

Review of Test Methods and Standards for Assessing Long-Term Performance of Polymer Matrix Composites

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ABSTRACT

This report provides a critical appraisal of test methods and related standards suitable for assessing the long-term properties, residual life and residual strength of polymer matrix composites subjected to aggressive agent and/or applied static or fatigue loads. The report covers material property degradation under these conditions. Particular attention has been given to examining methods for accelerated ageing, the use of thermal analysis techniques and aspects requiring additional care when testing under environmental and cyclic fatigue loading conditions. A number of methods identified within this review will be evaluated within the subsequent experimental programme in collaboration with a parallel task aimed at developing and validating models for predicting degradation and residual properties. Test methods developed are required to be suitable for determining the effect of temperature and the level of degrading agent on the degree of degradation, identifying any synergistic or superimposed effects between environmental and loading parameters.

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1. INTRODUCTION

There is a tendency to "overdesign" composite structures, particularly those structures that are used under fatigue loads and/or exposed for extended periods to hostile environments (ie marine, hot/wet, weathering and chemical). Conservative design practices result in heavier and more costly components. Increased acceptance of composites for use in engineering structures (eg pressure vessels, tanks and piping) exposed to combined stress, thermal and chemical environments hinges on the availability of validated data demonstrating the capability of the composite materials to maintain structural integrity over periods of 50 to 100 years, or preferably longer. Design allowables based on test data obtained under ambient conditions are no substitute for reliable data representative of actual service conditions. Equally, when using accelerated test procedures, there is a need to demonstrate that the failure mechanisms, etc. have not changed by the accelerating test procedure. The development of standard test methods and predictive methodologies for evaluating long-term properties and residual life/strength, when subjected to combinations of environmental attack and applied static or fatigue loads can be expected to result in more efficient use of composite materials, lower structural weight, and lower material and manufacturing costs.

This report provides a critical appraisal of life assessment methods currently available for characterising material property degradation of PMCs. The report covers:

- environmental degradation (ie elevated temperature, moisture, chemicals)
- static and cyclic fatigue testing
- environmental conditioning
- accelerated ageing
- thermal analysis techniques

and aspects associated with mechanical testing under environmental and cyclic fatigue loading conditions requiring additional care.

The results of the review will contribute to the subsequent experimental programme for the DTI funded project "Composite Performance and Design - Life Assessment and Prediction (CPD2)" directed towards the development and validation of test methods and procedures that can be used for characterising PMCs exposed to combined aggressive environments and applied loads (static and fatigue).

The report is divided into eight sections. Following the Introduction in Section 1, Sections 2 and 3 cover degradation agents and resultant material response, and concepts of life assessment for long-term testing. Environmental testing including moisture and chemical exposure, weathering, hot/wet conditioning and monitoring moisture/water content is covered in Section 4. Accelerated test methods are covered in Section 5 with two contrasting methods being considered. The first method employs severe test conditions to induce an accelerated response, whereas the second method uses fibre bundle specimens which are conditioned under less severe test conditions, but because of the relatively small size of the specimens the process of ageing is considerably reduced compared with similar tests conducted on standard coupon specimens. Problems relating to durability testing and measurement are considered in Section 6. This section considers testing under environmental and/or cyclic test conditions. Section 7 examines differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA) and heat distortion temperature (HDT). Concluding remarks are given in Section 8. Appendix I provides a listing of relevant durability standards, a description of environmental regimes and standards relating to design constraints and endurance calculations. **Throughout this report, statements of particular importance or relevance are highlighted in bold type.**

2. DEGRADATION AGENTS AND MATERIAL RESPONSE

Irreversible property changes in PMCs can be induced by any number of degradation agents (factors) acting alone or collectively [1-6]:

- Thermal (ie heat ageing or sub-zero exposure)
- Humidity (inc. hot/wet) exposure
- Complete immersion in water at ambient and elevated temperatures
- Freeze/thaw and dry/wet cyclic conditions
- Continuous or intermittent saltwater immersion or spray
- Acid and alkali solutions
- Solvents (inc. paint strippers and cleaning agents)
- Diesel and engine fuels
- Weathering (including rain, sand erosion, ultraviolet radiation)
- Microorganisms (eg fungi)
- Mechanical stress (ie static and fatigue)
- Electrical stress

In many applications, composite structures will be exposed to a combination of two or more factors, often resulting in complex synergistic degradation of the material. The relative importance of each agent will depend on the agents present and their levels. In addition to provoking chemical reactions, absorbed chemicals induce important physical effects (ie dimensional changes, matrix plasticisation, lowering of the glass-transition temperature (T_g), matrix crystallisation in thermoplastics and leaching of soluble constituents). In the absence of environmental stress cracking (ESC), the amount and type of chemical absorbed in a given time can be a useful guide as to the probable extent of physical damage induced [6]. A number of degradation factors relating to in-service environmental conditioning are discussed in this section.

2.1 DEGRADATION AT NON-AMBIENT TEMPERATURES

Prolonged, or even short term, exposure to elevated temperatures will often produce irreversible chemical and physical changes within PMCs. As the temperature increases, the interfacial and matrix strengths decrease. Testing at non-ambient temperature should be carried out according to the International Standard ISO 3025, which provides a list of recommended test temperatures applicable to all classes of material (see also NPL Measurement Note CMMT(MN) 18).

Oxidation is the primary degradation process at elevated temperatures with the rate of degradation increasing with the amount of oxygen present. The primary path for oxygen diffusion is through the polymer and along the fibre-matrix interface, which can be relatively rapid at elevated temperatures. Oxidation can result in loss of strength and discolouration. High temperature resistant polymers (eg polyimides) were developed to resist thermal oxidation. DSC can be used to ascertain the extent and rate of material oxidation of the fibre, matrix and fibre-matrix interface.

