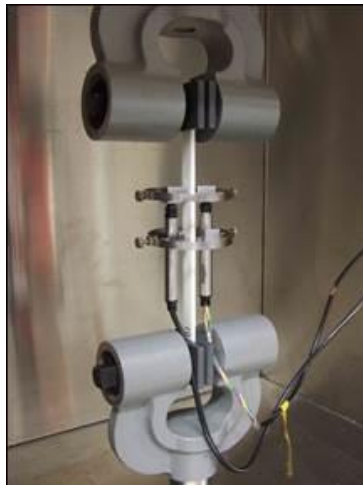


Figure 17. Tensile creep specimen with extensometers

Extension measurement: The extension gauge-length should cover the region of interest. Extension can be measured using either contact or non-contact devices. It is essential that when attaching contact extensometers that the devices do not influence material behaviour or cause damage. Extension should be measured using two separate lightweight extensometers, preferably with blunted knife-edges, applied either side of the specimen to check alignment (see Figure 17). The readings from the two extensometers are averaged. The accuracy of the extension-measuring device needs to be better than $\pm 1 \mu\text{m}$. For low absolute strain levels encountered at small stresses or during the initial period of creep tests the accuracy generally should be better than $\pm 0.25 \mu\text{m}$.

Non-contact or optical extensometers (e.g., video extensometers) are available, which avoid contact damage and can be used up to failure, since there is no possibility of damage to the extensometer. Video extensometers are not particularly suited to measuring small strains (e.g., movements of a few mm), which limits their applicability to systems involving small deformations, such as structural adhesive joints. There are however no restrictions on the upper limit. This is a convenient method for determination of strains above 10% (i.e., suitable for use with rubbers or thermoplastics). Video extensometers are recommended for creep rupture tests. The gauge length is marked on the specimen either by attaching metal clips with scratched on gauge marks or by ruling the gauge marks with an inert, thermally stable paint.

Strain gauges can be used, but the use is limited. Care has to be taken to ensure the strain measurements obtained are reliable, as strain gauges tend to locally stiffen polymers. The strain measured tends to be lower than that expected for a given load [74]. For many polymeric materials, it is difficult to produce a reliable adhesive bond between the gauge and the polymer. Strain gauges are not particularly suited for creep rupture tests where strains can be very high (5-10%). Before embarking on using strain gauges it is advisable to obtain specialist advice.

Strain and load should be recorded at regular intervals or at times that form regular intervals of $\log t$, where t is duration. A data acquisition system is recommended for recording load and displacement as a function of time. The time recoding equipment should be accurate to within $\pm 0.1 \%$ or $\pm 2 \text{ s}$. The data collection system should also be programmed to set the creep time to zero at the instant of the load application. It is recommended in ISO 899-1 [50] that measurement schedule should be:

- 1, 3, 6, 12 and 30 min and then 1, 2, 5, 10, 20, 50, 100, 200, 500, 1,000 h, etc.

More frequent checks may be required if discontinuities are suspected. Higher logging rates are generally required at the start of the creep test. It may be necessary to remove early points to remove the effect of load application.

Test conditions: The creep behaviour of polymeric materials is sensitive to temperature, and therefore it is critical to keep the temperature of the specimen constant to within $\pm 0.5^\circ\text{C}$, which should be recorded regularly. This also applies for tests performed at elevated temperatures. Creep tests should be carried out in a temperature-controlled chamber. The humidity should also be controlled. Strain repeatability for creep tests performed on the same specimen at low stresses should be within $\pm 2\%$.

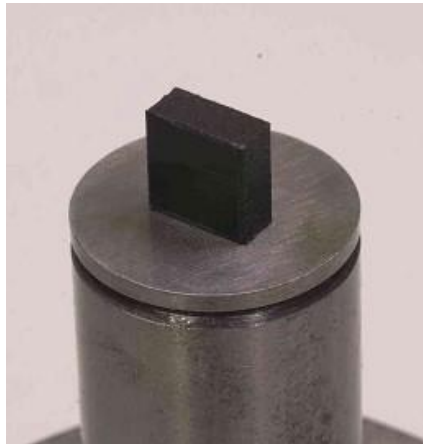
For material qualification purposes, it is recommended that for each stress level and temperature selected that at minimum of 6 welded and 6 unwelded specimens be tested. For modelling purposes, it may not be necessary to conduct that many tests in order to obtain reliable estimates for the creep parameters. The resistance to slow crack growth of the material can be determined using the FNCT – see [49, 61].

Loading modes

It is possible to generate creep compliance curves for all three modes of loading (i.e., tension, compression and shear) on polymers (including adhesives) and welded thermoplastics. This section provides a brief description of test methods suitable for generating creep data for the three loading modes.



Dumbbell Tensile Specimen



Compression Test Specimen

Figure 18. Tensile and compression test specimens

Tension: Tensile specimens typically consist of a waisted section with parallel sides (Figure 18) to facilitate strain measurements and to ensure failure occurs within the gauge-section away from the gripped ends. ISO 527-2 [75] specifies a method for determining the tensile properties of polymers. Modulus is determined over the strain range 0.05% to 0.25% (provided the region on the stress-strain curve is linear).

Strain distribution along the gauge section will invariably be non-uniform. In fact, rapid and large deformation may occur at several locations away from the mid-section. This is particularly an issue for polymers exhibiting large strain to failure, such as toughened epoxies and thermoplastics. Localised yielding within the welded region can be expected to be predominant for welded specimens.

For welded specimens, the creep deformation measured is an average effect of the material within the gauge section, which includes the melted zone. Although the melted zone is small (i.e., narrow strip less than 0.1 mm), its influence is large due to a lower creep resistance. Consideration should be given to using a smaller gauge section commensurate with the size and contribution made by the welded region to creep deformation.

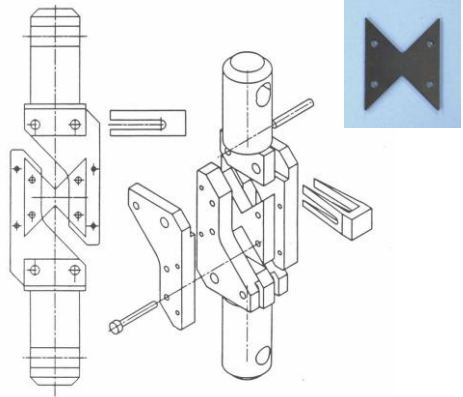
Compression: Creep tests under uniaxial compression are carried on small rectangular specimen (typically 12 mm long x 10 mm wide and approximately 4 mm thick) – see ISO 604 [76]. Specimens are loaded along the length direction between parallel platens. It is recommended that an alignment jig be attached to one of the platens to enable the platen faces to be set parallel using slip gauges. It is important that the two edges in contact with the top and bottom compression platens are flat and parallel to prevent buckling, which can result in premature failure and cause errors in strain measurements. Contact surfaces are also lightly oiled to reduce friction effects. Longitudinal strain is measuring using two contact extensometers applied directly between the loading platens mounted adjacent to the front and back surfaces of the test specimen. Nominal strains are derived from the original specimen length. Although the test geometry is used for determining Young's modulus, small displacement measurements tend to be inaccurate and difficult to repeat. Corrections need to be made to the strain values of around 0.005 arising from the initial displacements in the regions of the specimen/platen interface. Checks also need to be carried out to ensure specimens do not buckle.

Shear: Shear properties can be measured using either the Arcan or V-notched beam (ASTM D 5379 [77]) test methods. Both methods employ a notched (or a butterfly shaped) specimen (Figure 19) and can be used to generate stress-strain curves. The notches define the geometry, such that a predominantly pure stress state is established in the central region of the specimen. Strain can be measured using biaxial strain gauges bonded to both the front and back faces at $\pm 45^\circ$ to the applied load or contact extensometers mounted on the front and back surfaces as in the case for the Arcan method. Specimens are loaded in alignment fixtures (V-notched beam in compression and Arcan in tension).

The notch tip separation is 12 mm and radius of the notches 1.5 mm for both types of specimen. Although a special test fixture is required, testing is relatively straightforward. To minimise potential effects of out-of-plane movement or twisting of the specimen, it is recommended that the strain data used for determining shear modulus be the average of the indicated strains from each side of the specimen.



V-notched Beam Method



Arcan Method

Figure 19. Shear test methods

Measurements of shear modulus for both methods have been found to correlate well with other techniques, whereas the “apparent” shear strength value tends to be low. Failure often occurs as a result of high transverse tensile stresses on the cut out boundary, away from the gauge-section. Tensile failure is characteristic of brittle polymers (thermosets) with cracks propagating in an unstable manner along the plane of the principal tensile stress. For these materials, ultimate failure stress does not correlate with shear strength. Thermoplastic polymers (e.g., polyethylene and polypropylene) tested at room-temperature fail as a result of shear yielding along the notch root axis. Ductile materials can redistribute the tensile stress through plastic deformation, and therefore strain data can be obtained well beyond the shear yield stress of the material. Localised high compressive stress gradients may also produce plastic deformation in the vicinity of the loading points. Compliant materials, such as polyurethane, are prone to deform at high stresses. Measurements are invalidated unless constraints are applied the specimen clamps to maintain alignment.

Creep behaviour

At short creep times, the compliance curves exhibit slight non-linearity. This non-linear behaviour increases significantly with time under load. At higher stresses, the creep curves are observed to shift to shorter times. Figure 20 shows creep compliance curves for welded polypropylene at two different stress levels. The shift to shorter times is attributed to an increase in molecular mobility brought about by the application of elevated stresses that results in a reduction in the mean retardation time parameter t_0 . It is the enhanced mobility that gives rise to non-linear creep behaviour.

The value of t_0 is dependent not only on the magnitude of the applied stress, but also the stress state (i.e., tension, compression or multi-axial), physical age of the material, temperature, humidity and moisture content. The value of t_0 decreases with an increase in temperature and humidity, and moisture content of the polymer, and conversely increases with physically ageing of the polymer.

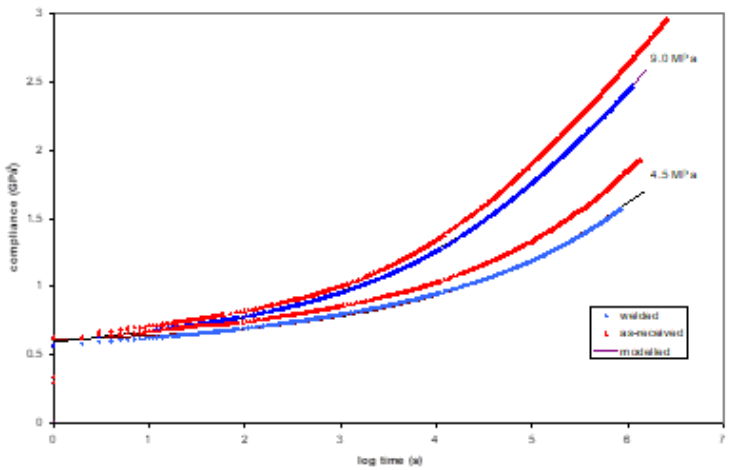


Figure 20. Tensile creep data for as-received and welded polypropylene (stress levels of 4.5 MPa and 9 MPa)

Physical ageing

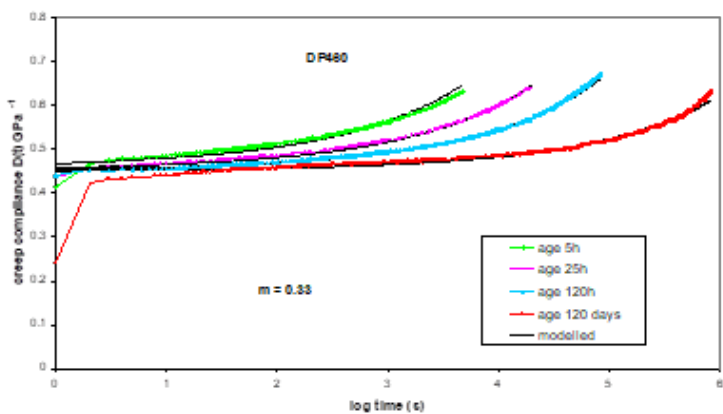


Figure 21. Creep curves for DP460 epoxy at different states of physical ageing

Physical ageing is defined as the process of progressive densification of a glassy material, which occurs with time due to conformational changes of the polymer. The driving force for these changes arises from the non-equilibrium change conformations that occur when the material is cooled from a high temperature to a low temperature (i.e., below T_g). Physical ageing is a reversible process (i.e., material aged for a number of years can be rejuvenated by short period of annealing at an appropriate elevated temperature) - see NPL Measurement Good Practice Guide No. 2 [29]. Figure 21 compares creep compliance curves for 3M DP460 epoxy adhesive at different states of physical ageing. In Figure 20, the as-received material (i.e., parent material used for producing the welded specimens) was tested at earlier time to the welded specimens, and hence the lower value of t_0 to that can be observed.

As the material is cooled, conformational changes that are needed to maintain an equilibrium structure are restricted by the increase in the relaxation times (reduced mobility) of molecular rearrangements at the lower temperatures. These non-equilibrium structures have a relatively high mobility to relaxation processes under creep loading, and this gives rise to relatively rapid creep at short elapsed times after cooling. Despite the low temperature of glassy polymers, there is sufficient molecular mobility for structural changes to take place with subsequent elapsed time (physical ageing) leading to structural states that become progressively closer to equilibrium for the low temperature. These ageing processes give rise to a reduction in molecular mobility under creep and a shift in creep curves to longer creep times as shown in Figure 21. The only parameter to change in each curve is the mean retardation time t_0 . The longer the time that has elapsed between quenching the material from an elevated temperature to a lower temperature, the larger retardation time (i.e., creep rate under load decreases with physical ageing).

The effect of physically ageing can become apparent during creep tests performed within a short period of time following quenching (i.e., within 10 days). Testing that is performed in which the age of the material is effectively the same at the start and end of the test is referred to as short-term (i.e., low stresses or short time duration (1-2 minutes)). Further ageing of material under load will occur in long-term creep tests, evident by a reduction in the rate at which the material creeps. The effect of physical ageing on creep behaviour diminishes with time. Creep tests conducted on polymers with a physical age of a 3-6 months, or more should see minimal effect on the rate of creep, provided the tests are kept short (i.e., 10 days or less).

De-ageing and pre-conditioning: It may be necessary to de-age a material by heating in order to set the clock to zero, particularly when the intention is to characterise the materials creep behaviour and where the age of the material is unknown. Physical de-ageing is the procedure of annealing test specimens at temperatures slightly above T_g for amorphous materials or below the pre-conditioning temperature for semi-crystalline materials (e.g., polypropylene), which erases any structure established by the previous thermal history – see NPL Measurement Good Practice Guide No. 2 [29] for advice on procedures for de-ageing and pre-conditioning.

Stress magnitude and stress state effects

Linear creep behaviour of polymeric materials is restricted to low stress levels (i.e., typically 1-2 MPa). At stress levels above this, non-linear creep behaviour arises because the mean retardation time parameter t_0 is dependent on the magnitude of the creep stress. The dependence of t_0 on stress level gives rise to non-linear creep behaviour. Creep curves are then observed to shift to shorter times with increasing stress (see Figure 20). At stress levels approaching the creep rupture stress, the material begins to yield (i.e., plastic deformation) and the mechanism controlling creep behaviour is different to that below the yield stress. The dominant mechanism controlling creep behaviour also alters at moderate stresses and very long test durations (i.e. plastic deformation).

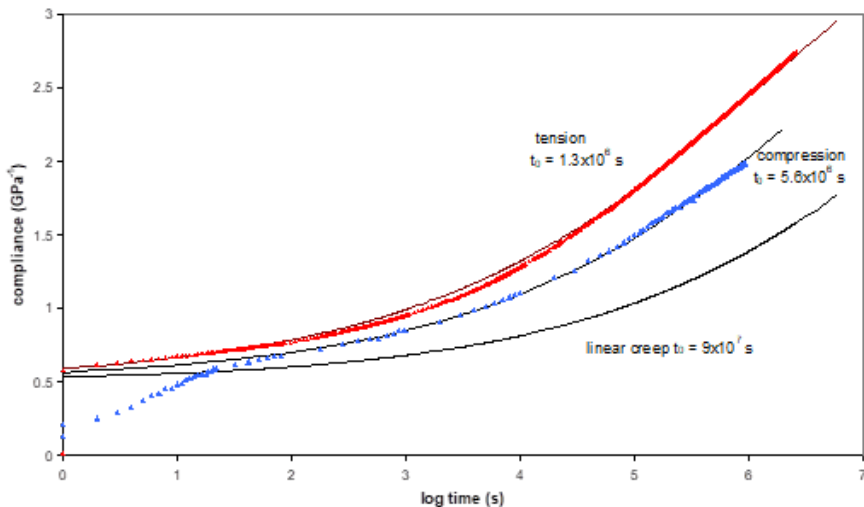


Figure 22. Tensile and compressive creep behaviour of polypropylene at 9 MPa
(calculated curve for linear behaviour under tension is also shown)

At low stresses, compliance curves in tension and compression can be expected to be the same. At higher stresses, where behaviour is non-linear, the results in Figure 22 show that the reduction in t_0 is less under compression than under tension. The rate of creep is higher in tension than in compression for the same applied stress. The magnitude of t_0 is determined, therefore, by the stress state as well as its magnitude. The retardation time parameter t_0 is determined by the magnitude of an effective stress that is a function of the shear component of applied stress as well as the hydrostatic component – see Section on modelling creep in polymers.

Temperature and moisture effects

Increasing the temperature increases molecular mobility, and hence increases the rate of creep under load. At elevated temperatures, it is important to look for signs of yielding in the creep test. It is advisable when planning creep tests at elevated temperature that viscous flow and the practical problems associated with measuring strain is taken into account. Yielding can be avoided by lowering either the test temperature or the applied stress. Non-linear behaviour may be observed at higher temperatures at stresses, which give rise to linear behaviour at lower temperatures. It should be noted that creep parameters, such as the retardation time are only valid at the temperature at which the data was obtained.

Polymers, such as nylon and epoxy adhesives are sensitive to moisture and will absorb significant amounts of moisture under relatively benign environments (i.e., standard laboratory conditions). The presence of water in epoxy adhesives results in plasticisation (essentially softening) of the polymeric material (i.e., increase in molecular mobility - a reduction in t_0) [79]. It is recommended that specimens be kept in a controlled environment, such as a dessicator) to ensure comparable moisture content. It may be necessary to dry the specimens to constant weight before testing (see [37]).

Cure and welding parameters

Degree of cure: The degree of cure of an adhesive will affect its creep performance. For poorly cured systems, where the cross-linking process is incomplete, T_g will be suppressed, resulting in an increase in molecular mobility and higher creep rates. It is important to ensure the adhesive or resin is completely cured. Thermal analysis should be carried out to ensure the state of cure meets specification. DSC and DMA can provide useful information relating to the final state of cure [58-60].

Welding processing parameters: The quality of a weld in thermoplastic polymers (e.g., polypropylene) has a significant affect on the weld strength and the long-term creep rupture behaviour of the welded system. In the case of hot plate welding, the hot plate temperature, fusion pressure, hot soak time and cooling time are key parameters in ensuring the quality of the weld meets specification. Similar parameters operate for other forms of welding. Hot plate temperature seems to have a dominant influence on creep rupture, although the other processing parameters are important and must be taken into account (see Figure 16).

Procedure for modelling creep in polymers

Creep tests are used to determine how material properties depend upon time under a constant applied load. This section gives an outline of the stages required to determine the parameters in creep functions commonly used to model creep in polymers.

Functions are first presented that relate strain to stress under a uniaxial tensile stress. These are then extended to cover non-linear behaviour and brief reference is given to how a model can be generalised to relate stress and strain components under a multiaxial stress state (see – [78-80]).

Creep tests are carried out under uniaxial tension and over a range of stress levels. The tensile strain $\varepsilon(t)$ is measured at suitable time intervals and tensile creep compliance values $D(t)$ at each time are derived by dividing the strain at that time by the constant applied stress σ_0 as follows:

$$D(t) = \frac{\varepsilon(t)}{\sigma_0} \quad (9)$$

The selected stress range depends on the load bearing capabilities of the polymer but, for guidance, the maximum stress could be $0.5 \times$ the peak stress measured in a short-time tensile test. For each stress, creep compliance curves are determined as a function of creep time.

Best fits are obtained to these data using the equation:

$$D(t) = D_0 + \Delta D \left[1 - \exp - \left(\frac{t}{t_0} \right)^n \right] \quad (10)$$

if the polymer is semi-crystalline, or

$$D(t) = D_0 \exp \left(\frac{t}{\tau} \right)^m \quad (11)$$

if the polymer is amorphous and the glass-to-rubber relaxation mechanism is responsible for the creep deformation.

Here D_0 is the tensile compliance at zero time, ΔD is the magnitude of the compliance change at long times when the relaxation process is complete, and t_0 , τ , n and m are parameters that define the time range over which the creep relaxation process takes place. A value for the parameter D_0 is readily estimated from results at short creep time. Values for the other parameters can be obtained by manual optimisation to achieve satisfactory fits.

At elevated stresses, compliance curves $D(t)$ are observed to shift to shorter creep times giving rise to non-linear behaviour. It is generally possible to obtain good fits to experimental data at different stress levels by keeping all parameters constant except t_0 or τ so that this parameter is the only parameter that is dependent on stress.

The dependence of t_0 on stress is then described using the expression:

$$t_0 = A \exp - (\alpha \sigma_0) \quad (12)$$

for crystalline polymers, and

$$D\tau = B \exp - (\beta \sigma_0^2) \quad (13)$$

For amorphous polymers.

Here, A , B , α and β are material parameters that determine the sensitivity of the time parameter to the stress magnitude.

Creep tests under uniaxial compression can also be modelled by the procedure used for tensile behaviour, but the time parameters t_0 and τ are not given by the above equations. This suggests that the stress state as well as the stress level determine the magnitude of the time parameter. The above equations should therefore be replaced by:

$$t_0 = A \exp - (\alpha \bar{\sigma}) \quad (14)$$

or

$$\tau = B \exp - (\beta \bar{\sigma}^2) \quad (15)$$

Here, $\bar{\sigma}$ is an effective stress that is defined by the shear and hydrostatic components of the creep stress as follows:

$$\bar{\sigma} = \mu \sigma_e + 3(1 - \mu) \sigma_m \quad (16)$$

σ_e and σ_m are the deviatoric and hydrostatic components of stress and μ is a parameter that determines the sensitivity of the creep time parameter to the hydrostatic component of stress. A value for the parameter μ can thus be derived from a value for the time parameter t_0 or τ determined from a compressive creep test.

The introduction of an effective stress that determines the magnitude of the time parameter allows the creep function presented above for describing creep under a uniaxial stress to be generalised to relate stress and strain components under a multiaxial stress. This is necessary for the model to be implemented in a finite element system, thus enabling stress analyses to be carried out for long-term loading applications.

Chapter 5

Handling, storage, and documentation

- Handling and storage of materials and chemicals
- Handling and storage of adhesives, resins and prepreg
- Handling and storage of adherends
- Handling, storage, and disposal of chemicals
- Documentation (record keeping)

Handling and storage of materials and chemicals

A number of issues relating to the handling and storage of materials and chemicals 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 polymeric materials, such as resins and 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, resins and prepreg

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 (e.g., adhesives and resins) to temperatures and humidity's above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [3]. The overall effect is to reduce the adhesive performance and induce premature failure of the polymeric system.

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

Ideally, temperature and humidity in the storage area should be tightly controlled and monitored (i.e., temperature recorders), although this may not always be physically or economically feasible. Standard laboratory conditions are typically $23 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ relative humidity (RH).

Refrigerators (or freezers) should be armed with a temperature alarm and the temperature should be monitored continuously to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. The maintenance of the refrigerator units should allow for regular manual or automatic defrosting.

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

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

Resins, adhesives and prepreps 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

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.

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

Handling, storage, and disposal of chemicals

In all cases involving solvents, primers, acid and alkaline solutions, extreme care and good laboratory practice should be followed at all times in storing, handling and disposing these materials (recommended further reading [20]). Personnel need to be trained in handling and using these materials, and should wear appropriate protective clothing as the chemicals can be harmful if they come contact with the skin or eyes. Ventilation and spill containment are particularly important safety considerations. Chemicals treating solutions should be prepared and stored in containers of glass, ceramic, or chemical resistant plastic. Checks should be made to ensure that the containing vessel is resistant to chemical attack from would be contents (e.g., hydrofluoric acid attacks glass). Stirring rods should be made of the same materials. Contact of the chemical solution with metals during preparation and storage should be avoided.

Continuous monitoring and quality control are required for all chemicals employed in chemical conditioning and surface treatments, and all containers with chemical agents should be clearly identified with labels. Labelling should be protected from being damaged through chemical contact or handling. Chemical solutions need to be changed regularly to prevent contamination and ensure repeatable concentration [20]. Quality assurance tests are recommended to check concentrations, and levels of contamination (e.g., opacity for checking particulate content, colour and pH for acidic and alkaline solutions).

Warning: It is important to note that many chemicals, such as acids, alkalis and solvents are toxic and present a risk to both human health and the environment, and that all safety requirements need to be met when handling, storing, and disposing of these materials.

Documentation (record keeping)

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

It is recommended that an accurate check as to the quality of the polymeric material 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 [20]). The colour, viscosity, shelf life, etc. should be verified. It may be necessary to carry out acceptance tests in-house to ensure mechanical properties are in accordance with the material specification. Acceptance tests should be directed towards assuring that the materials are identical from batch to batch. Accelerated environmental tests (i.e., high temperature and humidity) may also need to be carried out to confirm that the durability performance of the material meets the manufacturer specifications. ISO, BSI, and ASTM list various test methods for quality assurance of adhesives (see Chapter 9).

Standards

Standards

ISO Standards

Plastics

Mechanical

- ISO 178 Plastics - Determination of Flexural Properties.
- ISO 179 Plastics - Determination of Charpy Impact Properties - Part 1: Non-Instrumented Impact Test.
- ISO 179-2 Plastics - Determination of Charpy Impact Properties - Part 2: Instrumented Impact Test.
- ISO 527-1 Plastics - Determination of Tensile Properties - Part 1: General Principles.
- ISO 527-2 Plastics - Determination of Tensile Properties - Part 2: Test Conditions for Moulding and Extrusion Plastics.
- ISO 604 Plastics – Determination of Compressive Properties.
- ISO 10350-1 Plastics - Acquisition and Presentation of Comparable Single-Point Data - Part 1: Moulding Materials.
- ISO 11403-1 Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 1: Mechanical Properties.

Mechanical (Creep)

- ISO 899-1 Plastics - Determination of Creep Behaviour - Part 1: Tensile Creep.
- ISO 899-2 Plastics - Determination of Creep Behaviour - Part 2: Flexural Creep by Three-Point Loading.

Thermal properties

- ISO 75-1 Plastics - Determination of Temperature of Deflection Under Load - Part 1: General Test Method.
- ISO 75-2 Plastics — Determination of Temperature of Deflection Under Load — Part 2: Plastics and Ebonite.
- ISO 11403-2 Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 2: Thermal and Processing Properties.

Environmental conditioning

- ISO 62 Plastics – Determination of Water Absorption.

ISO 175	Plastics - Determining the Effect of Liquid Chemical Including Water.
ISO 291	Plastics – Standard Atmospheres for Conditioning and Testing.
ISO 305	Determination of Thermal Stability of Polyvinyl Chloride Related Chlorine Containing Polymers, and their Compounds - Discoloration Method.
ISO 483	Plastics - Small Enclosures for Conditioning and Testing Using Aqueous Solutions to Maintain Relative Humidity at Constant Value.
ISO 554	Standard Atmospheres for Conditioning and/or Testing - Specification.
ISO 1137	Plastics: Determination of Behaviour in a Ventilated Tubular Oven.
ISO 1599	Cellulose Acetate- Determination of Viscosity.
ISO 2578	Plastics: Determination of Time/Temperature Limits after Exposure to Prolonged Action of Heat.
ISO 3205	Preferred Test Temperatures.
ISO 3671	Determination of Volatile Matter of Amino-Plastics.
ISO 4611	Plastics - Determination of the Effect of Exposure to Damp Heat, Water Spray and Salt.
ISO 11403-3	Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 3: Environmental Influences on Properties.

Environmental stress cracking (ESC)

ISO 4599	Plastics: Determination of Time/Temperature Limits after Exposure to Prolonged Action of Heat Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Bent Strip Method.
ISO 4600	Plastics - Determination of Environmental Stress Cracking (ESC) - Ball and Pin.
ISO 6252	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Constant Tensile Stress Method.
ISO 16770	Plastics - Determination of Environmental Stress Cracking (ESC) of Polyethylene - Full-Notch Creep Test (FNCT).
ISO 22088-5	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Part 5: Constant Tensile Deformation Method.
ISO 22088-6	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Part 6: Slow Strain Rate Method.

Welded joints

- ISO 13953 Polyethylene (PE) Pipes and Fittings - Determination of the Tensile Strength and Failure Mode of Test Pieces from a Butt-Fused Joint.
- ISO 13954 Plastics Pipes and Fittings - Peel Decohesion Test for Polyethylene (PE) Electrofusion Assemblies of Nominal Outside Diameter Greater Than or Equal to

Composites

- ISO 75-3 Plastics - Determination of Temperature of Deflection under Load. Part 3: High-Strength Thermosetting Laminates and Long-Fibre-Reinforced plastics.
- BS EN 2378 Fibre Reinforced Plastics - Determination of Water Absorption by Immersion in Demineralised Water.
- BS EN 2489 Fibre Reinforced Plastics - Determination of the Action of Liquid Chemicals.
- BS EN 2823 Fibre Reinforced Plastics - Determination of the Effect of Exposure to Humid Atmosphere on Physical and Mechanical Characteristics.
- ISO 10350-2 Plastics — Acquisition and Presentation of Comparable Single-Point Data — Part 2: Long-Fibre-Reinforced Plastics.
- ISO 13003 Fibre-Reinforced Plastics - Determination of Fatigue Properties under Cyclic Loading Conditions.
- ISO 21746 Composites and Metal Assemblies - Galvanic Corrosion Tests of Carbon Fibre Reinforced Plastics (CFRPs) Related Bonded or Fastened Structures in Artificial Atmospheres - Salt Spray Tests.
- ISO 22836 Fibre-Reinforced Composites - Method for Accelerated Moisture Absorption and Supersaturated Conditioning by Moisture Using Sealed Pressure Vessel.

Note: The standards listed in this section refer specifically to fibre-reinforced composites. More general standards covering both plastics and composites are given in the section on ISO plastic related standards.

Adhesives

Mechanical

- ISO 4587 Adhesives - Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies.
- ISO 6922 Adhesives – Determination of Tensile Strength of Butt Joints.

- | | |
|-------------|--|
| ISO 9664 | Adhesives - Test Methods for Fatigue Properties of Structural Adhesives in Tensile Shear. |
| ISO 11003-1 | Adhesives - Determination of Shear Behaviour of Structural Adhesives — Part 1: Torsion Test Method Using Butt-Bonded Hollow Cylinders. |
| ISO 11003-2 | Adhesives - Determination of Shear Behaviour of Structural Adhesives — Part 2: Tensile Test Method Using Thick Adherends. |

Environmental testing

- | | |
|-----------|---|
| ISO 9142 | Adhesives - Guide to the Selection of Standard Laboratory Ageing Conditions for Testing Bonded Joints. |
| ISO 10354 | Adhesives - Characterisation of Durability of Structural Adhesive Assemblies - Wedge Rupture Test. |
| ISO 10363 | Hot Melt adhesives - Determination of Thermal Stability. |
| ISO 14615 | Adhesives - Durability of Structural Adhesive Joints - Exposure to Humidity and Temperature under Load. |

BSI and EN Standards

Welded joints

- | | |
|------------|---|
| EN 12814-1 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 1: Bend Test. |
| EN 12814-2 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 2: Tensile Test. |
| EN 12814-3 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 3: Tensile Creep Test. |
| EN 12814-4 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 4: Peel Test. |
| EN 12814-5 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 5: Macroscopic Examination. |
| EN 12814-6 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 6: Low Temperature Tensile Test. |
| EN 12814-7 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 7: Tensile Test with Waisted Test Specimens. |

- EN 12814-8 Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 8: Requirements.
- EN 13100-1 Non destructive Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 1: Visual Examination.

Adhesives

- BS EN 1465 Adhesives – Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies.
- BS 5350-A1 Methods of Test for Adhesives - Part A1: Adherend Preparation.
- BS 7079 General Introduction to Standards for Preparation of Steel Substrates Before Application of Paints and Related Products.
- BS EN 2243-5 Non-Metallic Materials Structural Adhesives Test Method Part 5: Ageing Tests.
- BS EN 13887 Structural Adhesives. Guidelines for Surface Preparation of Metal and Plastics Prior to Adhesive Bonding.
- BS EN 26922 Adhesives - Determination of Tensile Strength of Butt Joints.

ASTM Standards

Plastics

Mechanical

- ASTM D695 Standard Test Method for Compressive Properties of Rigid Plastics.
- ASTM D2990 Standard Test Methods for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics.

Environmental conditioning and testing

- ASTM D543 Standard Practices for Evaluating the resistance of Plastics for Chemical Reagents.
- ASTM D570 Standard Test Method for Water Absorption of Plastics.
- ASTM D618 Standard Practice for Conditioning Plastics for Testing.

Environmental stress cracking (ESC)

- ASTM D1693 Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics.
- ASTM F1248 Standard Test Method for Determination of Environmental Stress Crack Resistance (ESCR) of Polyethylene Pipe.

Adhesives

Mechanical

- ASTM D897 Standard Test Method for Tensile Properties of Adhesive Bonds.
- ASTM D905 Strength Properties of Adhesive Bonds in Shear by Compression Loading.
- ASTM D1002 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal).
- ASTM D1780 Standard Practice for Conducting Creep Tests of Metal-to-Metal Adhesives.
- ASTM D2095 Standard Test Method For Tensile Strength Of Adhesives By Means Of Bar And Rod Specimens.
- ASTM D2293 Standard Test Method for Creep Properties of Adhesives in Shear By Compression Loading (Metal-to-Metal).
- ASTMD 2294 Standard Test Method for Creep Properties of Adhesives in Shear By Tension Loading (Metal-to-Metal).
- ASTM D3163 Standard Test Method for Determining Strength of Adhesively Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading.
- ASTM D3166 Standard Test Method for Fatigue Properties of Adhesives in Shear by Tension Loading (Metal/Metal).
- ASTM D3528 Standard Test Method for Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading.
- ASTM D3983 Standard Test Method for Measuring Strength and Shear Modulus of Nonrigid Adhesives by the Thick-Adherend Tensile-Lap Specimen.
- ASTM D4896 Standard Guide for Use of Adhesive-Bonded Single Lap-Joint Specimen Test Results.
- ASTM D5656 Standard Test Method for Thick-Adherend Metal Lap-Shear Joints for Determination of the Stress-Strain Behavior Of Adhesives in Shear by Tension Loading.

Environmental conditioning and testing

- ASTM D896 Standard Practice for Resistance of Adhesive Bonds to Chemical Reagents.
- ASTM D1151 Standard Practice for Effect of Moisture and Temperature on Adhesive Bonds.
- ASTM D1183 Standard Practices for Resistance of Adhesives to Cyclic Laboratory Aging Conditions.

- ASTM D1828 Standard Practice for Atmospheric Exposure of Adhesive Joints Stressed in Peel.
- ASTM D2295 Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading at Elevated Temperatures (Metal-to-Metal).
- ASTM D2918 Standard Test Method for Durability Assessment of Adhesive Joints Stressed in Peel.
- ASTM D2919 Standard Test Method for Determining Durability of Adhesive Joints Stressed in Shear by Tension Loading.
- ASTM D3434 Standard Test Method for Multiple-Cycle Accelerated Aging Test (Automatic Boil Test) for Exterior Wet Use Wood Adhesives.
- ASTM D3632 Standard Test Method for Accelerated Aging of Adhesive Joints by the Oxygen-Pressure Method.
- ASTM D3762 Standard Test Method for Adhesive-Bonded Surface Durability of Aluminium (Wedge Test).
- ASTM D4498 Standard Test Method for Heat-Fail Temperature in Shear of Hot Melt Adhesives.

Surface treatment

- ASTM D2093 Standard Practice for Preparation of Surfaces of Plastics Prior to Adhesive Bonding.
- ASTM D2651 Standard Guide for Preparation of Metal Surfaces for Adhesive Bonding.

Composites

- ASTM C394 Standard Test Method for Shear Fatigue of Sandwich Core Materials.
- ASTM C480 Standard Test Method for Flexure Creep of Sandwich Constructions.
- ASTM D896 Standard Practice for Resistance of Adhesive Bonds to Chemical Reagents.
- ASTM D3479 Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite Materials.
- ASTM D5229 Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials.
- ASTM D5379 Standard Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method.
- ASTM D6115 Standard Test Method for Mode I Fatigue Delamination Growth Onset of Unidirectional Fiber-Reinforced Polymer Matrix Composites.

ASTM D7792 Standard Practice for Freeze/Thaw Conditioning of Pultruded Fiber Reinforced Polymer (FRP) Composites Used in Structural Designs.