Low temperature thermal cycling can induce micro-cracking within the polymer matrix and at the fibre-matrix interface. The presence of these cracks will increase moisture uptake, which can severely affect the performance of PMCs. Exposure to sub-zero temperatures as low as -55°C has minimal effect on the stiffness and strength of unidirectional laminates regardless of moisture content. Matrix dominated properties may be compromised.

2.2 EFFECT OF MOISTURE/WATER

PMCs will absorb relatively small, but potentially damaging amounts of moisture from the surrounding environment that relates directly with the degree of degradation that occurs. The moisture absorption kinetics of polymer systems differ widely and also change with physical ageing. Although the process of moisture absorption and desorption from the surface layers will occur immediately on contact with the environment, moisture flow into or out of the interior of the composite will be slow. It may take weeks or months of exposure to humid environments before a significant amount of water is absorbed by the composite. Moisture tends to enter at bolt holes and cut edges, regions in which bundles of fibres are exposed. The amount of moisture absorbed by a material is dependent upon specimen thickness and exposure time. Test methods for monitoring absorbed water include ISO 62, prEN 2378, prEN 2823 and ASTM D 5229. In some cases, water is considered alongside other chemicals.

Absorbed water may produce a number of adverse affects: (i) dimensional changes (swelling); (ii) reduction in T_g of the matrix; and (iii) reduction of the mechanical properties of the matrix and fibre-matrix interface. Composite properties dominated by matrix characteristics (eg transverse tension, longitudinal compression and shear) are particularly susceptible. A reduction in T_g will effectively lower the maximum service or operating temperature of the composite. Moisture induced effects are often reversible on drying the composite. In addition, entrapped moisture will expand and contract with changes in temperature, resulting in internal stresses and microcrack formation, promoting further moisture ingress.

2.3 CHEMICAL AND PHYSICAL AGENTS

A full list of recognised chemical agents and test methods for assessing the effects of chemical exposure are given in ISO 175 and prEN 2489. A number of these agents and situations most relevant to composites are briefly discussed below.

Solvents such as the paint stripper (eg methylene chloride) will soften and dissolve epoxy resins. Hydraulic fluid has a similar effect, but takes longer. It is recommended that solvents such as methyl-ethyl-ketone (MEK) and acetone should not be left standing on either thermoset or thermoplastic composite surfaces.

Paint strippers combined with **abrasion techniques** can cause irreversible material damage. Aircraft may be repainted between 10 and 15 times during a service life of 30 to 40 years. Old paint must be removed before recoating; otherwise it will add to the weight of the aircraft. A new method of chemical paint stripping has been adopted in the UK for metal aircraft. A permanent primer coating is applied to the surface. An intercoat is then applied to the primer, followed by the top coat. The upper two layers can be removed by benzyl alcohol. The intention is to extend the use of the method to composite structures.

Acid and alkaline agents can degrade fibre, resin and the fibre-matrix interface. The degree of degradation is dependent on concentration levels and temperature. In general, epoxy resins are more resistant than polyesters whilst high performance thermoplastics (eg polyether-ether-ketone) are virtually resistant to ordinary chemical attack. E-glass fibres are known to suffer stress corrosion when exposed to acidic solutions.

Hydrolysis by hot water, aqueous alkali and acids is a serious problem for polyesters and polyamides. Strong alkalis are particularly harmful to polyesters, which can be completely destroyed. This same group of chemical reagents can also induce ion exchange reactions in glass fibres, leading to flaw initiation and propagation under mechanical load.

A majority of matrix resins and carbon fibres are susceptible to **oxidation** by any one of numerous oxidising agents (eg hydrogen peroxide and certain bleaches such as sodium hypochlorite, nitric or sulphuric acid). Oxidation effects at elevated temperatures is covered in Section 2.1.

Osmosis causing surface blistering, although primarily cosmetic, is a very common problem that occurs in laminated structures that have been immersed for long uninterrupted periods in aqueous solutions (eg marine applications). The phenomenon affects laminates contaminated by water-soluble impurities. Osmosis was first recognised on GRP boats, and to a lesser extent in the water tanks, swimming pools, but there is now knowledge in the industry that allows the problem to be avoided providing the guidelines are followed..

Intercalation is an unusual process, affecting high modulus carbon fibres, caused by chemical molecules (eg halogens and many inorganic salts such as ferric chloride) inserting themselves between the graphite layer planes. The result is to induce large changes in fibre dimensions.

Ultraviolet radiation is known to degrade aramid fibres, although only the outer layer is affected. Epoxy resins are slightly sensitive to ultraviolet radiation; depending on the chemical formulation (ISO 4582, ISO 4892 and ASTM D 4329) - see also Section 2.4.

Exposure to **salt spray** or **salt water** will degrade glass-fibre reinforced systems with the degree of protection depending on the permeability of the resin to diffusion of chloride ions and the ability of the matrix and interface to resist premature cracking, which would allow the environment to come in direct contact with the fibres resulting in their corrosion.

Often other constituents, such as fillers, fire retardants and curing agents are more reactive than the resin or fibre reinforcement. Formulators and users, although usually aware of the potential durability problems associated with these additives, often overlook minor constituents such as catalysts, hardeners, pigments and processing aids.

2.4 LONG-TERM STATIC AND CYCLIC LOADS

For matrix dominated or discontinuously reinforced systems visco-elastic creep will occur under long term static or creep loads. In addition, for glass-fibre systems there is a well established stress-corrosion mechanism, whereby moisture aids the propagation of surface cracks leading to ultimate failure. Most testing under these conditions is for sections of pipes, as detailed in Section 5.3.