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Useful contacts

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Abstract

Welded thermoplastic and adhesively bonded joints are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the joined system. However, service conditions can often involve exposure to static and/or cyclic fatigue loading conditions. Fatigue damage can be particularly harmful to the structural integrity of bonded joints, shortening the life expectancy of the joined system by considerable margins, and is known to occur at relatively low stress levels, particularly in the presence of hostile environments. Similarly, premature failure can occur in bonded joints and welded thermoplastics under static loading conditions with deformation occurring at relatively low loads. This Good Practice Guide is intended to give guidance on fatigue testing of adhesively bonded joints and creep testing of bonded joints and welded thermoplastics for generating design data and for quality assurance purposes. The document is primarily concerned with structural adhesives and thermoplastics.

NPL has made every effort to ensure all information contained in this Good Practice Guide was correct at the time of publication. NPL is not responsible for any errors, omissions, or obsolescence, and does not accept any liability arising from the use of this Good Practice Guide.

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Glossary of Terms (Based on BSI and ASTM Definitions)

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

Adherend: Body that is or 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.

Ageing: Entirety of all irreversible chemical and physical processes occurring in a material in the course of time.

Amorphous: Non-crystalline or devoid of crystalline structure.

Amorphous regions: Regions within a polymeric material, that, based on X-ray diffraction or other suitable techniques, do not show any evidence of crystalline structure.

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.

Conditioning: A series of operations intended to bring a sample or specimen into a reference state with regard to temperature and humidity.

Coupling agent: Substance (e.g., organosilane) that promotes or establishes stronger bond at the interface of the resin matrix and the reinforcement.

Coupling size: Size designed to obtain a good bond between the fibre surface and surrounding resin matrix.

Crack: Fissure that may or may not penetrate the external surface of the material or its entire thickness, the polymeric material being completely separated between the crack walls.

Craze: Defect at or under the surface of a plastic attributable to apparent cracks, bridged by polymeric material of reduced (apparent) density.

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

Crystallinity: Presence of three-dimensional order at the level of molecular dimensions.

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

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

Cure temperature: Temperature at which an adhesive, an assembly or a polymeric composition achieves a state of cure.

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.

Diffusivity: The proportionality constant that describes the ability of chemical species, such as a liquid, to diffuse through a solid. Diffusivity is generally temperature dependent.

Dimensional stability: Consistency of dimensions of a material part or specimen under environmental conditions.

Discolouration: Colour change that involves lightening or darkening and/or change in hue.

Dry strength: Strength of a polymeric material part or specimen after drying under specified conditions.

Ductile failure: Failure in which the fracture surface clearly exhibits permanent deformation with stretching, elongation, and necking.

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

Elastic limit: Greatest stress, which a material is capable of sustaining without any permanent strain remaining upon complete release of stress.

Elongation: Increase in length of a specimen under tension, usually expressed as a percentage of the original length.

Embedding: Process of encasing completely an article, such as a sensor, in a polymeric material.

EN: European Norm

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

Fibre: Unit of matter of relatively short length, characterised by a high ratio of length to thickness or diameter.

Filler: Relatively inert material (e.g., talc) added to a plastic or resin to modify its strength, fire resistance or other qualities, or to lower costs.

Fillet: Portion of an adhesive that bridges the adherends outside the bond-line.

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.

Hardness: Resistance of a material to indentation or scratching.

Hygroscopic: Material capable of absorbing and retaining environmental moisture.

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.

Lay-up: Assembly of layers of resin-impregnated material for processing.

Ligament area: Cross-sectional area remaining after notching.

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.

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

Plastic deformation: That part of the strain in a stressed plastic, which remains after the applied stress has been removed.

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

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

Porosity: A condition of trapped pockets of air, gas, or vacuum within a solid material. Prepreg: Admixture of resins (with or without fillers), additives, and reinforcements in woven and filamentous form, ready for moulding.

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. Roving: Collection of parallel strands (assembled roving) or parallel filaments (direct roving) assembled without intentional twist.

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

Semi-crystalline: Polymer containing crystalline and amorphous phases.

Service life (N): Number of 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.

Size (coupling size): Material applied to fibres or filaments during the course of their manufacture.

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 the 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.

Swelling: Increase in volume of a test specimen immersed in a liquid or exposed to a vapour.

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

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

Toughness: That property of a material by virtue of which it can absorb energy, generally implying absence of brittleness and relatively high elongation to break.

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

True stress: Stress calculated from the supporting area at the time of measurement rather than from the original area.

Viscoelasticity: Stress response of a material acting as though it were a combination of an elastic-solid and a viscous fluid with flow dependent on time, temperature, load, and rate of loading.

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

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

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

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

Chapter 1

Introduction and scope

- Introduction
- Scope

Introduction

Welded thermoplastic and adhesively bonded joints are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the joined system. However, service conditions can often involve exposure to static and/or cyclic fatigue loading conditions. Fatigue damage can be particularly harmful to the structural integrity of joints, shortening the life expectancy of the joined system by considerable margins, and is known to occur at relatively low stress levels, particularly in the presence of hostile environments. Similarly, premature failure can occur in joined systems under static loading conditions. Deformation occurs at relatively low loads.

Scope

This Good Practice Guide is intended to give guidance to technologists, engineers and designers on fatigue and creep testing of adhesively bonded joints, and creep testing of welded thermoplastics for generating design data and for quality assurance purposes. The document is primarily concerned with structural adhesives and thermoplastics (polypropylene). Consideration is given to the effect of processing parameters, geometric factors, and hostile environments on the long-term performance of these joined systems.

The intention of the guide is to provide designers and users with sufficient information, which when coupled with their own expertise and a suitable accelerated test regime can be used to produce design data and enable screening of materials, surface treatments and processing parameters. The document provides guidance of the use of test methods (i.e., performance testing) that can be used to measure physical changes resulting from fatigue and creep. If the intention is to generate design data, then the guide should be used in conjunction with the appropriate structural design codes and standards.

The guide assumes some basic knowledge of the materials and mechanical engineering and is not intended as a textbook or as a design protocol. There are several published works, which provide a comprehensive coverage of plastics, adhesives, and composites technology, and testing and preliminary design [1-28]. Other NPL Measurement Good Practice Guides (see [29-38]), provide advice on issues relating to the preparation and testing of plastics, adhesives, and adhesive joints. The intention of the guide is to complement these published works.

It is recommended that specialist advice be sought from manufacturers and suppliers on material selection, and use of associated technologies and health and safety requirements. Expert advice should be obtained from the adhesive manufacturer on selection and use of surface treatments for adhesive bonding and welding thermoplastics, and that the detail requirements specified by the manufacturer are completely satisfied.

Where tests are performed to assess adhesive materials or processing parameters for welding thermoplastics then it is recommended that the surface preparation is as good as possible, to minimise premature failure. The surface preparation procedures for test specimens will need to mirror those for the final joined component. Organisations that can provide specialist advice are listed at the back of the guide along with relevant standards and publications.

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Chapter 2

Mechanical testing

- Introduction
- Preparation and assembly of joined systems
- Mechanical testing of joined systems
- Specimen geometry and adherend effects

Introduction

Several factors, not included in written standards, can significantly affect the reliability (i.e., repeatability and reproducibility) of data obtained from testing adhesively bonded and welded joints. Factors, including specimen and machine alignment, processing variables, specimen preparation, storage of materials and surface treatments need to be considered in addition to test parameters (e.g., displacement rate), and specimen dimensions that are generally covered in national and international standards. Careful consideration needs to be given to those factors that significantly influence strength and long-term performance of joined systems. This Chapter examines the key issues relating to the preparation, assembly and testing of adhesive joints and welded thermoplastics (including constituents).

Preparation and assembly of joined systems

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. Surface preparation also has a major influence on the quality of welded thermoplastic joints. It is worth noting that a high percentage of failures can be attributed to poor joint manufacture or a lack of understanding of those factors that influence joint performance. These problems can be minimised or eliminated through proper training and education. This section is concerned with issues relating to the preparation and assembly of adhesive joints and welded thermoplastics prior to curing or welding.

Specimen manufacture: Adherends should be manufactured and/or machined accurately to ensure specimen dimensions meet the specifications of the standard. It is important to ensure that the adherends are free of any edge or surface damage. The surfaces to be joined 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 joint and/or the adherend. It is advisable before preparing the surface to ensure that the adherend sections to be joined 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.

Moisture effects: The presence of moisture at the surface or within the system can adversely affect the properties of adhesives and thermoplastic welds during thermal processing (i.e., cure or fusion), and as a result the joint strength may be compromised. Moisture released from the substrates of bonded joints during cure will enter the adhesive. For example, moisture released from polymer composite adherends 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 [39]. Fibre-reinforced thermoset composites are known to absorb moisture in relatively benign environments. For example, epoxy based composite systems can absorb 0.2 wt.% moisture in a laboratory environment (i.e., 23 °C and 50% relative humidity (RH)) within 2 to 4 weeks.

The strength and fatigue performance of the bonded structure under these circumstances will be compromised. 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 adherends should 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 adherends.

Surface preparation: This process is recognised as the most critical step in the joining process and considerable joint testing is performed to optimise surface treatment. Surface preparation of coupon specimens should match that to be used in the bonded or welded component. 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 and welded joints. In the case of adhesive joints, unsatisfactory surface preparation will result in the joint failing unpredictably at the adhesive/adherent interface.

Many adhesive joint tests have been developed to assess bonded systems (including surface preparation) – information on surface preparation and relative effectiveness is given in NPL Measurement Good Practice Guide No. 47 [34]. General procedures used for preparing different material surfaces for adhesive bonding are described in “Guide to The Structural Use of Adhesives” produced by The Institution of Structural Engineers [18]. BS 5350-A1 [40], BS 7079 [41], ASTM D2093 [42], ASTM D2651 [43], and BS EN 13887 [44] – see also [45-48].

Note 1: Surface preparation procedures for adhesive joints often require potentially hazardous or environmentally damaging chemicals. All preparation should be carried out to COSHH specifications (see Chapter 5).

Specimen geometry, dimensions, and preparation for welded joints of thermoplastics are given in BS EN 12814-3 [49]. ISO 899-1 [50] recommends that specimen preparation for tensile creep testing on unwelded polymers should be carried out in accordance with ISO 527-1 [51]. Surface contaminants may contribute to failure of the weld. A region of 0.4 mm should be removed from the edges of the sections to be welded before fusing the surfaces.

Cleaning the surfaces with detergent, hot water, or other chemical agents (e.g., solvents) is not recommended. These chemical agents may result in the removal of plasticizers or stabilizers from the surface, which may lead to the breakdown of the surface and compromise the welded joint.

Bonding and welding fixtures

Bonded joints: A bonding fixture is recommended to ensure accurate alignment, correct bond length (in the case of lap joints) and uniform bond-line thickness. Alternatively, large test panels (typically 180 mm wide) capable of providing six specimens can be made and then machined into specimens.

Checks should always be made to ensure that there is no mechanical damage due to machining or handling (i.e., adherend bending) or that excessive adhesive is forced from the joint due to clamping forces applied to the test specimens. It may be necessary to check the clamping force applied by the fixture to the specimen during the curing process to ensure that clamping force remains constant and has not relaxed through adhesive flow. Mould release agent or thin polytetrafluorene (PTFE) film will need to be used to guarantee easy release of bonded components from the clamping fixture.

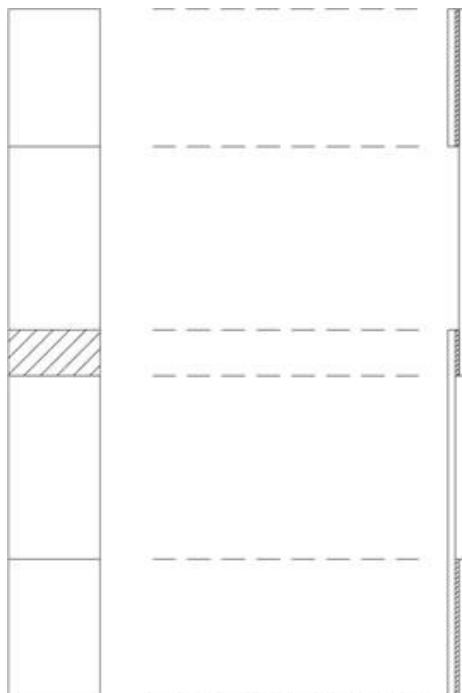


Figure 1. Schematic of a single-lap joint

Note 2: End tabs are introduced to single-lap joints in order to reduce (not eliminate) the eccentricity of the load path that causes out-of-plane bending moments, resulting in high peel stresses and non-uniform shear stresses in the adhesive layer (see Figure 1). BS EN 1465 [52] does not specify the use of end tabs but specifies that the long axis of the specimen coincides with the direction of the applied force through the centre line of the grip assembly. The introduction of end tabs, however, will add to the costs of this quality assurance test.

Welded joints: A similar approach to that employed for bonded joints needs to be adopted when welding thermoplastic sections. A fixture is required that ensures good alignment, and accurate and repeatable bond-lines. The specimen needs to be held securely (i.e., clamped) so there is no lateral or out-of-plane (i.e., buckling) movement. In the case of hot plate welding, the holding fixture needs to be square with respect to the heating plates. It is important to ensure that surface is only melted where required. Figure 2 shows hot plate welding equipment with holding clamps. The hot plate (core) is centrally located (further details on hot plate welding are given in Chapter 10 of [53]). The fixture should be sufficiently robust and capable of withstanding the temperatures required to weld the thermoplastic sections. The recommendation is to weld rectangular sections and then to machine test specimens from the welded panel.



*Figure 2. Hot plate welding rig
(Courtesy of TWI Limited)*

In conventional hot fusion (i.e., hot plate) welding, the cooling time (typically 6-15 minutes) accounts for a considerable portion of the total cycle time and is fixed for the size of the sections or components being welded. It is important, however, not to move the welded section from the holding frame until the welded material is solid and that the weld has sufficient strength to allow removing from the holding frame. Platen surfaces accumulate melted plastic and must be cleaned for high temperature welding materials. It is recommended that the weld bead (see schematic in Figure 3) should be removed before carrying out tests to assess joint performance. A large variation in total bead width, and to a lesser degree height, can be expected for the same welding conditions. The weld bead should be removed before machining individual specimens from the welded panel. Care should be taken to ensure the underlying material within the weld is not damaged due to machining of the specimen. Heating during machining should be minimised. Machined specimens should be visually inspected in accordance with the standard EN 13100-1 [54].

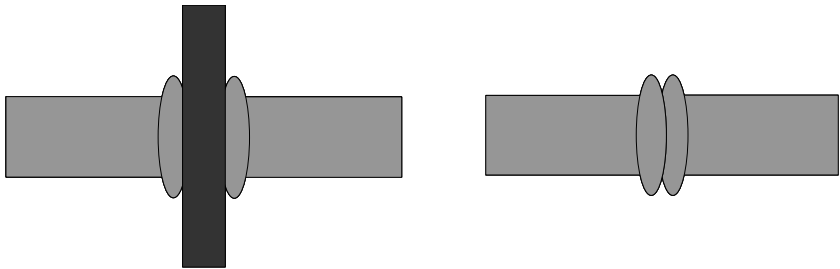


Figure 3. Schematic of hot plate welding

The quality of a weld in thermoplastic polymers (e.g., polypropylene) has a significant affect on the weld strength, and the long-term fatigue and creep rupture behaviour of the welded system. In the case of hot plate welding, the hot plate temperature, fusion pressure, hot soak time and cooling time are key parameters in ensuring the quality of the weld meets specification. Similar parameters operate for other forms of welding. Hot plate temperature seems to have a dominant influence on creep rupture, although the other processing parameters are important and must be considered.

Adhesive fillet

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

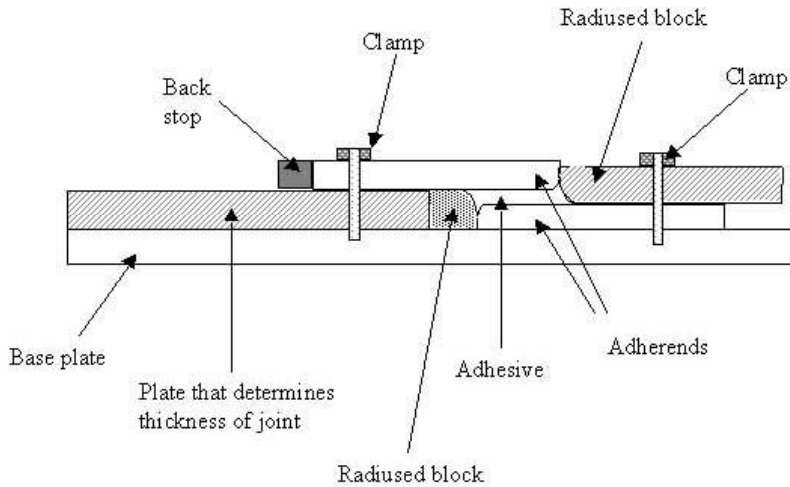


Figure 4. Fixture for controlling single-lap joint fillet

Several points are worth noting:

- Fillet size and shape should be controlled throughout the bonding process. This can be achieved using either a specially designed bonding fixture as shown in Figure 4 or a special tool shaped to fit within the bonded joint (e.g., cylindrical roller). The tool can be held in place using heat resistant tape. Figure 5 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 due to high viscosity.
- 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.

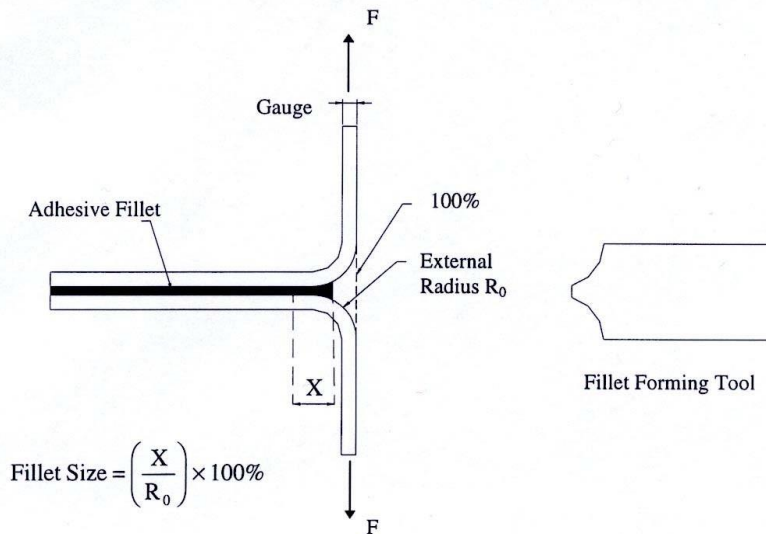


Figure 5. Definition of fillet size in T-peel joint (including fillet forming tool) [55]

Adhesive joint bond-line thickness

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

Control of bond-line thickness can be achieved by mechanical means (i.e., separation of adherends physically controlled by the bonding fixture), using thin wire spacers (e.g., stainless steel) inserted between the adherends or by ballontini glass balls, which can be mixed with single- and two-part adhesive pastes (typically 1 weight %). Film adhesives are available with carriers (e.g., nylon mat or mesh), which control bond-line thickness. It is essential that wire spacers used to control bond-line thickness are located well within the bonded area away from the specimen edges and regions of high stress concentrations (i.e., ends of joints). It may be difficult to employ wire spacers where the bond length is relatively short, such as single-lap joints (typically 12.5 mm overlap length).

When using glass beads to control bond-line thickness, the distribution of glass beads in the adhesive must be uniform, and therefore glass beads should be thoroughly mixed into the adhesive. Mixing should take place before applying the adhesive to the adherend surfaces.

Curing adhesive

Several key points should be considered when curing adhesive joint specimens:

- Porosity, in the form of entrapped air and volatiles, is a common cause of premature failure. In many cases it is virtually impossible to produce void free specimens, particularly for materials with a high viscosity. Specimens should be prepared using methods that minimise the inclusion of air in the test specimens. Visual inspection should be carried out to ensure there is no air entrapment.
- The cure state of the adhesive layer in the adhesive joint specimen should be similar to the adhesive in the bonded structure or to bulk adhesive specimens [56-57]. Failure to achieve similar thermal histories can result in significant differences in material properties. Differences between thermal histories will lead to differences in mechanical properties (See [33]).
- 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 in adhesive joints at the end of the cure period can differ significantly.
- For heat curing systems, the temperature of the specimen will lag behind the oven temperature, and it may therefore be necessary to elevate the oven temperature when curing joint specimens.
- Adhesives should be fully cured prior to conditioning and testing otherwise an adhesive will continue to cure, thus invalidating the test data.
- Adhesives have a low thermal conductivity. This may prevent dissipation of heat generated by exothermic cure reactions, thus causing heat damage.
- Residual thermal stresses may be generated as a result of non-uniform (rapid) cooling, resin shrinkage and thermal expansion coefficient mismatch between the adhesive and adherend. As the joint is cooled down from the cure temperature, residual stresses are frozen in the material.
- As mentioned previously, handling adhesives can be hazardous to human health, thus COSHH procedures should be followed to minimise operator exposure. Ovens and work areas should be suitably ventilated, ensuring minimal levels of hazardous vapours/gases in the work area.

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

Real-time monitoring of material property development in adhesives can be achieved using oscillatory rheometry or ultrasonic methods. Thermal analytical techniques, such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), can provide useful information relating to adhesive composition and final state of cure [58-60].

Note 3: The analysis of material by any technique is complicated by changes in the material during testing. For adhesive materials, there will most likely be further curing or loss of moisture. In some cases, these changes will prevent the accurate determination of T_g . Repeat measurements need to be taken to check completeness of cure. If the difference in T_g is greater than 5 °C between the first and second runs, then a third run is recommended. It is important that the material used for DSC and DMA samples is representative of the adhesive within the bonded structure. The suggested method for preparing samples is to place the adhesive between two flat, smooth, parallel metal adherends (i.e., stainless steel), which have been coated with a thin layer of lubricant or release agent to prevent adhesion. Adhesive samples can be easily removed without sticking from the bonded joint after curing.

Quality assurance

After the adhesive is cured or weld has cooled, joint specimens should be inspected to detect gross flaws or defects, particularly at the edges and ends of the joined region. 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.

Adhesive joints: 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.

Welded Joints: The size (i.e., width and height) of the weld bead and weld type should be recorded. If the bead has been removed, then a check should be carried out to ensure no damage has occurred to the material within the welded region and adjacent material. Special care needs to be taken when notching material (e.g., full-notch creep test (FNCT) – see ISO 16770 [61]) to ensure the notches are coplanar and the plane of notching is perpendicular to the tensile axis of the test specimen.

The notch radius needs to be less than 10 μm . It is recommended that razor blades be used for this purpose, although a cutting machine with a tool like a broaching device can be used provided the notch tip radius is less than 10 μm . Due care needs to be taken to avoid blunting the notch during this operation. The recommended tolerance on the notch depth is ± 0.1 mm.

Several techniques are available for the non-destructive inspection of adhesive and welded joints including ultrasonic C-scan, X-ray radiography and thermography.

Note 4: There is no NDE technique that can provide a quantitative assessment of joint strength.

Ultrasonic C-scan technique is particularly suited to the detection of planar type defects (e.g., debonds and delaminations) normal to the incident beam. Voids and porosity in the adhesive and adherends are also detectable. The minimum size of voids and delaminations detectable with C-scan is approximately 2-3 mm. The technique is not suitable for detecting surface contaminants (e.g., oils and grease). Scanning acoustic microscopy (SAM) can detect 0.2-0.3 mm size sub-surface defects in polymeric materials.

X-radiography can be used to inspect for debonds in polymer composite joints and between the outer skins and inner core of bonded composite sandwich structures. The technique is also capable of detecting micro-cracks in polymeric materials. X-radiography is not suitable for inspecting joints with metal adherends. Thin debonds and delaminations are difficult to detect because the presence of these defects has minimal effect on the absorption characteristics of polymeric materials.

The use of penetrant fluids can enhance the imaging process; however, these fluids can adversely affect the short-term properties and fatigue performance of polymeric materials. The fatigue life can be reduced by a factor of 2 or more. Penetrants should not be used in those tests where the test data is to be used for design or quality assurance purposes. Small tensile loads or the use of a vacuum pump can be used to promote fluid penetration.

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

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

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

Specimens should be stored in a desiccator (unless otherwise specified) to prevent moisture ingress, which can degrade the adhesive joint. Adhesive joints have been known to deteriorate under standard laboratory conditions (10% strength loss within 3-6 months). It is recommended that an accurate record be maintained for all batches of adhesive joints. Documentation should include details on date of manufacture, precursor materials (i.e., adhesive and adherends), description of method of joint fabrication and assembly (including processing variables - curing time, temperature, and pressure), surface preparation, and specimen dimensions (including width, bond length, adhesive layer and adherend thickness).

Mechanical testing of joined systems

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 polymers, and adhesive and welded joints. Guidance is provided on the main factors that need to be controlled when carrying out mechanical testing. Appendix 1 in NPL Measurement Good Practice Guide No. 47 [34] provides a summary of commonly used adhesive joint test methods and related standards.

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.

Note 5: Poor alignment of test specimens can result in low strength values and a significant reduction in fatigue life.

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. Alignment specimens can be in the form of a rectangular or circular bar. These specimens need to be accurately machined to ensure errors in parallelism are $< 0.2 \text{ mm/m}$ and in concentricity (lateral offset) of 0.03 mm [62]. 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. A positioning device should be used 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.

Gripping 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 (torsion) 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

Several contact and non-contact techniques are available for measuring strain and displacement many of which are not suited to fatigue tested (e.g., electronic speckle pattern interferometry). These techniques are described in NPL Measurement Good Practice Guide No. 47 [34] and No. 115 [63].

Contact extensometers are the preferred method for measuring strain and displacement, and hence stiffness of adhesive and welded joints. It is recommended that two extensometers, attached to opposite faces of the specimen, be used to measure displacement. Any bending of the specimen will be apparent from diverging displacement readings. It is also 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. This also applies to welded sections where the measured extension is the average effect of the deformation within the welded region and surrounding material within the gauge section.

Knife-edged tensile extensometers, as described in reference [30], can be used provided the extensometer straddles the bond-line. The deformation of the adherends needs to be accounted for when analysing the data, but where the stiffness of the adherends is very much greater than that of an 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 $\pm 0.5 \mu\text{m}$ 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. Contact extensometers and data acquisition system must also have sufficient response time to cope with the test frequency in cyclic fatigue testing.

Strain gauges: Currently there are no standard tests using strain gauges to monitor strain in an adhesive layer or welded region. However, structural monitoring capabilities, where strain-sensing optical fibre devices are embedded in materials, are the subject of research in many organisations. Care should be taken to ensure that measurements of strain derived from strain gauges mounted on the test specimen are reliable; as strain gauges tend to locally stiffen polymers, such that the strain is less than expected for a given load.

Strain gauges can be attached to adherends and will measure the strain in these adherends. The usefulness of such measurements may be limited except in the cases where changes in joint performance are manifested in measurable changes in the adherend strain. One such application is back-face strain gauging of thin lap-shear joints where crack growth in the adhesive layer can be monitored through strains measured by gauges bonded to the external surface of the adherends at the overlap. Strain gauges are occasionally used for monitoring strain in bonded structures (e.g., four-point bending of sandwich structures).

Strain gauges are generally limited to the measurement of strains less than 10%. Biaxial rosettes are available for measuring longitudinal and lateral strains. Large strain gauges are preferable as alignment and handling is easier, and they average out local strain variations. Local strain variations can cause premature failure of the strain gauges.

Correct alignment of strain gauges is important, as significant errors can be caused by careless application of the strain gauges to the specimen. Errors of 15% can occur from a 2° misalignment.

The adhesive used to bond the strain gauge should be capable of withstanding the test environment for the complete duration of the test. Most adhesives are sensitive to moisture (and other chemicals), which can often preclude bonding prior to specimen conditioning. Moisture attack of an adhesive and strain gauges will occur from the top, edges and in the case of polymeric materials through the substrate beneath the gauge. The situation is exacerbated at elevated temperatures. It is therefore important to ensure that the adhesive selected for bonding the strain gauge and associated electrical wiring is suitably encapsulated. Strain gauge manufacturers can provide information on adhesive selection, surface preparation and procedures for protecting strain gauges, and providing advice on fatigue performance and strain limits of these devices.

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

Crosshead displacement: An approximate measurement of strain, and hence stiffness can be obtained from measuring the crosshead displacement of the test frame [30]. The strain is the ratio of crosshead displacement and the initial grip separation. Hence, any slippage within the loading train will produce errors in the strain measurement. The strain values obtained from crosshead measurements will differ from the actual strain in the central region of the specimen. Stiffness measurements directly obtained from the crosshead movement need to be corrected to take into account the stiffness of the loading chain. This can be a difficult task as the specimen size and geometry, and the deformation behaviour of the specimen need to be taken into account. Given the small adhesive layer deflections that occur even at large strains owing to thin bond-lines, the accuracy of strains determined using crosshead displacements must be considered suspect and used only for qualitative purposes.

Linear voltage displacement transducers (LVDTs): LVDTs are recommended in preference to monitoring crosshead movement. These devices provide a direct reading of the moving part and can be attached at any point on the structure as required. LVDTs tend to be used to monitor global rather than localised deformation. Accurate alignment is essential otherwise measurement errors will occur and the movement of the device can be restricted.

It is important to ensure the device can operate effectively in the test environment and that electrical wiring is suitably protected. There is potential problem of friction, which arises from the movement of the core within the barrel, which is normally designed to have limited

rotational freedom [29]. Friction between the core and barrel can be significant resulting in “stick-slip” movement of the device.

Optical fibre-based sensors: Optical fibre-based sensors (e.g., Fibre Bragg grating (FBG)) devices can be used to monitor a wide range of parameters including strain, temperature, pressure, humidity, moisture ingress, in-situ cure kinetics, vibration, pH levels, chemical concentration, and gamma radiation. In addition, these devices can be used to monitor degradation processes and fracture. Most of the evaluation of these devices tends to be laboratory based under ideal conditions. As with other sensors, fibre optic devices can be in obscure locations not readily accessible by conventional NDE methods. These devices are small with low weight and in many cases can be embedded into or bonded to the structure (to produce smart structures). Composite laminates are particularly suited to their use simply by the fibrous nature of the material, and the fact that these devices can be introduced during the manufacturing of composite components. Bonding these devices to surfaces pose similar problems to those for strain gauges. Optical fibre diameters are large compared with carbon and glass fibres and can cause local distortion within composite structures. Optical techniques require sensors to remain fully bonded with the composite throughout the life of the component to ensure effective strain transfer.

Non-contact or optical extensometers: Non-contact extensometers (e.g., video extensometers) avoid the problems of contact damage and use up to failure. There are no temperature or environmental restrictions as video extensometers can be located outside the test chamber (provided that the specimen can be imaged clearly). Standard systems measure the separation of marks in one direction, mimicking contact extensometers, but some systems provide capabilities for dot location measurements, which allows two-dimensional measurements and a limited strain mapping capability. Dot location provides versatility and, potentially, enables measurement for a wide variety of different specimen geometries using a single system.

The technique uses remote cameras and image analysis software to monitor the separation of high contrast marks or lines inscribed on the test specimen. The initial separation of the marks defines the gauge-length and the change in separation of the marks is recorded throughout the test. Gauge marks should not be made on the specimen in any way that may cause damage to the specimen. Accuracy tends to be low for small strain measurements. Developments in resolution, sensitivity and speed of digital imaging and data processing are leading to improved capabilities. One limitation of the technology is that, unless a dual camera system is used, measurement is normally only possible at one side of the specimen so that bending cannot be evaluated.

Digital image correlation (DIC): DIC is a non-contact full-field strain measurement technique. The basic concept of DIC is to compare two images of a component before and after deformation. It uses computer image analysis to track the movement of blocks of applied speckle patterns on the surface of the specimen (see [63]).

Displacements and strains are determined by correlating the position of pixel subsets or blocks in the original and deformed image, normally based upon contrast (i.e., grey intensity levels). In order to identify if there is any movement between the two blocks there must be sufficient detail for it to be considered unique. It may be the case that the specimen or component already has a suitable level of surface features which can be imaged directly, but if not, some form of spray paint or coating or scratches on the surface can be used.

For best results a unique surface finish and a good distribution of intensity values must be obtained. This can be achieved by spraying the surfaces to be inspected with black, grey, and white paint to produce a random specular pattern for the image correlation. The deformations of the specimens are then calculated by correlating the positions and displacements of pixel subsets or blocks in the original and deformed image to produce a deformation vector map. This is then processed further to produce a full-field strain map. Calculation of 3D deformations is possible if two or more cameras are used. Calibration of the image by using a calibration plate is straightforward.

Mechanical testing

The stiffness, strength, creep deformation and fatigue life measured form only part of the useful data that are required when assessing the performance of joined systems. The mode of failure in adhesive joints (i.e., cohesive failure in the adhesive or interfacial failure) and degree of cohesive and adhesive failure should be recorded for each specimen. Similarly, the fracture behaviour should be recorded for creep rupture tests conducted on welded joints noting the degree of brittle and ductile fracture and location of failure. Fracture can occur within either the welded region or in the parent material adjacent to the weld. Optical microscopy or scanning electron microscopy may be required to analyse the fracture morphology, particularly if failure is close to the adhesive/adherend interface or at the interface between the welded region and parent material. Adhesive standards often recommend that results from specimens where failure occurs in the adherend should be discarded and replaced. However, unless the reason for adherend failure can be traced to inherent flaws in the test adherend that are unlikely to be replicated in the bonded component then these tests can still provide some valuable information.

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

Number of test specimens: A minimum number of five specimens should be tested at each stress level. If a greater precision of the mean value is required, then the number of specimens tested should be increased (see ISO 2602 [64]).

Specimen dimensions: Specimen dimensions and crack length need to be accurately measured, as small measurement errors can translate into large variations in strength or fracture toughness, particularly if the calculation includes squares or cubes terms of the measured parameter (see Table 1). The uncertainty in strength or fracture toughness calculation is compounded where there is more than term (i.e., width, thickness, and crack length, etc), each with an associated uncertainty. The bond-line thickness, being a very small dimension, tends to be the dimension where accuracy and precision of dimensional measurement are most critical. Measurements at different locations should be carried out to check the uniformity of bond-line thickness.

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

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

Table 1. Associated uncertainty with measurement error (percentage)

Test speed and frequency: Adhesives and thermoplastics are viscoelastic materials; that is their mechanical properties (strength and stiffness) are sensitive to the rate at which they are loaded (or more accurately the strain rate). Standards relating to testing of adhesive and welded joints infrequently specify the speed/rate or frequency of testing required. 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 quasi-static test trials to failure are recommended to ascertain the equivalent frequency required to match 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.

Specimen geometry and adherend effects

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

Adherend Thickness/Overlap Length	Load/Width (N/mm)
CR1 Mild rolled steel	
1.5 mm thick/12.5 mm overlap	334 ± 11
2.5 mm thick/12.5 mm overlap	354 ± 10
2.5 mm thick/25.0 mm overlap	428 ± 38
2.5 mm thick/50.0 mm overlap	633 ± 63
5251 aluminium alloy	
1.6 mm thick/12.5 mm overlap	191 ± 14
3.0 mm thick/12.5 mm overlap	325 ± 28
6AL-4V titanium alloy	
2.0 mm thick/12.5 mm overlap	457 ± 52
Unidirectional T300/924 carbon/epoxy	
2.0 mm thick/12.5 mm overlap	369 ± 41
Plain woven fabric (Tufnol 10G/40)	
2.5 mm thick/12.5 mm overlap	275 ± 28
2.5 mm thick/25.0 mm overlap	454 ± 27
2.5 mm thick/50.0 mm overlap	511 ± 32
5.1 mm thick/12.5 mm overlap	327 ± 27

Table 2. Failure load per unit width for epoxy bonded single-lap joints

The strength, and hence fatigue resistance of bonded joints is dependent on the adherend material, adherend thickness, bond-line thickness and bond length. Table 2 compares the failure load per unit width (N/mm) for various single-lap joint configurations and materials bonded with an epoxy adhesive [65-67]. Table 3 demonstrates the sensitivity of single-lap strength on bond-line thickness and bond length.

The joints consisted of mild steel sections bonded with an elastomer adhesive. Small variations in bond-line thickness can result in significant changes in bond strength. For comparative studies, careful consideration should therefore be given to ensuring that the stress and strain distributions (i.e., maximum peel and shear stresses at the ends of the joint) for different systems are at least similar.

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

Table 3. Failure load (N) for mild steel/elastomer single-lap joints

Basic design considerations for maximising the static strength and fatigue performance of adhesively bonded joints include:

- Increasing the overlap area results in higher joint strength. The failure load is directly proportional to specimen width. Increasing the joint width by a factor of 2 will double the failure load, however increasing the bond length by a factor of 2 will not necessarily result in a two-fold increase in bond strength (see Table 2).
- Increasing either the adherend stiffness (i.e., elastic modulus) or adherend thickness results in an increase in load-bearing capacity of the single-lap joint. The use of stiff or thick adherends will minimise peak stress levels and yield a more uniform adhesive stress distribution. However, the use of absolutely rigid adherends will not prevent the formation of stress concentrations at the bond-line.
- The total overlap length must be sufficiently long to ensure that the shear stress in the middle of the overlap is low enough to avoid creep. Short overlaps can result in failure through creep-rupture. It is recommended that the overlap length is ~10 times the minimum adherend thickness to ensure a uniform shear distribution.
- Increasing the overlap lengths beyond this value does not result in substantial increases in static and fatigue performance. The low stress region in the middle of a long overlap contributes to joint strength by providing elastic restoring force or reserve. It is recommended to maximise bond area. Longer overlap lengths are highly desirable (provided cost and weight penalties are not too high).
- Minimise shear and peel stress concentrations - shear and peel stress concentrations present at bond-line ends can be reduced by the use of a combination of tapered or bevelled external scarf or radiused adhesive fillets. Significant increases in the joint strength compared with square-ended bond-lines can be achieved. It is recommended that the taper ends of lap joints should have a thickness of 0.76 mm and a slope of 1/10.