Fatigue damage is due to irreversible processes (eg microcracking, fibre debonding, fibre fractures and delaminations) which occur when a material is subjected to cyclic loading. The extent of fatigue damage and its importance is dependent primarily on the stress level at which irreversible damage occurs relative to the stress for complete failure (ie ultimate static strength). These fatigue processes lead to general material degradation (ie reduced strength and stiffness) rather than producing a discrete crack as in metals. The actual type of damage and accumulation depends critically on the type of fibre, matrix, fibre-matrix interface, laminate lay-up, specimen geometry and the level, type, frequency and direction of the applied load. In addition, fatigue performance will depend on the test temperature and/or the applied environment (eg water and acid). Unidirectional glass fibre-reinforced laminates exhibit classical fatigue behaviour (ie short fatigue life at high stress amplitudes with life expectancy increasing as the stress amplitude is reduced), whereas unidirectional carbon-fibre laminates appear relatively unaffected by fatigue. Fatigue damage in these materials only occurs at stress levels close to ultimate fracture and so their fatigue resistance is excellent.

These materials display a relatively flat S-N curve (ie stress-number of cycles) curve. Matrix dominated properties are particularly susceptible to fatigue induced damage. Materials that undergo either plastic or visco-elastic deformation at relatively low stresses will exhibit poor resistance to fatigue. Additionally, autogenous (self-generated) heating will occur due to the visco-elastic nature of the material (and poor heat conduction for GRPs) during cyclic loading, which can lead to increases in temperature. The effect is enhanced towards the end of the test when the microdamage causes a large change in the material damping factor and hence the temperature rises. Frequencies above 10Hz can result in a substantial increase in the mean surface temperature from a few degrees to 80°C, or more [7].

3. TEST DATA MANIPULATION FOR LIFE ASSESSMENT

This section examines general principles used to assess the degree and rate of material degradation due to prolonged exposure to combinations of stress, elevated temperature and chemical (eg moisture) attack. The philosophical approach described is universal, applying equally to the determination of residual strength following prolonged environmental exposure and cyclic fatigue. Alternative approaches for assessing long-term degradation are covered in NPL Report CMMT(A) 95 [8].

A concept often employed to assess thermal degradation is to plot material property data against time (Figure 2) for one temperature (see ISO 2578). The data can often be represented by an empirical relation of the form shown below [8]:

$$\log P = P_1 + B_1 t \quad (1)$$

where P is the material property (eg shear strength), P_1 is the material property at zero ageing time, B_1 is the degradation rate and t is the ageing time. The initial temperature is chosen to obtain a duration of one-quarter of the end-point chosen. Similar data are generated at different temperatures. The time required for the strength to decrease to a given percentage of the initial value (eg 50% loss (ie half-life)) is either read directly from the plots of relative property value (%) against ageing time for the various exposure temperatures or calculated from fitted equations, such as Equation (1).

The next step is to plot the log of these times against the exposure temperature to give a straight line from which the **temperature index (TI)** (eg 50% loss after 20,000 hours) can be obtained. A typical plot of these values as a function of ageing temperature will result in a straight line (Figure 3). The plot of half-life against T can be represented by the following equation:

$$\log t_{1/2} = A_2 + B_2 T \quad (2)$$

where $t_{1/2}$ is the half-life property, A_2 and B_2 are material constants and T is the ageing temperature (K). The service life (or half-life at service temperature) can be estimated by extrapolation from the plot of $\log t_{1/2}$ versus T or from the fitted Equation (2).

This approach can also be used for determining the effects of moisture and other chemicals on material performance at both ambient and non-ambient temperatures, and can be applied to both static and cyclic fatigue data. The user is free to select the property most relevant to the material and/or intended application. The test method requires an extensive number of tests to be conducted. Although failure criteria of 20,000 hours and a 50% reduction in the measured property are recommended, other criteria can be chosen. For example, the aerospace standard for heat ageing prEN 4238 allows any choice of time and degradation level to be chosen making comparisons extremely difficult.

A similar staircase approach is used in ISO 11248 for thermosetting moulding materials. In this standard, the test temperature divisions are set at 25°C with the first test temperature set at the next lowest multiple of 25°C below the T_g (obtained from DMA measurements or from the heat deflection temperature). The time of exposure is set to 24 hours and the percentage loss at 50%. This standard, developed for electrical applications, recommends that the assessment should be carried out for one mechanical property and one other property, such as dielectric strength.

4. ENVIRONMENTAL AND MECHANICAL STRESS TESTING

A major concern has been that there are many similar, but different test methods available (eg ASTM, BSI, and ISO) and as a result users of plastics and composites find that available data cannot always be readily used to compare the properties of similar materials obtained using the different methods. Although there is a trend to harmonisation at the ISO level, standards can still include more than one detailed method (eg size of notch and specimen direction in ISO 179 for Charpy impact).

An international draft ISO/F-DIS 10,350, currently being prepared, has identified specific methods and test conditions to be used for the acquisition and presentation of data in order that valid comparisons can be made between different materials (ie Part 1 - Plastics, Part 2 - Composites). This standard is concerned with tests employed to present "single-point" (eg Young's modulus) data on a limited range of properties commonly specified in manufacturers data sheets and used for the preliminary selection of materials. A complementary international standard (ISO 11,403) is concerned with the standardised acquisition and presentation of "multi-point" data for demonstrating the variation of mechanical properties with time and temperature and exposure to natural and chemical environments. This standard also covers thermal and processing properties.

4.1 MOISTURE/WATER EXPOSURE

The approach usually adopted for simulating in-service conditions is to define a constant relative humidity environment that will produce a moisture level equivalent to that experienced in-service by the composite structure [9]. The temperature and humidity variability of a natural environment is a major factor in determining both the final moisture equilibrium level in the materials bulk and the distribution of moisture in the outer surface layers.

Moisture diffusivity and moisture equilibrium are commonly determined using gravimetric test methods. ASTM D 5229 provides procedures for the determination of moisture absorption or desorption properties in the through-thickness (T-T) direction, conditioning to equilibrium in standard and non-laboratory environments, and to moisture free states. This method can be applied to vapour exposure and liquid immersion. Single-phase Fickian behaviour is assumed when determining the T-T moisture diffusivity of the material. PMCs tested below their T_g will normally behave in this manner, although two-phase toughened epoxy systems may require a multi-phase moisture absorption model. To determine the actual moisture diffusion coefficient, the measured T-T diffusion coefficient needs to be corrected to account for moisture ingress through the specimen edges. The correction factor is extremely complicated for laminated composites because the diffusivity in the x, y and z directions can be expected to be different. The analysis assumes that the T-T moisture absorption properties are constant throughout the laminate thickness.