- Ensure the joint is loaded in the direction of maximum strength of the adherend. The bonded joint needs not only to be loaded in the direction of maximum strength, but also loads in the weak directions need to be minimised.
- Maintain a uniform bond thickness and wherever possible join identical adherends to minimise skewing of the peak and normal stresses, and to minimise thermal residual stresses and bending stresses due to differences in coefficient of thermal expansion (CTE) values of the adhesive and adherends.
- In the case of fibre-reinforced laminates, avoid interlaminar shear or tensile failures of composite adherends. Also, ensure the laminated adherend is symmetric, thus ensuring the coupling stiffness components of the laminate are zero (i.e., no twisting).
- Mode of loading has a significant affect on joint strength and long-term performance (i.e., compression > shear > tension). The presence of tensile or peel stresses at the end of overlap regions will reduce joint strength.

Additional points to note regarding adhesive joint testing are listed below.

- The stress distribution within the bond-line tends to be non-uniform in a majority of test configurations with stress concentrations existing at the bond-line ends. Premature failure will often occur as a result of these stress concentrations.
- Generally, the time taken for environment effects to become apparent increases with joint size, thus test joints with small bond areas, or with large bond-line perimeters compared with the bonded area are preferred for accelerated testing.
- The accuracy and reliability of displacement measurements are often in question as the magnitude of displacements is often small.

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Chapter 3

Fatigue testing

- Introduction
- Constant amplitude cyclic loading
- Fatigue life assessment
- Welding parametric effects on fatigue performance

Introduction

This Chapter gives guidance on cyclic fatigue testing of adhesively bonded and welded joints, and is primarily concerned with structural adhesives and thermoplastics, and constant amplitude and frequency tension-tension fatigue loading conditions. It is recognised that for many engineering applications, variable amplitude, and frequency loading (i.e., spectral loading) is more realistic of what happens in practice, and that analysis and life prediction for spectral loading is more complicated than constant amplitude and frequency regimes. It should also be recognised that the stiffness of plastic adherends may be considerably less than structural materials, such as metals and fibre-reinforced plastics (FRPs) encountered in bonded structures. This may have relevance when monitoring stiffness changes and strain control during testing.

Constant amplitude cyclic loading

Minimum stress, σ_{MIN}
Maximum stress, σ_{MAX}
Stress range, $\Delta\sigma = \sigma_{MAX} - \sigma_{MIN}$
Stress amplitude, $\sigma_A = \Delta\sigma/2 = (\sigma_{MAX} - \sigma_{MIN})/2$
Mean stress, $\sigma_{MEAN} = (\sigma_{MAX} + \sigma_{MIN})/2$
Stress ratio, $R = \sigma_{MIN}/\sigma_{MAX}$
$0 \leq R \leq 1$ for tension-tension loading
$-\infty \leq R \leq 0$ for tension-compression loading
$1 \leq R \leq +\infty$ for compression-compression

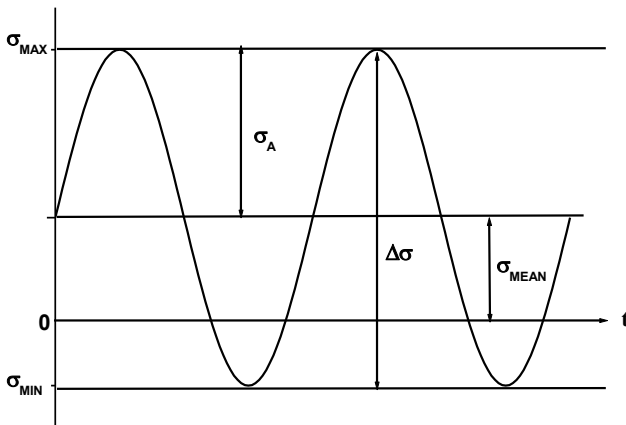


Figure 6. Nomenclature for stress parameters for constant amplitude cyclic loading

The principle of the method, as described in ISO 13003 [68], is that a continuously alternating mechanical load or displacement is applied at a constant frequency to the specimen under test until the specimen either fails or reaches a certain number of fatigue cycles (fatigue life). This load is applied in combination with a specified mean load (which may be zero) – see Figure 6 for the nomenclature for stress parameters for constant amplitude cyclic loading. By testing specimens at each of several percentage levels of the ultimate stress or strain, a plot of the stress-strain versus number of fatigue cycles can be constructed. This plot provides information on the fatigue life of the material or the number of fatigue cycles the material can sustain at a certain stress-strain level before failure occurs.

The test may be carried out at a constant stress (load) amplitude, constant strain amplitude or constant displacement amplitude. The test method, specimen geometry, dimensions and calculations are the same as those used in the equivalent test mode under static (monotonic) loading conditions. The fatigue properties of adhesively bonded joints are a function of the joint geometry and adhesive, and therefore cannot be determined from the intrinsic properties of the adhesive. It is therefore necessary to conduct cyclic fatigue tests on representative joints to those to be used in service.

Fatigue regimes

Fatigue regimes that exist range from compression-compression ($1 \leq R \leq +\infty$), tension-compression ($-\infty \leq R \leq 0$) to tension-tension ($0 \leq R \leq 1$) – see Figure 2 in ISO 13003. The results presented in this guide relate to a sinusoidal cyclic waveform only (Figure 6), but it is noted here that other waveform shapes are commonly used (i.e., triangular, square, sawtooth, etc.) and that proprietary test machine control software allows the user to input bespoke waveforms. The selection of the most appropriate fatigue regime to use is directly linked to the material application requirements and anticipated stress field.

Test frequency

Fatigue data are normally obtained at the highest frequency possible to minimise the duration of tests. Restrictions on test frequency can arise from test equipment limitations (response time), time dependent processes and hysteretic (self-generated) heating. Hysteretic heating, which increases with increasing load and frequency, can result in thermal softening of the polymer (and FRP adherends), adversely affecting the fatigue performance of the material and causing erroneous stiffness and life expectancy measurements. The amount of heat generated in a material under fatigue loading will depend on several factors, including stress/strain amplitude, specimen displacement range (stroke), test frequency and the ability of the material or component to dissipate heat.

The temperature rise of the specimen surface is normally limited to 10 °C, but this depends on the sensitivity of material properties to rises in temperature. It is recommended that even where prior knowledge of test frequency versus heat rise is known for a material, a thermocouple should be attached to the surface of initial test specimens to monitor the degree of hysteretic heating.

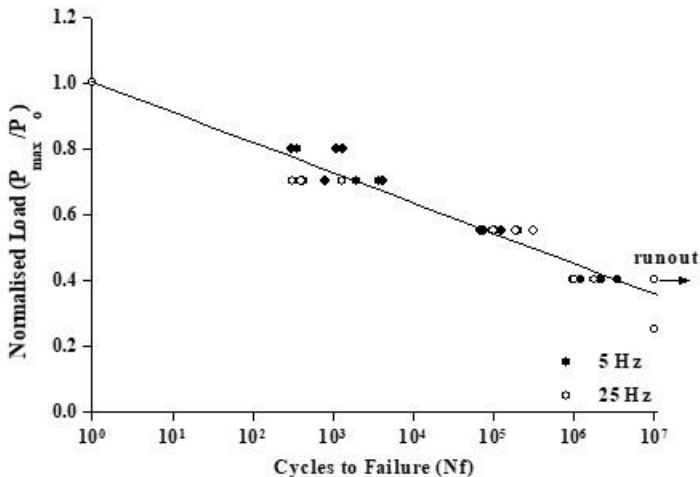


Figure 7 Normalised S-N curve for mild steel/epoxy single-lap joints

It is recommended that the temperature rise of the material surface be kept to a minimum. It may be necessary to stop testing to allow the specimen to cool. Alternatively, the test could be carried out in an environmental cabinet with a thermocouple attached to the specimen surface for monitoring and controlling the temperature of the test specimen, although the cooling rate may be too slow to be practical. Thermal imaging equipment can be used to monitor surface temperature, although the latter is beyond the budget of most industrial facilities. The temperature resolution is ~1 °C for the two methods. Tests should be carried out at each stress level to ensure the temperature rise is kept to less than 10 °C, and if necessary, the test frequency should be reduced to prevent over-heating. For temperature sensitive materials, it may be necessary to set a lower limit on the temperature rise. These comments do not apply to any rapid temperature rise associated with final failure.

Reliable data can be obtained at high frequencies provided the stress levels are low. Test frequencies of the order of 10 to 30 Hz can result in substantial heating, particularly in the grip regions. The upper frequency limit will be dependent upon the thermal conductivity of the joined system, mode of loading and specimen size. Trials may be necessary to determine the upper frequency limit. The results presented in Figure 7 for single-lap joints show no evidence of a self-heating effect, and hence the merging of fatigue results for 5 Hz and 25 Hz.

It is unlikely that test frequencies of the order of 5 Hz (except for low amplitude vibration), or higher will be experienced in service. Fatigue resistance decreases with a reduction in frequency; the decrease is attributable to creep deformation. At a test frequency of 5 Hz, 10^7 loading cycles is equivalent to 23 days testing. Increasing the test frequency to 25 Hz reduces the test duration to less than 5 days.

Determination of ultimate material properties

To determine the relative stress/strain levels to use in a fatigue test the ultimate properties (i.e., strength, strain to failure, etc.) should first be measured at a loading rate equivalent to fatigue testing conditions (i.e., test frequency). When undertaking measurements of ultimate properties, it is important to recognize that stiffness and strength of polymeric materials are rate dependent. The fatigue test rate is defined as that resulting in failure in a time equivalent to $0.5 \times$ the cycle time. Mean values for ultimate properties should be determined from tests undertaken on at least 6 specimens.

Number of test specimens

For the determination of the lifetime diagram, five specimens should be tested at a minimum of four stress levels, or preferably five stress levels (80%, 70%, 55%, 40% and 25% of the fatigue rate ultimate strength). A minimum of 6 fatigue tests should be conducted at each stress level (unless otherwise specified). It is advisable to increase the number of tests per level when carrying out statistical analysis for generating design data.

Specimen preparation and test conditions

Specimen preparation, specimen geometry, loading arrangement and environmental test conditions should be the same as those employed for the monotonic tests (see chapter 2). The applied conditions (i.e., peak load, strain and/or displacement data) when stabilized should be recorded throughout the duration of test.

Analysis of fatigue data

Cyclic fatigue data is generally presented in the form of a stress-cycle (S - N) diagram (i.e., a plot of the fatigue life (number of cycles to failure N_f at various levels of fluctuating stress). For inter-comparative purposes, fatigue strength data (see Figure 7) are normalised with respect to the ultimate static strength P_{ULT} (or P_0) of identically conditioned specimens measured at an equivalent loading rate to the test frequency. The uncertainty in life expectancy at any stress level is large (typically an order of magnitude).

Joint stiffness: In addition to recording the number of cycles to failure, the stiffness properties of specimens can be monitored throughout the test at each logged cycle. This is carried out to see whether changes in stiffness occur due to growth of the damage throughout the fatigue tests, and if there are changes in stiffness, where in relation to the failure cycle these changes

occur. The simplest way of calculating the modulus is to use the minimum and maximum stresses and corresponding strain levels derived from the recorded actuator positions. Whilst this method is relatively simple, the fact that actuator positions are used to calculate strains can lead to errors due to the compliance of the loading train. Using devices such as clip gauge extensometers, LVDTs and strain gauges, can eliminate compliance errors but ensuring that these types of devices do not detach or de-bond from specimens during the fatigue test is crucial (see Chapter 2).

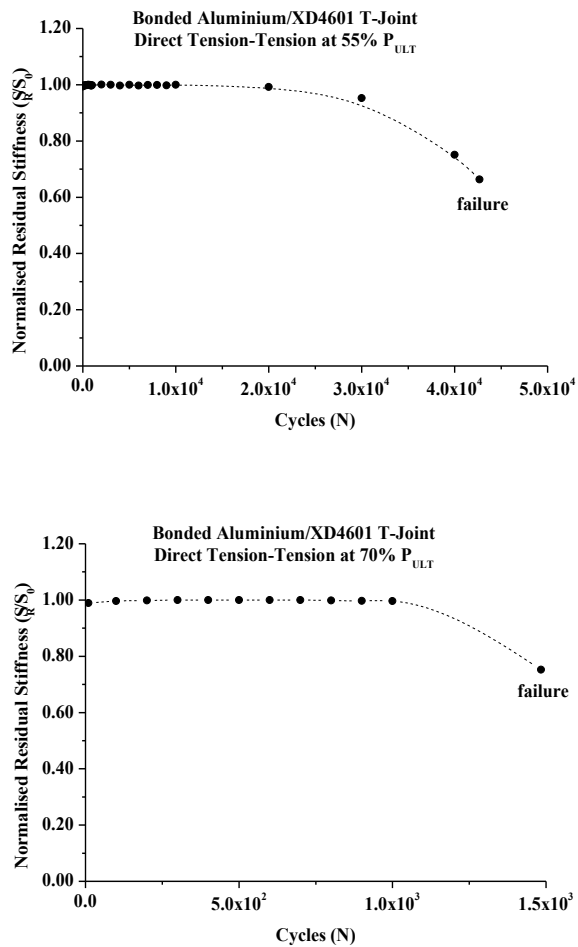


Figure 8. Normalised residual stiffness for fatigue loading of aluminium T-joints

The joint stiffness (i.e., dynamic compliance) will often decrease with the onset of damage within the joint (Figure 8). Ultimate failure is marked by a rapid reduction in joint stiffness and an increase in the loss or damping factor ($\tan \delta$). The loss factor $\tan \delta$ is the ratio between the storage modulus E' and the loss modulus E'' (i.e., $\tan \delta = E'/E''$) where δ is the phase angle between dynamic load/stress and the dynamic displacement/strain (see Figure 9) [59, 68]. The storage modulus is proportional to the maximum energy stored during a loading cycle and represents the stiffness of the joint. The loss modulus is proportional to the energy dissipated (lost) during one loading cycle.

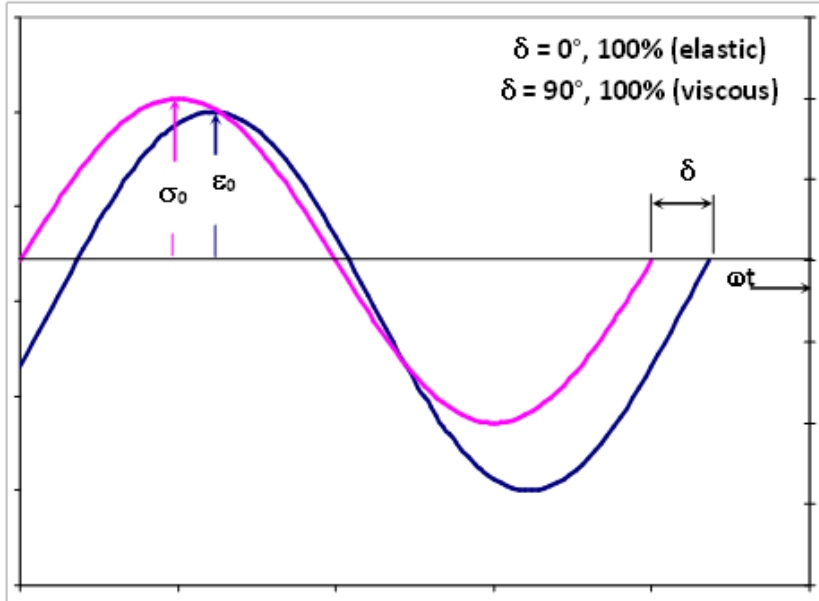


Figure 9. Out-of-phase stress strain response for viscoelastic material, $\delta = 20^\circ$ [68]

The stress σ and strain ε are given by the following relationships (see Figure 9):

$$\sigma = \sigma_0 \sin(\omega t + \delta); \quad \varepsilon = \varepsilon_0 \sin(\omega t) \quad (1)$$

Where ω is the period of strain oscillation, t is time and δ is the phase angle in radians.

The storage modulus E' and the loss modulus E'' are given by:

$$E' = \frac{\sigma_0}{\varepsilon_0} \sin \delta; \quad E'' = \frac{\sigma_0}{\varepsilon_0} \cos \delta \quad (2)$$

For a purely elastic material, the mechanical energy stored during loading is returned completely when the specimen is unloaded. This generates load/displacement or stress/strain curves for the material, which are completely in phase with each other (i.e., $\delta = 0^\circ$), producing no hysteresis (see Figure 10). At the other extreme a purely viscous material (i.e., $\delta = 90^\circ$), which exhibits no elasticity only damping, results in all the energy being dissipated and the load/displacement or stress/strain curves being 90° out of phase with each other. Materials that fall between these two categories exhibit a phase lag (Figure 9) and are classified as viscoelastic.

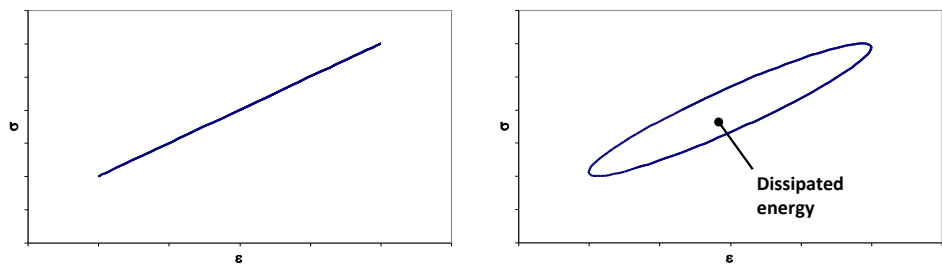


Figure 10. Hysteresis (stored energy): (left) elastic and (right) viscoelastic response [68]

When viscoelastic materials are subjected to fatigue or cyclic motion a proportion of the mechanical energy per cycle is converted to thermal energy (heat) with the rest of the energy being stored. A hysteresis loop develops whose area is equal to the dissipated energy per cycle (Figure 10). The stiffness and hysteresis can be derived mathematically – see [68].