Specimens are exposed to a steady-state environment (ie constant temperature and constant moisture exposure level) until the material reaches effective moisture equilibrium. All surfaces of the specimen are usually in contact with the test environment. Prior to environmental conditioning, specimens are dried in a vacuum oven until a near equilibrium

weight is obtained to ensure a zero datum level. The surface moisture should also be removed before the specimen is weighed. This technique enables the moisture equilibrium and diffusion coefficient of the material to be determined. From an accuracy perspective, the specimen should be of substantial dimensions (75 mm square and 2 mm thick [9]). The test materials should be void free; as the presence of voids will be a source of error.

In the case of aramid composites, determination of diffusion coefficients can be complicated by the hygroscopic nature of the aramid fibres. For these materials, it is advisable to measure the moisture uptake of both the fibre and resin separately in order to isolate constituent contributions. After long exposure times, the moisture content may deviate above that predicted by the Fickian analysis, exceeding the saturation level predicted by Fickian theory. This non-Fickian behaviour could possibly be due to defect growth.

4.2 WEATHERING

Weather resistance data is usually obtained through actual outdoor exposure of test specimens or from tests carried out in special environmental (weathering) cabinets. Outdoor exposure tests often consist of specimens attached to a test surface at a suitable angle to maximise exposure to sunlight. Relatively complex arrangements consisting of mirrors and solar tracking equipment to accelerate the effective exposure are also in use. Accelerated weathering cabinets are also available. These cabinets generally employ high intensity radiation sources (eg carbon arc, xenon, metal halide or fluorescent lamps).

A number of national (ASTM D 5272) and international (ISO 827) standards cover natural and accelerated weathering. These standards consider the ageing behaviour of polymer materials due to solar radiation. Some of these standards include the additional effect of temperature and/or moisture. A number of radiation sources and test regimes are recommended. To assess accurately the ageing behaviour in polymeric materials, the weathering device is required to closely simulate the in-service spectral distribution of solar radiation in the ultraviolet and visible spectral range experienced by the material [6]. This is not always possible, thus the use of partial range of the global spectral distribution is permissible, provided this spectral component is the major contributor to photochemical degradation of the material in question. Infrared radiation does not contribute directly to photochemical degradation, however surface heating due to the absorption of the visible infrared radiation has an indirect influence on ageing. Samples are exposed to a mixture of direct and scattered radiation, with the ratios of the two types of radiation depending on the design of the accelerated weathering device.

4.3 CHEMICAL EXPOSURE

British standard BS 4994 specifies requirements for the design, materials selection, construction, inspection, testing and erection of vessels and tanks in reinforced plastics. The standard covers a wide range of materials and processing routes, and includes design factors to account for the deterioration of the laminated structure due to long-term exposure to combinations of chemical environments, elevated and sub-zero temperatures, and alternating loads. Geometries covered include cylindrical and spherical shells, rectangular tanks, flanges and domes. The document claims that the adoption of a minimum design factor of 8, and taking into account the other design factors, will prevent environmental stress cracking of the laminated vessels and tanks exposed to aqueous environments (see also Section 5.3).

4.4 HOT/WET CONDITIONING

The aerospace industry consider the worst world-wide environment to be represented by a relative humidity (RH) of 85% and a temperature of 70°C. Dry condition results (ie as removed from the autoclave), although not representative of actual usage, provide a convenient datum level for assessing the influence of environmental exposure on material

properties. Environmental conditioning is more severe than actual service conditions, but the degree of "acceleration" is not qualified.

Test specimens generally take the form of coupons or simple structural elements of constant thickness. Travellers are required to monitor specimen moisture content throughout the environmental history (ie manufacture, storage, pre-conditioning and testing) of the test component. ASTM D 5229 recommends a maximum test temperature of 70°C and 80°C for 121°C and 177°C cure epoxies, respectively. For other systems the maximum recommended test temperature is 25°C less than the wet T_g . These limits should not be exceeded. Moisture content is measured at constant intervals, usually $\sqrt{\text{time}}$. To minimise moisture uptake prior to preconditioning, specimens are stored in either a vacuum or a desiccator (sealed impervious bag containing desiccant), at room temperature. A moisture content datum can be established by using a second traveller, which is dried in a vacuum oven to constant weight, thus providing the moisture level at the start of the pre-conditioning.

4.5 LONG TERM LOADS

Static fatigue tests can be carried out using: (i) dead-weight and lever creep testing machines; (ii) a screw jack in series with a load cell; or (iii) self-stressing fixture where specimens are placed in a tube equipped with a pre-calibrated spring system for loading the specimens. Long-term cyclic tests can be carried out using either a standard servo-hydraulic test machine or a mechanical lever-arm machine incorporating a variable cam [3]. For small to medium enterprises, the use of a servo-hydraulic test machine for long-term testing is not an economic option. A bank of small creep machines can be assembled at a considerably lower cost compared with the capital outlay involved with purchasing servo-hydraulic units.