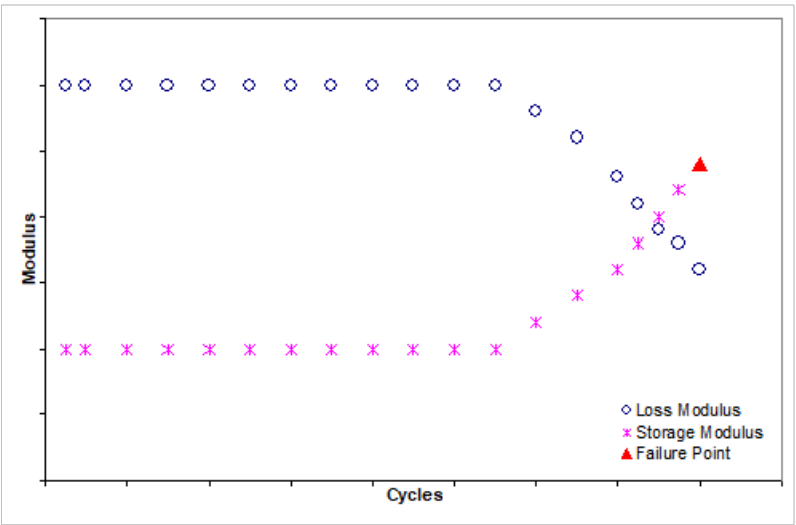


Figure 11. Storage and loss modulus changes [68]

Analysis of data on specimens that have been fatigued to failure should show that over time the stiffness of the material decreases towards failure with the energy loss of the material becoming greater as damage accumulates and friction causes material heating (Figure 11). As damage accumulates within the material, the slope of the ellipse will decrease and the area within the ellipse will increase. For a considerable proportion of the fatigue life (50-60%), stiffness and hysteresis will remain reasonably constant (see Figure 8). There will be a rapid change in stiffness and hysteresis within the last few hundred cycles. Although measurement of global stiffness is relatively straightforward using a linear voltage displacement transducer (LVDT) or strain gauges, it is often difficult to obtain an accurate measurement of the hysteresis. Localised deformation measurements can be obtained using strain gauges bonded to the specimen surface. These gauges if strategically located at critical stress regions, may indicate the onset of localised damage.

Fatigue (endurance) limit is the maximum fluctuating stress a material can endure for an infinite number of cycles (Figure 12). Under constant amplitude loading conditions, most materials or structures seem to exhibit a plateau in the stress-cycle curve, which typically occurs at $N > 10^6$ cycles. The plateau level corresponds to the fatigue or endurance limit. Below this limit, the material or structure can in principle be cycled indefinitely without causing failure. Recent work conducted at NPL on unidirectional glass/fibre-reinforced epoxy suggests that given sufficient time, fatigue failure would occur at stress levels less than 25% of the ultimate strength. This may also apply to adhesive and welded joints.

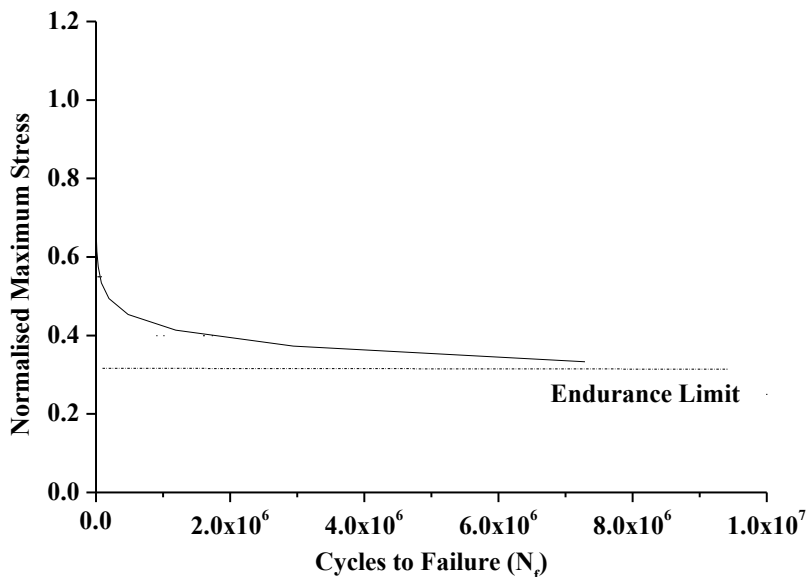


Figure 12. Typical S-N curve with fatigue or endurance limit

Fatigue life assessment

In order to compare fatigue performance, fatigue strength data are often normalised with respect to the ultimate static strength P_{ULT} of identically conditioned specimens measured at an equivalent loading rate to the test frequency. Normalised $S-N$ curves for bulk adhesives and bonded joints (see Figures 7 and 13) can be approximated by the following relationship:

$$P_{MAX}/P_{ULT} = 1 - k \log N_f \tag{3}$$

P_{MAX} is the maximum load applied to the specimen, P_{ULT} is the ultimate strength of the joint and N_f is the number of cycles to failure.

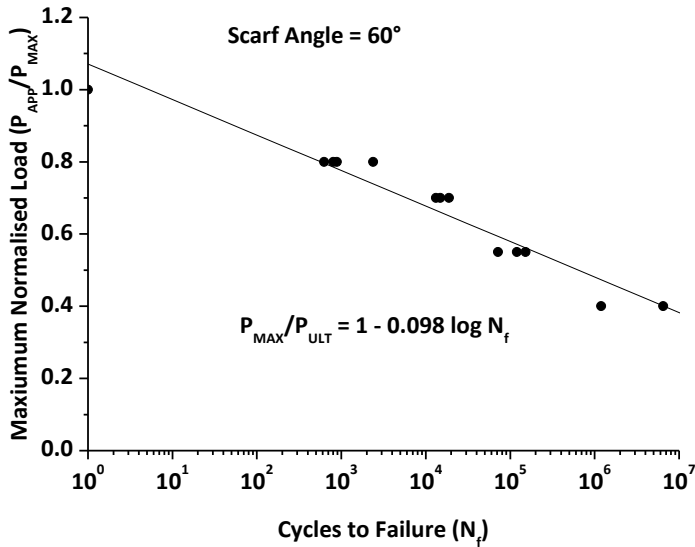


Figure 13. Normalised $S-N$ curve for a mild steel/epoxy scarf joint ($R = 0.1$, $f = 5$ Hz)

Fatigue performance of an adhesive joint can be approximated by (rule of thumb) Equation 3 where k the fractional loss in strength per decade of cycles is a measure of fatigue resistance of the joint. The lower the k value the better the fatigue performance. The value of k is dependent on joint geometry and stress ratio. Table 4 shows typical k values for several metal and composite joints bonded with epoxy adhesives. A scarf joint with an angle of 15° with respect to the horizontal where failure is dominated by shear stresses has a far better fatigue performance than tests where peel or hydrostatic tensile stresses are the major cause of failure (e.g., T-peel joints and 60° scarf joints (see Figure 13)). Cyclic fatigue performance is highly sensitive to bond-line thickness.

Joint Type	k
Scarf (steel adherends with 15° angle)	0.063
Thick adherend shear test (steel adherends)	0.063
Double-lap (titanium adherends)	0.075
Double strap (aluminium adherends)	0.088
Single-lap (mild steel adherends)	0.093
Double-lap (woven fabric)	0.097
Scarf (steel adherends with 60° angle)	0.098
T-joint (aluminium adherends)	0.104
T-peel (mild steel adherends)	0.130

Table 4. Typical k Values for bonded joints ($R = 0.1$ and $f = 5$ Hz)

A bond-line thickness of the order of 0.2-0.3 mm tends to have far superior fatigue resistance than thicker bond-lines (> 0.5 mm); probably because of lower porosity within the adhesive. An increase in temperature or moisture ingress will compromise the fatigue performance of polymers and adhesive joints (k can be expected to increase).

Constant-life diagrams are often used to represent the effects of mean stress and stress amplitude on fatigue performance of adhesive joints. Different combinations of normalised stress amplitude, $\Delta P/P_{ULT}$, and the normalised mean stress, P_{MEAN}/P_{ULT} , can be plotted to give constant fatigue life curves. Figure 14 shows normalised stress-amplitude plots for different mean stress values. The stress values are normalised with respect to the ultimate tensile strength, σ_{UTS} , of the material. In principle, the curves should converge to the static strength of the material on the mean stress axis (i.e., when the mean load is increased to the static strength then no amplitude is required to cause failure).

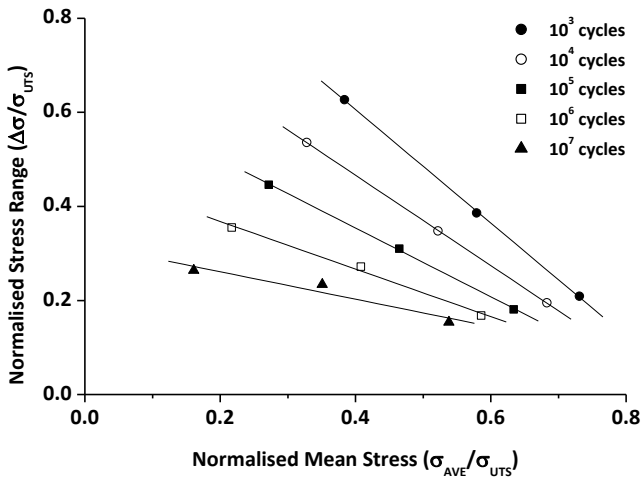


Figure 14. Stress amplitude-life plots for different mean stress values

Several models have been suggested for determining stress amplitude-life plots of polymeric materials [69]. A Goodman type curve has been shown to be a valid method of representing the effect of mean stress and stress amplitude on fatigue performance of single-lap and T-peel joints [70]. The Goodman relation is given below [69]:

$$P_A = P_{FS} \left(1 - \frac{P_{MEAN}}{P_{ULT}} \right) \quad (4)$$

P_A is the stress amplitude (for a non-zero mean stress), P_{FS} is the fatigue strength (for a fixed life), P_{MEAN} is the mean stress and P_{ULT} is the ultimate strength of the material.

An alternative approach for estimating fatigue life is to apply a model originally developed for fibre-reinforced plastics known as the normalised life prediction model [71]:

$$\frac{(1 - R)\sigma_{MAX}^*}{2 - (1 + R)\sigma_{MAX}^*} = \left(\frac{1}{2N_f} \right)^{\frac{1}{n^*}} \quad (5)$$

The fatigue strength is normalised with respect to the ultimate (static) strength in the loading direction:

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{ULT}} \quad (6)$$

For tension-tension and compression-compression fatigue, respectively:

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{UTS}} \quad (7)$$

$$\sigma_{MAX}^* = \frac{\sigma_{MAX}}{\sigma_{UCS}} \quad (8)$$

σ_{UTS} and σ_{UCS} are the ultimate tensile and compressive strengths, respectively.

The value of n^* is derived from fatigue tests carried out at three different stress ratios R (e.g., 0.1, 0.5 and 0.75) and at different values of maximum stress (i.e., 80%, 70%, 55%, 40% and 25%).

For tension-tension fatigue ($|R| \leq 1$), the value of n^* has been determined for unidirectional glass/epoxy and bulk epoxy adhesive DP460 and found to be approximately 10 and 12, respectively.

The value of n^* for steel scarf joints bonded with DP460 epoxy adhesive with a 60° scarf angle with respect to the horizontal was observed to be ~10. The higher the value of n^* the better the overall fatigue resistance of the polymeric system.

Welding parametric effects on fatigue performance

Cyclic fatigue behaviour seems to be sensitive to welding conditions with fatigue resistance at elevated stresses decreasing with an increase in welding temperature. Table 5 compares the average fatigue life obtained from tension-tension fatigue tests conducted at a stress level of 70% of the ultimate tensile strength. The stress ratio was 0.1 and the test frequency 5 Hz. Five test specimens were tested per batch. Fatigue performance is highly sensitive to the presence of the weld as shown by the large decrease in fatigue performance. The uncertainty in fatigue life data was typically $\pm 25\%$ for both the parent and welded materials.

Material	Average Fatigue Life (Cycles)
Parent	45,581
Welded	
198 °C	8,141
216 °C	5,637
232 °C	5,519
250 °C	4,000

Table 5. Average fatigue life of welded and unwelded polypropylene

Table 6 indicates that the differences between the tensile strength obtained for polypropylene for different weld temperatures is minimal. The difference in tensile strength between parent material and welded polypropylene is ~12% at 23 °C and 6% at 50 °C.

Material	Tensile Strength (MPa)
Parent	27.0 \pm 3.0
Welded	
198 °C	23.7 \pm 0.3
216 °C	23.6 \pm 0.4
232 °C	23.1 \pm 0.3
250 °C	23.0 \pm 0.4

Table 6. Tensile strength of welded and unwelded polypropylene

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Chapter 4

Creep testing

- Introduction
- Creep rupture
- Creep testing
- Creep behaviour
- Procedure for modelling creep in polymers

Introduction

Creep is the increase in strain or deformation of a material with time when the material is subjected to a constant load for an extended period of time (i.e., time-dependent deformation). The change of strain at any time increases with load (stress), stress state, temperature and relative humidity. Viscoelastic materials, such as thermoplastics and adhesives, can undergo creep deformation at relatively low stress levels (well below the ultimate strength of the material) and low temperatures (i.e., room temperature - referred to as cold flow) compared with many structural materials (metals, glass and concrete). This can lead to considerable reduction in life expectancy of the component. Strain (or deformation) increases with load, temperature, relative humidity and time.

It is therefore important to be able to predict the effects of long-term loading on deformation and failure behaviour. Confidence in predictions requires the use of models and test methods that accurately characterises the deformation behaviour of the polymeric materials in particular joined systems. This sChapter provides guidance on creep testing of polymers, and adhesive and welded joints. Many of the aspects of specimen preparation and testing have been covered in Section 2.

Creep rupture

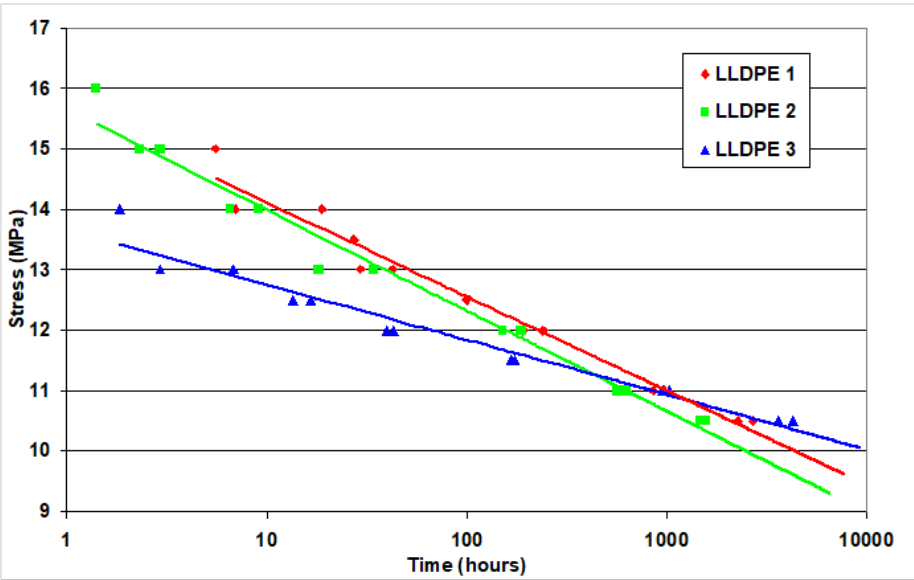


Figure 15. Time to failure of three grades of LLDPE full notch creep test (FNCT) specimens [38]

Creep rupture (or static fatigue), which refers to time-dependent failure of the material when subjected to constant load, is used as a measure for gauging the relative resistance of the material to chemical environments. It can also be used to assess the quality of welds. Tests are conducted at several stress levels (i.e., 80%, 60%, 40% and 20% of the ultimate strength) and the deformation and time-to-failure are recorded. A plot is produced of applied stress versus time-to-failure (Figure 15). It is recommended that six specimens be tested per stress level. The less resistant a material is to a particular environment, the more rapid the drop in the rupture stress time curve. For creep rupture tests, the duration of the test is taken from the moment the test load is reached until the specimen fractures.

For a given applied load, the stress rupture time decreases with increasing humidity and temperature, or chemical concentration. The Full Notch Creep Test (FCNT) [61] shows differences in the ESC resistance, but can be misleading if creep tests are not conducted for a sufficient length of time (up to 1 year). Figure 15 compares the time to failure of three grades of linear low-density polyethylene (LLDPE) in which there is a crossover point at approximately 1,000 hrs – see NPL Measurement Good Practice Guide No. 103 [38].

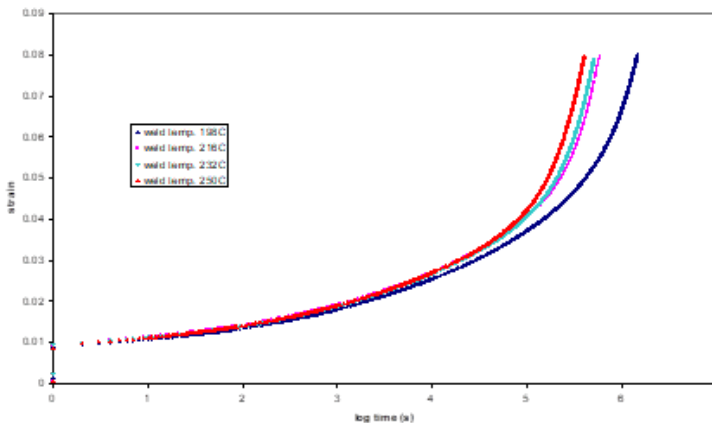


Figure 16. Tensile creep of welded PP for different hot plate temperatures (applied stress of 13.6 MPa)

In thermoplastic systems that have been well fused (bonded), there will be minimal difference in creep behaviour at moderate stresses (≤ 9 MPa for polypropylene). At higher stresses, however, differences in creep behaviour of hot plate welded polypropylene becomes apparent with the creep rate increasing with increasing weld temperature. Figure 16 compares the tensile creep behaviour of hot plate welded polypropylene tested at 13.6 MPa and 23 °C. The hot plate temperature was varied (198 °C, 216 °C, 232 °C and 250 °C) whilst fusion pressure, hot soak time and cooling time were kept constant. Fusion pressure was 0.1 Nmm⁻², hot soak time 135 seconds and cooling time 6 minutes.

Creep testing

The information given below is intended to complement BS EN 12814-3 [49], ISO 899-1 [50], NPL Measurement Good Practice Guide No. 2 [29] and Chapter 2.

Test apparatus and test conditions

Loading system and gripping device: The creep rig should have a rigid frame (no deformation of the test frame is allowed within the applied loading range), which allows the load to be applied smoothly, without causing transient loading, and be capable of maintaining the load to within $\pm 1\%$ of the required load. Dead weight cantilever loading frames are recommended with the construction of the link between the lever arm and the test specimen designed so that the lever arm is independent of the arm – see NPL Measurement Good Practice Guide No. 2 [29] and BS 1610 [72].