Most mechanical test methods used for static testing have the potential to be used in cyclic fatigue testing and as with environmental testing there are a number of inherent problems associated with load transfer and stiffness changes throughout the test duration. Software exists at NPL that allows for dynamic measurement of stiffness and damping factor continuously without the need to stop the test through examination of the hysteresis loops. The loss in stiffness may be quantified by discontinuing cyclic loading at selected cycle intervals to obtain the quasi-static axial stress-strain curve. Residual strength can also be determined by discontinuing cyclic loading and carrying out a quasi-static test to failure on the damaged material. **The main requirement for fatigue testing using these methods is that the damage mechanisms and failure mode are essentially identical to that observed for static tests.**

Fatigue behaviour is usually expressed in terms of the maximum applied fatigue stress S and fatigue life (number of cycles to failure N). For intercomparative purposes, fatigue strength data are normalised with respect to the ultimate strength of identically conditioned specimens measured at the fatigue test loading rate. The scatter associated with fatigue testing is large. Several techniques (eg radiography and AE) are used to monitor the initiation and accumulation of micro-damage within a laminate under fatigue loads. There is a concerted effort to develop ISO standards (ISO/CD 13,003) for fatigue, compatible with static test methods. The ISO draft standard defines the general principles applicable to fatigue tests for constant force, strain and displacement amplitude loading conditions at constant frequency. Test mode, specimen dimensions and applicable calculations are the same as those obtained from the equivalent test mode under static or monotonic loading conditions. ASTM D 3479 provides a testing specification for tension-tension fatigue of PMCs, using the same approach, based on ASTM D3039 for static properties. This test method, which allows for both constant load and constant strain loading conditions, is suitable for both design and quality assurance purposes. ASTM E 739 provides guidance on the determination of linearised stress-life ($S-N$) or strain-life ($\epsilon-N$) curves (see also [6]).

5. ACCELERATED AGEING

A number of techniques have been employed to accelerate ageing in PMCs. The usual approach for environmental testing has been to increase either the temperature, humidity level or concentration level of chemical degradant. The onset and accumulation of damage is further accelerated by testing materials under applied loads. For static fatigue testing, strain levels in excess of those normally experienced during service are applied to the test component. In the case of cyclic fatigue tests, increasing test frequency and amplitude will result in shorter life expectancy. Alternatively, the use of relatively small specimens conditioned for longer times under less severe conditions could be a more promising approach, provided the durability data generated can be scaled up to predict the long-term performance of more realistic size structures. This section examines test methods for inducing accelerated moisture and chemical degradation in PMCs.

5.1 EFFECT OF TEMPERATURE AND MOISTURE

The natural process of moisture absorption in composite structures is normally very slow, and this makes it very difficult to reach an adequate degree of degradation in a structural test element on practical timescales. It has been found necessary, therefore, to speed up the moisture diffusion process by employing an accelerated conditioning technique that can ensure a representative level of degradation in a significantly reduced time. The usual approach used to accelerate moisture uptake is to increase the diffusivity of the polymer by elevating the temperature of the conditioning environment. The diffusion coefficient of Hexcel Composites 914 epoxy resin system increases by a factor of 5 for an increase in temperature from 20°C to 60°C. This increase in diffusion rate is still slow, and ideally an increase in temperature to further accelerate the conditioning would be an advantage. However, there is a temperature limit to which most epoxy resins can be raised without affecting a change in the mechanism by which moisture is absorbed. Above this critical temperature, there is a strong possibility that the degradation mechanism is altered. At high temperatures and high humidity, the Fickian model is no longer valid.

An alternative approach to attempting to reach an equilibrium condition, involves altering the acceptance criteria to a given percentage of the chosen equilibrium condition. Conditioning the material to 95% of the full equilibrium state takes a relatively shorter time to reach than the full (100%) equilibrium condition. The time required to obtain the last 5% can take longer than the time taken to reach the 95% level. Clearly a very large saving in time is possible if a 95% of equilibrium can be justified in terms of a non-significant change in the structural strength and stiffness properties. Further acceleration can be obtained by increasing the RH to the maximum that can be readily maintained (ie 96%). It is required that the acceleration method takes full advantage of the high rates of diffusion given by temperature and humidity and still achieve a reasonably realistic moisture absorption. It is not satisfactory to precondition at 96% RH, so that the equilibrium level in the outer surface layers exceeds the bulk equilibrium level obtained due to exposure in service.

Three-stage conditioning

The approach is to expose the material to the maximum practical humidity level that is available (ie 96% RH) until the moisture level in the bulk reaches the bulk equilibrium level at the lower humidity/temperature condition. The second stage is to expose the material to a lower humidity level (eg 15% RH), a drying stage, to remove excess moisture in the outer surface layers. The third stage uses an RH that will give the desired final moisture level (ie 84% RH). The distribution flattens out across the entire material thickness. The effective use of this technique relies upon mathematically modelling the moisture diffusion for each stage of conditioning.

Accelerated ageing is considered a conservative approach as it yields greater degradation of properties compared with slower tests that closely simulate actual service conditions. Currently, there is no agreement upon a satisfactory accelerated test.

5.2 FIBRE BUNDLE TESTS

Static fatigue and stress-rupture tests have been successfully carried out on fibre bundles (loose and impregnated) and laminated coupon specimens [10-13] in order to determine resistance to the combined effect of moisture, elevated temperature and stress. This approach has also been successfully applied to fibre bundles exposed to acids and alkali solutions [1, 3]. There are no national or international standards specifically related to determining static fatigue and stress-rupture of fibres and laminated coupons.

Static fatigue testing of resin impregnated strands has been used at NPL as a convenient method of obtaining basic strength-time data in a relatively short period of time. The test can be used to evaluate the combined effects of applied stress and aggressive environments. **The small diameter (eg 0.5 to 1.0 mm) of the specimen enables rapid equilibrium with the surrounding environment.** The recommended gauge length is 80 mm and 150 mm for glass and carbon fibre systems respectively (Figure 4). Medium-term data combined with diffusion data for the composite has been used to determine the long-term strength and stiffness properties of bulk composites. The strength data are considered less reliable than the stiffness data, possibly due to scaling effects. Testing involves the application of a constant or changing load to the resin impregnated fibre bundle in the presence of an aggressive environment, often at elevated temperatures, for an extended period of time.

The static fatigue test is applicable to both impregnated and loose fibre bundles. **Testing unimpregnated tows offers a relatively quick means of evaluating fibre sensitivity to chemical attack.** A simple environmental cell, usually designed and built in-house, is used to encase the chemical agent. Conditioned specimens are then tested in tension to determine the stiffness and residual strength of the material. The degree of material degradation is determined by comparing the results from conditioned specimens with those obtained from unconditioned material (ie normalisation).

Concern has been expressed that diffusion of moisture or other chemical agents through the composite may differ between the unstressed and stressed states with the diffusion rate increasing with applied stress. The appearance of micro-cracks within the matrix or at the fibre-matrix interface will accelerate water ingress.

5.3 PRODUCT TESTING

Combined stress and environmental conditions are used in some product test methods. ASTM D 3681 and BS 5480 are often used by industry to determine the chemical-resistant properties of glass fibre-reinforced pipes. Testing consists of exposing the interior of a pipe section to a corrosive solution (Figure 1) while the pipe is subjected to a constant compressive deflection until the structure fails. The test is carried out at several deflections and the time-to-failure is measured for each test. The long-term chemical resistance of the pipe is obtained by extrapolating to 50 years the log-log linear regression line for the initial strain level. This test is appropriate to structures buried underground, such as sewer pipes, which will experience a constant strain throughout the life of the structure. A self-stressing fixture can be used to apply a constant displacement. Alternatively, a constant load, often in the form of a dead weight, is applied to the pipe section and the creep behaviour is monitored. Time to failure is plotted either as a function of stress or as a function of initial strain. This test is appropriate to above-ground pipes. It is quite common for creep tests under adverse environmental conditions to be carried out using standard test coupons (ie flexure and tension coupons). Acoustic emission (AE) is often used to monitor the structural integrity of glass fibre-reinforced plastic tanks and vessels under pressure.

6. ASPECTS REQUIRING ADDITIONAL CONSIDERATION

Most methods used for static testing have the potential to be used under environmental and cyclic fatigue loading conditions, although there are a number of inherent problems associated with load transfer and measurement of property changes (ie stiffness) throughout the duration of the test. This section examines aspects associated with testing under these conditions.

6.1 STRAIN GAUGING AND END TABBING

There are a number of contact and non-contact techniques available for measuring strain and displacement [14]. This section considers the use contact type transducers and end tabs for environmental testing, and the issue of autogenous heating associated with cyclic fatigue.

Most adhesives are sensitive to moisture (and other chemicals), which can often preclude bonding prior to preconditioning the polymer or composite. Moisture attack of an adhesive and strain gauges will occur from the top, edges and through the substrate beneath the gauge or tab. The situation is exacerbated at elevated temperatures. It is therefore important to ensure that the adhesive selected for bonding the strain gauge or end tab remains unaffected for the entire duration of the test and that strain gauges and associated electrical wiring is suitably encapsulated. Silicone rubber and foil have been placed over strain gauges in order to isolate the gauges during environmental conditioning. This process is only partially effective as moisture will eventually penetrate the bondline.

An approach often used, is to first condition the specimens and then bond the strain gauges and end tabs prior to testing, however this may require heat and pressure, which will induce drying out of preconditioned specimens. Room temperature curing anaerobic adhesives have proved satisfactory for bonding strain gauges and end tabs to moisture conditioned specimens. For hot/ wet conditions, a high temperature anaerobic adhesive can be used. Although anaerobic adhesives have good moisture, solvent and temperature degradation resistance, these adhesives are known to attack certain plastics. Hence, precautions need to be taken when selecting these materials for use with plastics. Cyanoacrylates (or super glues), which are sensitive to surface moisture and low pH levels, are unsuitable for environmental testing. Gauge manufacturers can provide information on adhesive selection and procedures for protecting strain gauges.

For cyclic loading, it is essential that fretting in the gripped region be prevented to avoid the possibility of premature failure. 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 heating can degrade the mechanical properties of the adhesive bond between strain gauges and the specimen. This could result in small errors in strain measurement, thus requiring correction of the data to account for the temperature rise. Measurements should also be carried out to determine the magnitude of creep within the strain gauge adhesive.

6.2 CONTROL OF MOISTURE CONTENT

At elevated temperatures, preconditioned specimens tend to dry out during the test, although for static tests the effects are minimal. For fatigue testing however, drying of the test component can be considerable, particularly from the outer plies of a laminate. To maintain constant moisture levels, a humidity controlled chamber is the preferred option, which is not affordable by most laboratories. Alternative approaches, include enclosing the specimen in a polythene bag containing a salt solution (specimen not in direct contact) appropriate to the humidity requirements of the test, or totally encapsulating the specimen with a sealant. For example, saturated potassium chlorate solution at 70°C will give a relative humidity of 85%.

For unencapsulated specimens, travellers are required to monitor the moisture content of the specimen throughout the duration of the test. The traveller should be machined from material adjacent to that used for the test component, thus ensuring the same lay-up, thickness and processing sequence. Ideally, the traveller should have similar dimensions to the specimen test section thus ensuring an accuracy in moisture content of $\pm 1\%$. For coupon testing, it has been common practice to allow a soak period of 10 minutes at the test temperature prior to testing. The purpose of "heat soaking" is to eliminate distortion due to non-uniform temperature distributions [9].

6.3 MEASUREMENT OF COEFFICIENT OF MOISTURE EXPANSION

Determination of the coefficient of moisture expansion involves measuring the dimensional change of the material in the principal material directions as a function of moisture concentrations (ie moisture weight gains). Tests involve immersing pre-dried specimens in a temperature controlled water bath and measuring the swelling strains as a function of weight gain. Specimens are pre-dried in an oven at a moderately high temperature (70°C or less) until the specimen weight reaches a constant value.

Moisture expansion or swelling can be measured with a micrometer and/or vernier calliper, or a travelling microscope. Strain gauges are not particularly suited for this purpose due to environmental attack on the strain gauge adhesive. A reduction in accuracy of the strain gauge measurements can be expected with exposure time. In addition, the presence of strain gauges on the surface of the specimen may inhibit moisture absorption within the vicinity of the gauge. Embedding encapsulated strain gauges within the laminate may be a future option, provided that the gauge does not cause any local disturbance, which may affect the process of moisture diffusion. The advantage of using embedded strain gauges is that swelling strains can be monitored throughout the duration of conditioning without the need to remove the specimen from the water bath. It is assumed that all absorbed moisture is translated into a change in matrix volume. In fact, during the initial stages of conditioning, water may also be filling microvoids and cracks. A plot of swelling strain versus weight gain will show a change in gradient for high porosity materials.