The load train should be as short and as stiff as possible (i.e., no universal joints included). Steps should be taken to ensure good axial alignment, and to avoid bending and torsion forces acting on the specimen. Good alignment is critical, particularly for bonded systems (see Chapter 2). The gripping mechanism should be sufficient to prevent slippage within the grips, but without damaging the material in the grip zone that could cause premature failure at high stresses. Wedge action grips are preferred. Self-locking grips that allow the specimen to move as the load increases are unsuitable for creep tests. It is important when handling and testing adhesively bonded and welded specimens that care is taken to avoid any lateral loads being placed on the specimens, and that overloading is also avoided. There are no recommended loads, however ISO 11403-1 [73] recommends 20%, 40%, 60% and 80% of the ultimate load [23].

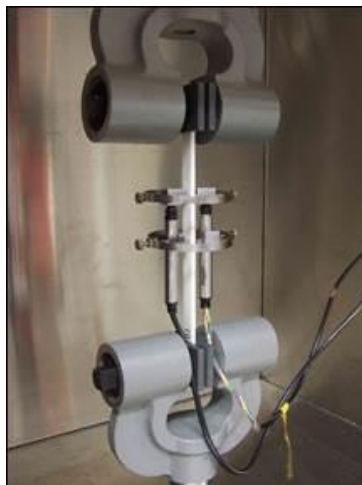


Figure 17. Tensile creep specimen with extensometers

Extension measurement: The extension gauge-length should cover the region of interest. Extension can be measured using either contact or non-contact devices. It is essential that when attaching contact extensometers that the devices do not influence material behaviour or cause damage. Extension should be measured using two separate lightweight extensometers, preferably with blunted knife-edges, applied either side of the specimen to check alignment (see Figure 17). The readings from the two extensometers are averaged. The accuracy of the extension-measuring device needs to be better than $\pm 1 \mu\text{m}$. For low absolute strain levels encountered at small stresses or during the initial period of creep tests the accuracy generally should be better than $\pm 0.25 \mu\text{m}$.

Non-contact or optical extensometers (e.g., video extensometers) are available, which avoid contact damage and can be used up to failure, since there is no possibility of damage to the extensometer. Video extensometers are not particularly suited to measuring small strains (e.g., movements of a few mm), which limits their applicability to systems involving small deformations, such as structural adhesive joints. There are however no restrictions on the upper limit. This is a convenient method for determination of strains above 10% (i.e., suitable for use with rubbers or thermoplastics). Video extensometers are recommended for creep rupture tests. The gauge length is marked on the specimen either by attaching metal clips with scratched on gauge marks or by ruling the gauge marks with an inert, thermally stable paint.

Strain gauges can be used, but the use is limited. Care has to be taken to ensure the strain measurements obtained are reliable, as strain gauges tend to locally stiffen polymers. The strain measured tends to be lower than that expected for a given load [74]. For many polymeric materials, it is difficult to produce a reliable adhesive bond between the gauge and the polymer. Strain gauges are not particularly suited for creep rupture tests where strains can be very high (5-10%). Before embarking on using strain gauges it is advisable to obtain specialist advice.

Strain and load should be recorded at regular intervals or at times that form regular intervals of $\log t$, where t is duration. A data acquisition system is recommended for recording load and displacement as a function of time. The time recoding equipment should be accurate to within $\pm 0.1 \%$ or $\pm 2 \text{ s}$. The data collection system should also be programmed to set the creep time to zero at the instant of the load application. It is recommended in ISO 899-1 [50] that measurement schedule should be:

- 1, 3, 6, 12 and 30 min and then 1, 2, 5, 10, 20, 50, 100, 200, 500, 1,000 h, etc.

More frequent checks may be required if discontinuities are suspected. Higher logging rates are generally required at the start of the creep test. It may be necessary to remove early points to remove the effect of load application.

Test conditions: The creep behaviour of polymeric materials is sensitive to temperature, and therefore it is critical to keep the temperature of the specimen constant to within $\pm 0.5^\circ\text{C}$, which should be recorded regularly. This also applies for tests performed at elevated temperatures. Creep tests should be carried out in a temperature-controlled chamber. The humidity should also be controlled. Strain repeatability for creep tests performed on the same specimen at low stresses should be within $\pm 2\%$.

For material qualification purposes, it is recommended that for each stress level and temperature selected that at minimum of 6 welded and 6 unwelded specimens be tested. For modelling purposes, it may not be necessary to conduct that many tests in order to obtain reliable estimates for the creep parameters. The resistance to slow crack growth of the material can be determined using the FNCT – see [49, 61].

Loading modes

It is possible to generate creep compliance curves for all three modes of loading (i.e., tension, compression and shear) on polymers (including adhesives) and welded thermoplastics. This section provides a brief description of test methods suitable for generating creep data for the three loading modes.



Dumbbell Tensile Specimen



Compression Test Specimen

Figure 18. Tensile and compression test specimens

Tension: Tensile specimens typically consist of a waisted section with parallel sides (Figure 18) to facilitate strain measurements and to ensure failure occurs within the gauge-section away from the gripped ends. ISO 527-2 [75] specifies a method for determining the tensile properties of polymers. Modulus is determined over the strain range 0.05% to 0.25% (provided the region on the stress-strain curve is linear).

Strain distribution along the gauge section will invariably be non-uniform. In fact, rapid and large deformation may occur at several locations away from the mid-section. This is particularly an issue for polymers exhibiting large strain to failure, such as toughened epoxies and thermoplastics. Localised yielding within the welded region can be expected to be predominant for welded specimens.

For welded specimens, the creep deformation measured is an average effect of the material within the gauge section, which includes the melted zone. Although the melted zone is small (i.e., narrow strip less than 0.1 mm), its influence is large due to a lower creep resistance. Consideration should be given to using a smaller gauge section commensurate with the size and contribution made by the welded region to creep deformation.

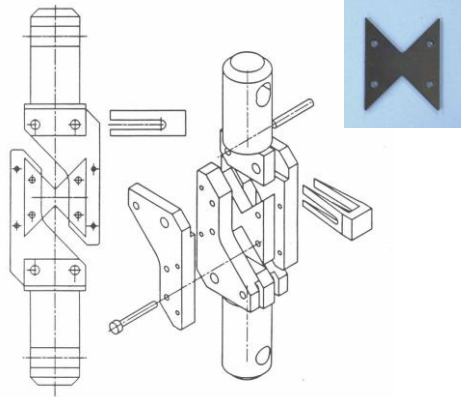
Compression: Creep tests under uniaxial compression are carried on small rectangular specimen (typically 12 mm long x 10 mm wide and approximately 4 mm thick) – see ISO 604 [76]. Specimens are loaded along the length direction between parallel platens. It is recommended that an alignment jig be attached to one of the platens to enable the platen faces to be set parallel using slip gauges. It is important that the two edges in contact with the top and bottom compression platens are flat and parallel to prevent buckling, which can result in premature failure and cause errors in strain measurements. Contact surfaces are also lightly oiled to reduce friction effects. Longitudinal strain is measuring using two contact extensometers applied directly between the loading platens mounted adjacent to the front and back surfaces of the test specimen. Nominal strains are derived from the original specimen length. Although the test geometry is used for determining Young's modulus, small displacement measurements tend to be inaccurate and difficult to repeat. Corrections need to be made to the strain values of around 0.005 arising from the initial displacements in the regions of the specimen/platen interface. Checks also need to be carried out to ensure specimens do not buckle.

Shear: Shear properties can be measured using either the Arcan or V-notched beam (ASTM D 5379 [77]) test methods. Both methods employ a notched (or a butterfly shaped) specimen (Figure 19) and can be used to generate stress-strain curves. The notches define the geometry, such that a predominantly pure stress state is established in the central region of the specimen. Strain can be measured using biaxial strain gauges bonded to both the front and back faces at $\pm 45^\circ$ to the applied load or contact extensometers mounted on the front and back surfaces as in the case for the Arcan method. Specimens are loaded in alignment fixtures (V-notched beam in compression and Arcan in tension).

The notch tip separation is 12 mm and radius of the notches 1.5 mm for both types of specimen. Although a special test fixture is required, testing is relatively straightforward. To minimise potential effects of out-of-plane movement or twisting of the specimen, it is recommended that the strain data used for determining shear modulus be the average of the indicated strains from each side of the specimen.



V-notched Beam Method



Arcan Method

Figure 19. Shear test methods

Measurements of shear modulus for both methods have been found to correlate well with other techniques, whereas the “apparent” shear strength value tends to be low. Failure often occurs as a result of high transverse tensile stresses on the cut out boundary, away from the gauge-section. Tensile failure is characteristic of brittle polymers (thermosets) with cracks propagating in an unstable manner along the plane of the principal tensile stress. For these materials, ultimate failure stress does not correlate with shear strength. Thermoplastic polymers (e.g., polyethylene and polypropylene) tested at room-temperature fail as a result of shear yielding along the notch root axis. Ductile materials can redistribute the tensile stress through plastic deformation, and therefore strain data can be obtained well beyond the shear yield stress of the material. Localised high compressive stress gradients may also produce plastic deformation in the vicinity of the loading points. Compliant materials, such as polyurethane, are prone to deform at high stresses. Measurements are invalidated unless constraints are applied the specimen clamps to maintain alignment.

Creep behaviour

At short creep times, the compliance curves exhibit slight non-linearity. This non-linear behaviour increases significantly with time under load. At higher stresses, the creep curves are observed to shift to shorter times. Figure 20 shows creep compliance curves for welded polypropylene at two different stress levels. The shift to shorter times is attributed to an increase in molecular mobility brought about by the application of elevated stresses that results in a reduction in the mean retardation time parameter t_0 . It is the enhanced mobility that gives rise to non-linear creep behaviour.

The value of t_0 is dependent not only on the magnitude of the applied stress, but also the stress state (i.e., tension, compression or multi-axial), physical age of the material, temperature, humidity and moisture content. The value of t_0 decreases with an increase in temperature and humidity, and moisture content of the polymer, and conversely increases with physically ageing of the polymer.

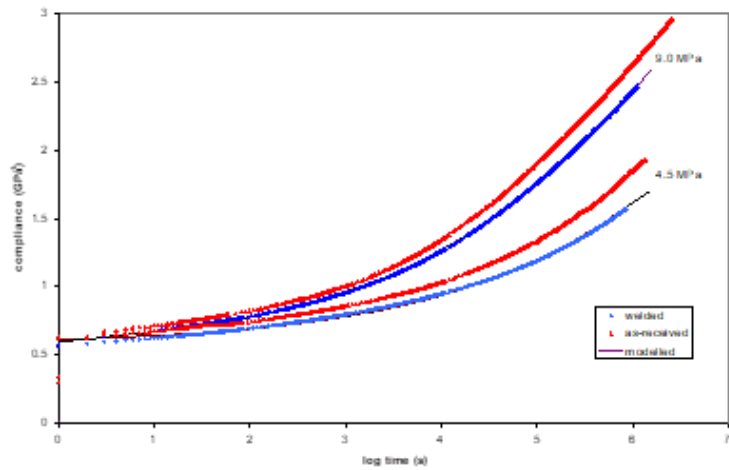


Figure 20. Tensile creep data for as-received and welded polypropylene (stress levels of 4.5 MPa and 9 MPa)

Physical ageing

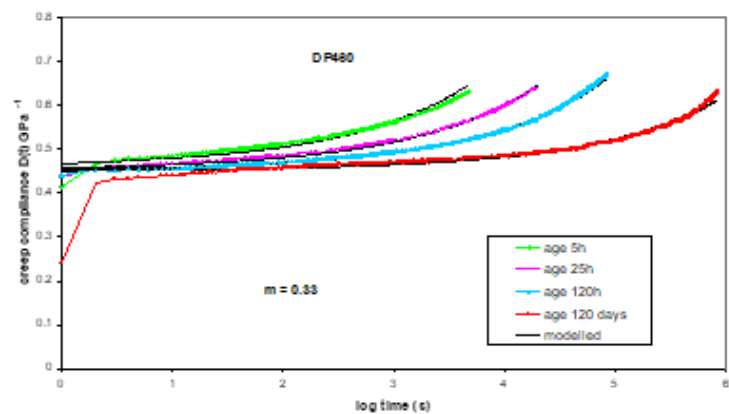


Figure 21. Creep curves for DP460 epoxy at different states of physical ageing

Physical ageing is defined as the process of progressive densification of a glassy material, which occurs with time due to conformational changes of the polymer. The driving force for these changes arises from the non-equilibrium change conformations that occur when the material is cooled from a high temperature to a low temperature (i.e., below T_g). Physical ageing is a reversible process (i.e., material aged for a number of years can be rejuvenated by short period of annealing at an appropriate elevated temperature) - see NPL Measurement Good Practice Guide No. 2 [29]. Figure 21 compares creep compliance curves for 3M DP460 epoxy adhesive at different states of physical ageing. In Figure 20, the as-received material (i.e., parent material used for producing the welded specimens) was tested at earlier time to the welded specimens, and hence the lower value of t_0 to that can be observed.

As the material is cooled, conformational changes that are needed to maintain an equilibrium structure are restricted by the increase in the relaxation times (reduced mobility) of molecular rearrangements at the lower temperatures. These non-equilibrium structures have a relatively high mobility to relaxation processes under creep loading, and this gives rise to relatively rapid creep at short elapsed times after cooling. Despite the low temperature of glassy polymers, there is sufficient molecular mobility for structural changes to take place with subsequent elapsed time (physical ageing) leading to structural states that become progressively closer to equilibrium for the low temperature. These ageing processes give rise to a reduction in molecular mobility under creep and a shift in creep curves to longer creep times as shown in Figure 21. The only parameter to change in each curve is the mean retardation time t_0 . The longer the time that has elapsed between quenching the material from an elevated temperature to a lower temperature, the larger retardation time (i.e., creep rate under load decreases with physical ageing).

The effect of physically ageing can become apparent during creep tests performed within a short period of time following quenching (i.e., within 10 days). Testing that is performed in which the age of the material is effectively the same at the start and end of the test is referred to as short-term (i.e., low stresses or short time duration (1-2 minutes)). Further ageing of material under load will occur in long-term creep tests, evident by a reduction in the rate at which the material creeps. The effect of physical ageing on creep behaviour diminishes with time. Creep tests conducted on polymers with a physical age of a 3-6 months, or more should see minimal effect on the rate of creep, provided the tests are kept short (i.e., 10 days or less).

De-ageing and pre-conditioning: It may be necessary to de-age a material by heating in order to set the clock to zero, particularly when the intention is to characterise the materials creep behaviour and where the age of the material is unknown. Physical de-ageing is the procedure of annealing test specimens at temperatures slightly above T_g for amorphous materials or below the pre-conditioning temperature for semi-crystalline materials (e.g., polypropylene), which erases any structure established by the previous thermal history – see NPL Measurement Good Practice Guide No. 2 [29] for advice on procedures for de-ageing and pre-conditioning.

Stress magnitude and stress state effects

Linear creep behaviour of polymeric materials is restricted to low stress levels (i.e., typically 1-2 MPa). At stress levels above this, non-linear creep behaviour arises because the mean retardation time parameter t_0 is dependent on the magnitude of the creep stress. The dependence of t_0 on stress level gives rise to non-linear creep behaviour. Creep curves are then observed to shift to shorter times with increasing stress (see Figure 20). At stress levels approaching the creep rupture stress, the material begins to yield (i.e., plastic deformation) and the mechanism controlling creep behaviour is different to that below the yield stress. The dominant mechanism controlling creep behaviour also alters at moderate stresses and very long test durations (i.e. plastic deformation).

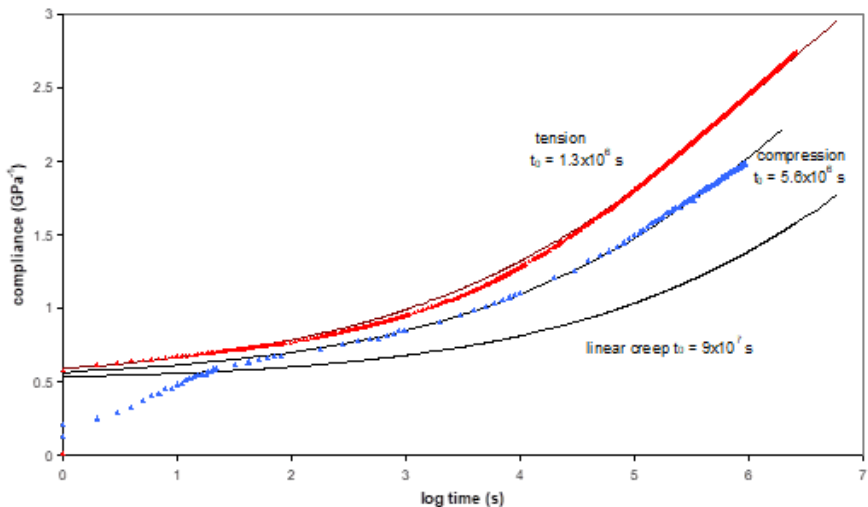


Figure 22. Tensile and compressive creep behaviour of polypropylene at 9 MPa
(calculated curve for linear behaviour under tension is also shown)

At low stresses, compliance curves in tension and compression can be expected to be the same. At higher stresses, where behaviour is non-linear, the results in Figure 22 show that the reduction in t_0 is less under compression than under tension. The rate of creep is higher in tension than in compression for the same applied stress. The magnitude of t_0 is determined, therefore, by the stress state as well as its magnitude. The retardation time parameter t_0 is determined by the magnitude of an effective stress that is a function of the shear component of applied stress as well as the hydrostatic component – see Section on modelling creep in polymers.

Temperature and moisture effects

Increasing the temperature increases molecular mobility, and hence increases the rate of creep under load. At elevated temperatures, it is important to look for signs of yielding in the creep test. It is advisable when planning creep tests at elevated temperature that viscous flow and the practical problems associated with measuring strain is taken into account. Yielding can be avoided by lowering either the test temperature or the applied stress. Non-linear behaviour may be observed at higher temperatures at stresses, which give rise to linear behaviour at lower temperatures. It should be noted that creep parameters, such as the retardation time are only valid at the temperature at which the data was obtained.

Polymers, such as nylon and epoxy adhesives are sensitive to moisture and will absorb significant amounts of moisture under relatively benign environments (i.e., standard laboratory conditions). The presence of water in epoxy adhesives results in plasticisation (essentially softening) of the polymeric material (i.e., increase in molecular mobility - a reduction in t_0) [79]. It is recommended that specimens be kept in a controlled environment, such as a dessicator) to ensure comparable moisture content. It may be necessary to dry the specimens to constant weight before testing (see [37]).

Cure and welding parameters

Degree of cure: The degree of cure of an adhesive will affect its creep performance. For poorly cured systems, where the cross-linking process is incomplete, T_g will be suppressed, resulting in an increase in molecular mobility and higher creep rates. It is important to ensure the adhesive or resin is completely cured. Thermal analysis should be carried out to ensure the state of cure meets specification. DSC and DMA can provide useful information relating to the final state of cure [58-60].