6.4 AUTOGENOUS HEATING DURING CYCLIC TESTING

Fatigue data are normally obtained at the highest frequency possible in order to minimise the duration of tests (ie accelerated testing). Restrictions on test frequency can arise from test equipment limitations (response time), time-dependent processes and autogenous (self-generated) heating. Autogenous heating, which increases with increasing load and frequency, can adversely effect the fatigue performance of the composite. Reliable data can be obtained at high frequencies provided the stress levels are low. As previously mentioned, test frequencies of the order of 10 to 30 Hz can result in a substantial increase in the mean surface temperature (up to 150°C, or greater). Autogenous heating effect can limit the test frequency to 1 to 2 Hz. This low maximum frequency and the resultant length of testing have contributed to the lack of data in excess of 10^6 cycles and the absence of evidence for fatigue limits in PMCs. The upper frequency limit will be dependent upon the thermal conductivity of the fibre/ matrix system, mode of loading and specimen size. The effect of autogenous heating is more marked in resin dominated laminates as the associated strains are larger in comparison with fibre dominated laminates. It has been recommended that the maximum acceptable temperature rise of the material surface should be limited to 10°C by reducing the test frequency. Stopping the test and allowing the specimen to cool before recommencing is also an option providing the dwells are acceptable [7]. Surface temperature needs to be monitored using either strategically placed thermocouples or thermal imaging equipment, although the latter is beyond the budget of most industrial facilities. Cooling is not a practical option as the cooling rate is too slow.

The mechanical properties of fibre-reinforced plastics will invariably be rate dependent, and this may be expected to be reflected in the fatigue properties. A number of factors however, such as autogenous heating and scatter for poorly reproducible materials, makes it difficult to isolate rate effects. In addition, the rate of stress application for constant frequency sine-wave testing will decrease with decreasing stress amplitude. This can be avoided by using a triangular waveform providing modern servo-controlled equipment is available. It is worth noting that for service applications, it is unlikely that the structure will be under constant conditions of stress and frequency.

7. THERMAL ANALYSIS TECHNIQUES

This section briefly examines the suitability of using thermal analysis methods for characterising environmental degradation on PMCs.

7.1 DIFFERENTIAL SCANNING CALORIMETRY (DSC)

DSC is used to determine the heat flow associated with material transitions as a function of time and temperature or changes in heat capacity using minimal amounts of material [15]. The technique provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions caused by phase changes, melting, oxidation and environmental degradation. The technique involves slowly heating a small sample of material and measuring the heat absorbed or emitted by the sample as a function of temperature compared to a reference material. Commercial systems are capable of operating over a temperature range of 150°C to 800°C. DSC can be used to measure the lowering of T_g with increasing moisture content for neat resins and PMCs.

For polymeric composites the samples extracted from the laminated material contains fibres and filler in addition to the matrix. The presence of these inert materials reduces measurement sensitivity and the result is often reported after normalising to 100% resin content. It has many advantages, including fast analysis time (30 minutes), easy sample preparation, a wide range of temperature applicability and excellent quantitative capability. ISO 11,357 specifies methods for the thermal analysis of plastics, including composite materials using DSC. Also several aerospace drafts which NPL is re-drafting refer to the ISO standard.

7.2 DYNAMIC MECHANICAL ANALYSIS (DMA)

This technique enables the determination of transition temperatures, storage modulus and loss modulus of the sample over a range of temperatures (-150°C to 800°C), frequencies (0.01 to 200 Hz) or time, by free vibration, and resonant or non-resonant forced vibration techniques (ISO 6721). Flexural samples are popular, whereby the sample is mounted on a clamp and then subjected to sinusoidal changes in strain (or stress) while undergoing a change in temperature. DMA is suitable for use with single fibres, fibre bundles, neat resins and laminated composites [15] with stiffnesses ranging from 1 kPa to 1,000 GPa. Tension, compression and flexure loading configurations can be employed. Three-point flexure specimens can be up to 50 mm in length, 15 mm wide and 7 mm thick. This configuration is readily adaptable for both resins and PMCs. It has many advantages, including fast analysis time (typically 30 minutes) and easy sample preparation. ISO 6721 specifies several modes for using DMA for plastics, including composites.

7.3 HEAT DISTORTION TEMPERATURE (HDT)

The heat distortion temperature (HDT) is often the only criterion used in determining a materials "fitness for purpose" at elevated temperatures (ie the upper limit of safe operating temperature at which a material can support a load for any appreciable time) [15]. The test procedure for determining HDT, as described in ISO 75, consists of three-point loading of a rectangular beam (120 mm long, 3 to 4 mm wide and 10 to 15 mm thick) immersed in a heated mineral oil bath, whose temperature is constantly increased at a rate of 2°C/min. The separation between the test specimen supports is 100 mm. The central deflection is continuously monitored. HDT is reached when the central deflection of the test specimen reaches a specified deflection value for a given nominal surface stress. The standard deflection will depend upon the specimen thickness (eg 0.32 mm for a specimen thickness of 10 mm and nominal surface stress of 1.80 MPa). The ISO standard applies to both neat resins (Part 2) and PMCs (Part 3) although as noted below some difficulties have been encountered in using Part 3. **Test data are only suitable for materials selection and acceptance, and not for design purposes.**

DMA and DSC provide a far better overview of the performance of environmentally conditioned material as function of temperature. The HDT method, which provides only a single-point value, is in comparison relatively affordable to small industrial organisations as testing and specimen preparation are relatively straightforward and economic; although a moderate capital outlay is required for the oil bath and displacement measurement equipment. **The results obtained using this method do not necessarily represent maximum use temperatures, because in practice essential factors such as time, loading conditions and nominal surface stress may differ from the different testing conditions.** Results obtained using ISO 75 indicate that the HDT values are higher for the unreinforced plastics (measured using Part 2) compared with corresponding results obtained from the reinforced version measured using Part 3. Both ISO 10,350-2 and Lloyds regulations recommend Part 2 until the anomaly associated with incorrectly specified applied stress levels is resolved.