Welding processing parameters: The quality of a weld in thermoplastic polymers (e.g., polypropylene) has a significant affect on the weld strength and the long-term creep rupture behaviour of the welded system. In the case of hot plate welding, the hot plate temperature, fusion pressure, hot soak time and cooling time are key parameters in ensuring the quality of the weld meets specification. Similar parameters operate for other forms of welding. Hot plate temperature seems to have a dominant influence on creep rupture, although the other processing parameters are important and must be taken into account (see Figure 16).

Procedure for modelling creep in polymers

Creep tests are used to determine how material properties depend upon time under a constant applied load. This section gives an outline of the stages required to determine the parameters in creep functions commonly used to model creep in polymers.

Functions are first presented that relate strain to stress under a uniaxial tensile stress. These are then extended to cover non-linear behaviour and brief reference is given to how a model can be generalised to relate stress and strain components under a multiaxial stress state (see – [78-80]).

Creep tests are carried out under uniaxial tension and over a range of stress levels. The tensile strain $\varepsilon(t)$ is measured at suitable time intervals and tensile creep compliance values $D(t)$ at each time are derived by dividing the strain at that time by the constant applied stress σ_0 as follows:

$$D(t) = \frac{\varepsilon(t)}{\sigma_0} \quad (9)$$

The selected stress range depends on the load bearing capabilities of the polymer but, for guidance, the maximum stress could be $0.5 \times$ the peak stress measured in a short-time tensile test. For each stress, creep compliance curves are determined as a function of creep time.

Best fits are obtained to these data using the equation:

$$D(t) = D_0 + \Delta D \left[1 - \exp - \left(\frac{t}{t_0} \right)^n \right] \quad (10)$$

if the polymer is semi-crystalline, or

$$D(t) = D_0 \exp \left(\frac{t}{\tau} \right)^m \quad (11)$$

if the polymer is amorphous and the glass-to-rubber relaxation mechanism is responsible for the creep deformation.

Here D_0 is the tensile compliance at zero time, ΔD is the magnitude of the compliance change at long times when the relaxation process is complete, and t_0 , τ , n and m are parameters that define the time range over which the creep relaxation process takes place. A value for the parameter D_0 is readily estimated from results at short creep time. Values for the other parameters can be obtained by manual optimisation to achieve satisfactory fits.

At elevated stresses, compliance curves $D(t)$ are observed to shift to shorter creep times giving rise to non-linear behaviour. It is generally possible to obtain good fits to experimental data at different stress levels by keeping all parameters constant except t_0 or τ so that this parameter is the only parameter that is dependent on stress.

The dependence of t_0 on stress is then described using the expression:

$$t_0 = A \exp - (\alpha \sigma_0) \quad (12)$$

for crystalline polymers, and

$$D\tau = B \exp - (\beta \sigma_0^2) \quad (13)$$

For amorphous polymers.

Here, A , B , α and β are material parameters that determine the sensitivity of the time parameter to the stress magnitude.

Creep tests under uniaxial compression can also be modelled by the procedure used for tensile behaviour, but the time parameters t_0 and τ are not given by the above equations. This suggests that the stress state as well as the stress level determine the magnitude of the time parameter. The above equations should therefore be replaced by:

$$t_0 = A \exp - (\alpha \bar{\sigma}) \quad (14)$$

or

$$\tau = B \exp - (\beta \bar{\sigma}^2) \quad (15)$$

Here, $\bar{\sigma}$ is an effective stress that is defined by the shear and hydrostatic components of the creep stress as follows:

$$\bar{\sigma} = \mu \sigma_e + 3(1 - \mu) \sigma_m \quad (16)$$

σ_e and σ_m are the deviatoric and hydrostatic components of stress and μ is a parameter that determines the sensitivity of the creep time parameter to the hydrostatic component of stress. A value for the parameter μ can thus be derived from a value for the time parameter t_0 or τ determined from a compressive creep test.

The introduction of an effective stress that determines the magnitude of the time parameter allows the creep function presented above for describing creep under a uniaxial stress to be generalised to relate stress and strain components under a multiaxial stress. This is necessary for the model to be implemented in a finite element system, thus enabling stress analyses to be carried out for long-term loading applications.

Chapter 5

Handling, storage, and documentation

- Handling and storage of materials and chemicals
- Handling and storage of adhesives, resins and prepreg
- Handling and storage of adherends
- Handling, storage, and disposal of chemicals
- Documentation (record keeping)

Handling and storage of materials and chemicals

A number of issues relating to the handling and storage of materials and chemicals 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 polymeric materials, such as resins and 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, resins and prepreg

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 (e.g., adhesives and resins) to temperatures and humidity's above those recommended by the manufacturer will reduce the cohesive and adhesive strength of the material when cured [3]. The overall effect is to reduce the adhesive performance and induce premature failure of the polymeric system.

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

Ideally, temperature and humidity in the storage area should be tightly controlled and monitored (i.e., temperature recorders), although this may not always be physically or economically feasible. Standard laboratory conditions are typically $23 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ relative humidity (RH).

Refrigerators (or freezers) should be armed with a temperature alarm and the temperature should be monitored continuously to ensure that the temperature remains below the upper temperature limit specified by the adhesive manufacturer. The maintenance of the refrigerator units should allow for regular manual or automatic defrosting.

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

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

Resins, adhesives and prepreps 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

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.

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

Handling, storage, and disposal of chemicals

In all cases involving solvents, primers, acid and alkaline solutions, extreme care and good laboratory practice should be followed at all times in storing, handling and disposing these materials (recommended further reading [20]). Personnel need to be trained in handling and using these materials, and should wear appropriate protective clothing as the chemicals can be harmful if they come contact with the skin or eyes. Ventilation and spill containment are particularly important safety considerations. Chemicals treating solutions should be prepared and stored in containers of glass, ceramic, or chemical resistant plastic. Checks should be made to ensure that the containing vessel is resistant to chemical attack from would be contents (e.g., hydrofluoric acid attacks glass). Stirring rods should be made of the same materials. Contact of the chemical solution with metals during preparation and storage should be avoided.

Continuous monitoring and quality control are required for all chemicals employed in chemical conditioning and surface treatments, and all containers with chemical agents should be clearly identified with labels. Labelling should be protected from being damaged through chemical contact or handling. Chemical solutions need to be changed regularly to prevent contamination and ensure repeatable concentration [20]. Quality assurance tests are recommended to check concentrations, and levels of contamination (e.g., opacity for checking particulate content, colour and pH for acidic and alkaline solutions).

Warning: It is important to note that many chemicals, such as acids, alkalis and solvents are toxic and present a risk to both human health and the environment, and that all safety requirements need to be met when handling, storing, and disposing of these materials.

Documentation (record keeping)

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

It is recommended that an accurate check as to the quality of the polymeric material 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 [20]). The colour, viscosity, shelf life, etc. should be verified. It may be necessary to carry out acceptance tests in-house to ensure mechanical properties are in accordance with the material specification. Acceptance tests should be directed towards assuring that the materials are identical from batch to batch. Accelerated environmental tests (i.e., high temperature and humidity) may also need to be carried out to confirm that the durability performance of the material meets the manufacturer specifications. ISO, BSI, and ASTM list various test methods for quality assurance of adhesives (see Chapter 9).

Standards

Standards

ISO Standards

Plastics

Mechanical

- ISO 178 Plastics - Determination of Flexural Properties.
- ISO 179 Plastics - Determination of Charpy Impact Properties - Part 1: Non-Instrumented Impact Test.
- ISO 179-2 Plastics - Determination of Charpy Impact Properties - Part 2: Instrumented Impact Test.
- ISO 527-1 Plastics - Determination of Tensile Properties - Part 1: General Principles.
- ISO 527-2 Plastics - Determination of Tensile Properties - Part 2: Test Conditions for Moulding and Extrusion Plastics.
- ISO 604 Plastics – Determination of Compressive Properties.
- ISO 10350-1 Plastics - Acquisition and Presentation of Comparable Single-Point Data - Part 1: Moulding Materials.
- ISO 11403-1 Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 1: Mechanical Properties.

Mechanical (Creep)

- ISO 899-1 Plastics - Determination of Creep Behaviour - Part 1: Tensile Creep.
- ISO 899-2 Plastics - Determination of Creep Behaviour - Part 2: Flexural Creep by Three-Point Loading.

Thermal properties

- ISO 75-1 Plastics - Determination of Temperature of Deflection Under Load - Part 1: General Test Method.
- ISO 75-2 Plastics — Determination of Temperature of Deflection Under Load — Part 2: Plastics and Ebonite.
- ISO 11403-2 Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 2: Thermal and Processing Properties.

Environmental conditioning

- ISO 62 Plastics – Determination of Water Absorption.

ISO 175	Plastics - Determining the Effect of Liquid Chemical Including Water.
ISO 291	Plastics – Standard Atmospheres for Conditioning and Testing.
ISO 305	Determination of Thermal Stability of Polyvinyl Chloride Related Chlorine Containing Polymers, and their Compounds - Discoloration Method.
ISO 483	Plastics - Small Enclosures for Conditioning and Testing Using Aqueous Solutions to Maintain Relative Humidity at Constant Value.
ISO 554	Standard Atmospheres for Conditioning and/or Testing - Specification.
ISO 1137	Plastics: Determination of Behaviour in a Ventilated Tubular Oven.
ISO 1599	Cellulose Acetate- Determination of Viscosity.
ISO 2578	Plastics: Determination of Time/Temperature Limits after Exposure to Prolonged Action of Heat.
ISO 3205	Preferred Test Temperatures.
ISO 3671	Determination of Volatile Matter of Amino-Plastics.
ISO 4611	Plastics - Determination of the Effect of Exposure to Damp Heat, Water Spray and Salt.
ISO 11403-3	Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 3: Environmental Influences on Properties.

Environmental stress cracking (ESC)

ISO 4599	Plastics: Determination of Time/Temperature Limits after Exposure to Prolonged Action of Heat Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Bent Strip Method.
ISO 4600	Plastics - Determination of Environmental Stress Cracking (ESC) - Ball and Pin.
ISO 6252	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Constant Tensile Stress Method.
ISO 16770	Plastics - Determination of Environmental Stress Cracking (ESC) of Polyethylene - Full-Notch Creep Test (FNCT).
ISO 22088-5	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Part 5: Constant Tensile Deformation Method.
ISO 22088-6	Plastics - Determination of Resistance to Environmental Stress Cracking (ESC) - Part 6: Slow Strain Rate Method.

Welded joints

- ISO 13953 Polyethylene (PE) Pipes and Fittings - Determination of the Tensile Strength and Failure Mode of Test Pieces from a Butt-Fused Joint.
- ISO 13954 Plastics Pipes and Fittings - Peel Decohesion Test for Polyethylene (PE) Electrofusion Assemblies of Nominal Outside Diameter Greater Than or Equal to

Composites

- ISO 75-3 Plastics - Determination of Temperature of Deflection under Load. Part 3: High-Strength Thermosetting Laminates and Long-Fibre-Reinforced plastics.
- BS EN 2378 Fibre Reinforced Plastics - Determination of Water Absorption by Immersion in Demineralised Water.
- BS EN 2489 Fibre Reinforced Plastics - Determination of the Action of Liquid Chemicals.
- BS EN 2823 Fibre Reinforced Plastics - Determination of the Effect of Exposure to Humid Atmosphere on Physical and Mechanical Characteristics.
- ISO 10350-2 Plastics — Acquisition and Presentation of Comparable Single-Point Data — Part 2: Long-Fibre-Reinforced Plastics.
- ISO 13003 Fibre-Reinforced Plastics - Determination of Fatigue Properties under Cyclic Loading Conditions.
- ISO 21746 Composites and Metal Assemblies - Galvanic Corrosion Tests of Carbon Fibre Reinforced Plastics (CFRPs) Related Bonded or Fastened Structures in Artificial Atmospheres - Salt Spray Tests.
- ISO 22836 Fibre-Reinforced Composites - Method for Accelerated Moisture Absorption and Supersaturated Conditioning by Moisture Using Sealed Pressure Vessel.

Note: The standards listed in this section refer specifically to fibre-reinforced composites. More general standards covering both plastics and composites are given in the section on ISO plastic related standards.

Adhesives

Mechanical

- ISO 4587 Adhesives - Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies.
- ISO 6922 Adhesives – Determination of Tensile Strength of Butt Joints.

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|-------------|--|
| ISO 9664 | Adhesives - Test Methods for Fatigue Properties of Structural Adhesives in Tensile Shear. |
| ISO 11003-1 | Adhesives - Determination of Shear Behaviour of Structural Adhesives — Part 1: Torsion Test Method Using Butt-Bonded Hollow Cylinders. |
| ISO 11003-2 | Adhesives - Determination of Shear Behaviour of Structural Adhesives — Part 2: Tensile Test Method Using Thick Adherends. |

Environmental testing

- | | |
|-----------|---|
| ISO 9142 | Adhesives - Guide to the Selection of Standard Laboratory Ageing Conditions for Testing Bonded Joints. |
| ISO 10354 | Adhesives - Characterisation of Durability of Structural Adhesive Assemblies - Wedge Rupture Test. |
| ISO 10363 | Hot Melt adhesives - Determination of Thermal Stability. |
| ISO 14615 | Adhesives - Durability of Structural Adhesive Joints - Exposure to Humidity and Temperature under Load. |

BSI and EN Standards

Welded joints

- | | |
|------------|---|
| EN 12814-1 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 1: Bend Test. |
| EN 12814-2 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 2: Tensile Test. |
| EN 12814-3 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 3: Tensile Creep Test. |
| EN 12814-4 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 4: Peel Test. |
| EN 12814-5 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 5: Macroscopic Examination. |
| EN 12814-6 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 6: Low Temperature Tensile Test. |
| EN 12814-7 | Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 7: Tensile Test with Waisted Test Specimens. |

- EN 12814-8 Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 8: Requirements.
- EN 13100-1 Non destructive Testing of Welded Joints of Thermoplastics Semi-Finished Products - Part 1: Visual Examination.

Adhesives

- BS EN 1465 Adhesives – Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies.
- BS 5350-A1 Methods of Test for Adhesives - Part A1: Adherend Preparation.
- BS 7079 General Introduction to Standards for Preparation of Steel Substrates Before Application of Paints and Related Products.
- BS EN 2243-5 Non-Metallic Materials Structural Adhesives Test Method Part 5: Ageing Tests.
- BS EN 13887 Structural Adhesives. Guidelines for Surface Preparation of Metal and Plastics Prior to Adhesive Bonding.
- BS EN 26922 Adhesives - Determination of Tensile Strength of Butt Joints.

ASTM Standards

Plastics

Mechanical

- ASTM D695 Standard Test Method for Compressive Properties of Rigid Plastics.
- ASTM D2990 Standard Test Methods for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics.

Environmental conditioning and testing

- ASTM D543 Standard Practices for Evaluating the resistance of Plastics for Chemical Reagents.
- ASTM D570 Standard Test Method for Water Absorption of Plastics.
- ASTM D618 Standard Practice for Conditioning Plastics for Testing.

Environmental stress cracking (ESC)

- ASTM D1693 Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics.
- ASTM F1248 Standard Test Method for Determination of Environmental Stress Crack Resistance (ESCR) of Polyethylene Pipe.

Adhesives

Mechanical

- ASTM D897 Standard Test Method for Tensile Properties of Adhesive Bonds.
- ASTM D905 Strength Properties of Adhesive Bonds in Shear by Compression Loading.
- ASTM D1002 Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal).
- ASTM D1780 Standard Practice for Conducting Creep Tests of Metal-to-Metal Adhesives.
- ASTM D2095 Standard Test Method For Tensile Strength Of Adhesives By Means Of Bar And Rod Specimens.
- ASTM D2293 Standard Test Method for Creep Properties of Adhesives in Shear By Compression Loading (Metal-to-Metal).
- ASTMD 2294 Standard Test Method for Creep Properties of Adhesives in Shear By Tension Loading (Metal-to-Metal).
- ASTM D3163 Standard Test Method for Determining Strength of Adhesively Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading.
- ASTM D3166 Standard Test Method for Fatigue Properties of Adhesives in Shear by Tension Loading (Metal/Metal).
- ASTM D3528 Standard Test Method for Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading.
- ASTM D3983 Standard Test Method for Measuring Strength and Shear Modulus of Nonrigid Adhesives by the Thick-Adherend Tensile-Lap Specimen.
- ASTM D4896 Standard Guide for Use of Adhesive-Bonded Single Lap-Joint Specimen Test Results.
- ASTM D5656 Standard Test Method for Thick-Adherend Metal Lap-Shear Joints for Determination of the Stress-Strain Behavior Of Adhesives in Shear by Tension Loading.

Environmental conditioning and testing

- ASTM D896 Standard Practice for Resistance of Adhesive Bonds to Chemical Reagents.
- ASTM D1151 Standard Practice for Effect of Moisture and Temperature on Adhesive Bonds.
- ASTM D1183 Standard Practices for Resistance of Adhesives to Cyclic Laboratory Aging Conditions.

- ASTM D1828 Standard Practice for Atmospheric Exposure of Adhesive Joints Stressed in Peel.
- ASTM D2295 Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading at Elevated Temperatures (Metal-to-Metal).
- ASTM D2918 Standard Test Method for Durability Assessment of Adhesive Joints Stressed in Peel.
- ASTM D2919 Standard Test Method for Determining Durability of Adhesive Joints Stressed in Shear by Tension Loading.
- ASTM D3434 Standard Test Method for Multiple-Cycle Accelerated Aging Test (Automatic Boil Test) for Exterior Wet Use Wood Adhesives.
- ASTM D3632 Standard Test Method for Accelerated Aging of Adhesive Joints by the Oxygen-Pressure Method.
- ASTM D3762 Standard Test Method for Adhesive-Bonded Surface Durability of Aluminium (Wedge Test).
- ASTM D4498 Standard Test Method for Heat-Fail Temperature in Shear of Hot Melt Adhesives.

Surface treatment

- ASTM D2093 Standard Practice for Preparation of Surfaces of Plastics Prior to Adhesive Bonding.
- ASTM D2651 Standard Guide for Preparation of Metal Surfaces for Adhesive Bonding.

Composites

- ASTM C394 Standard Test Method for Shear Fatigue of Sandwich Core Materials.
- ASTM C480 Standard Test Method for Flexure Creep of Sandwich Constructions.
- ASTM D896 Standard Practice for Resistance of Adhesive Bonds to Chemical Reagents.
- ASTM D3479 Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite Materials.
- ASTM D5229 Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials.
- ASTM D5379 Standard Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method.
- ASTM D6115 Standard Test Method for Mode I Fatigue Delamination Growth Onset of Unidirectional Fiber-Reinforced Polymer Matrix Composites.

ASTM D7792 Standard Practice for Freeze/Thaw Conditioning of Pultruded Fiber Reinforced Polymer (FRP) Composites Used in Structural Designs.

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