8. CONCLUSIONS

It was clearly evident when reviewing the test methods and related standards for use in characterising environmental degradation of PMCs that there was no generic approach to accelerated ageing of these materials nor any consistency in data interpretation (ie life assessment). The general tendency has been to expose laminated structures to relatively severe test conditions, often well in excess of actual service conditions, resulting in conservative design practices. These structures usually take at least 12 months to condition, and in some case far longer (3 to 5 years), and may not be loaded fully during this time. Conditioning at 70°C/85% RH is frequently used in aerospace applications but may yield greater degradation of properties compared with longer tests under reduced conditions that more closely simulate actual service conditions.

Concern has been expressed that diffusion of moisture or other chemical agents through the composite may differ between the unstressed and stressed states with the diffusion rate increasing with applied stress. **The preferred approach is to expose composite materials to lower temperatures and lower chemical concentration levels (ie less severe environmental conditions) more representative of actual service conditions and that these materials should be mechanically stressed throughout the conditioning process (ie static or cyclic fatigue).** This is to ensure that the cumulative damage induced closely simulates damage incurred by engineering structures in service and that the data can reliably be extrapolated to longer time scales than is practically possible within the time frame of most test programmes.

One approach showing considerable potential as a means of accelerated ageing of PMCs employed small circular rods or impregnated tows manufactured from cured resin impregnated fibre bundles. The technique capitalises on the relatively short conditioning time and relatively low cost involved in manufacture and testing in comparison with standard test methods that use considerably larger coupon specimens. Static fatigue testing of resin impregnated strands is a convenient method of obtaining basic strength-time data under more realistic environmental conditions (ie closely simulating actual service conditions) than those used to induce accelerated ageing in laminated composites. Fibre tows can be used to evaluate quantitatively the combined effects of applied stress (static and cyclic fatigue) and aggressive environments with unimpregnated fibre tows offering a relatively fast method for evaluating fibre sensitivity to chemical attack. Tensile test data obtained from rovings with cured resin have a fairly direct correlation with the tensile properties of laminated composites. Combined with interfacial characterisation and thermal analysis techniques, it should be possible to relate the degradation measurements to the level of degrading agent, exposure time and loading conditions, and to extrapolate the data to predict the long term performance of generic PMCs. The development of accelerated ageing methods can be expected to result in improved material and design selection and to generate suitable design parameters for environmental and fatigue loading of composite structures. Design parameters based on test data obtained under ambient conditions are no substitute for reliable data representative of actual service conditions.

Combined with interfacial characterisation and thermal analysis techniques, it should be possible to relate the degradation measurements to the level of degrading agent, exposure time and loading conditions, and to extrapolate the data to predict the long term performance of generic PMCs. The development of accelerated ageing methods can be expected to result in improved material and design selection and to generate suitable design parameters for environmental and fatigue loading of composite structures.

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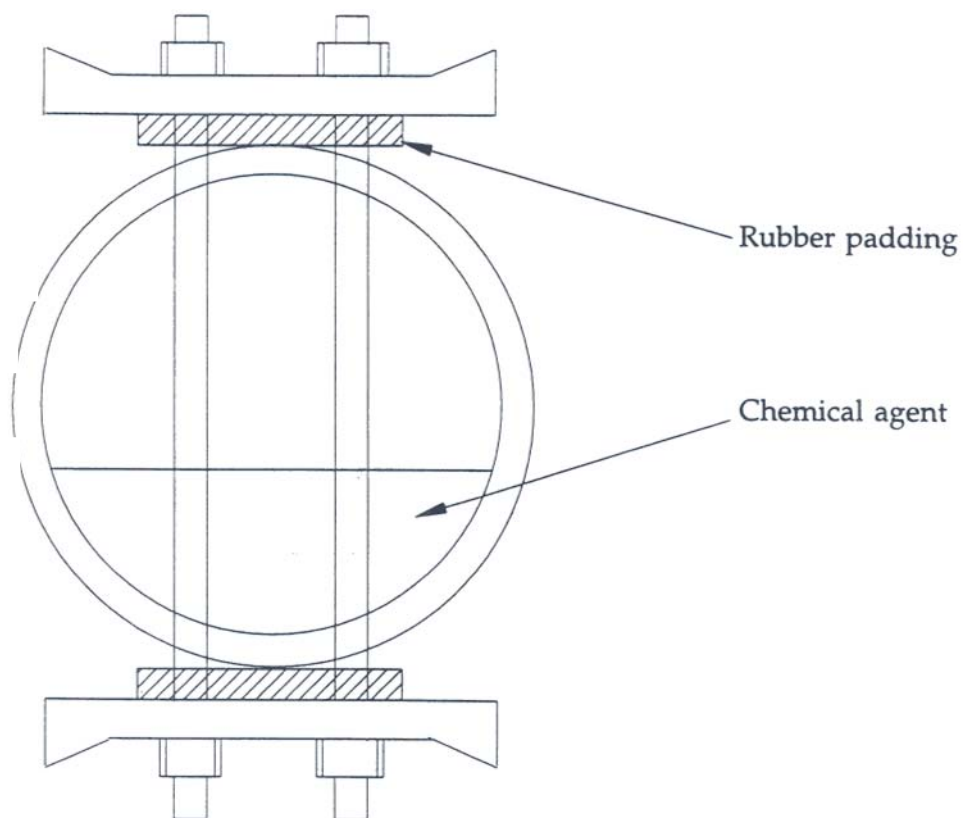


Figure 1 Strain-corrosion stress apparatus.

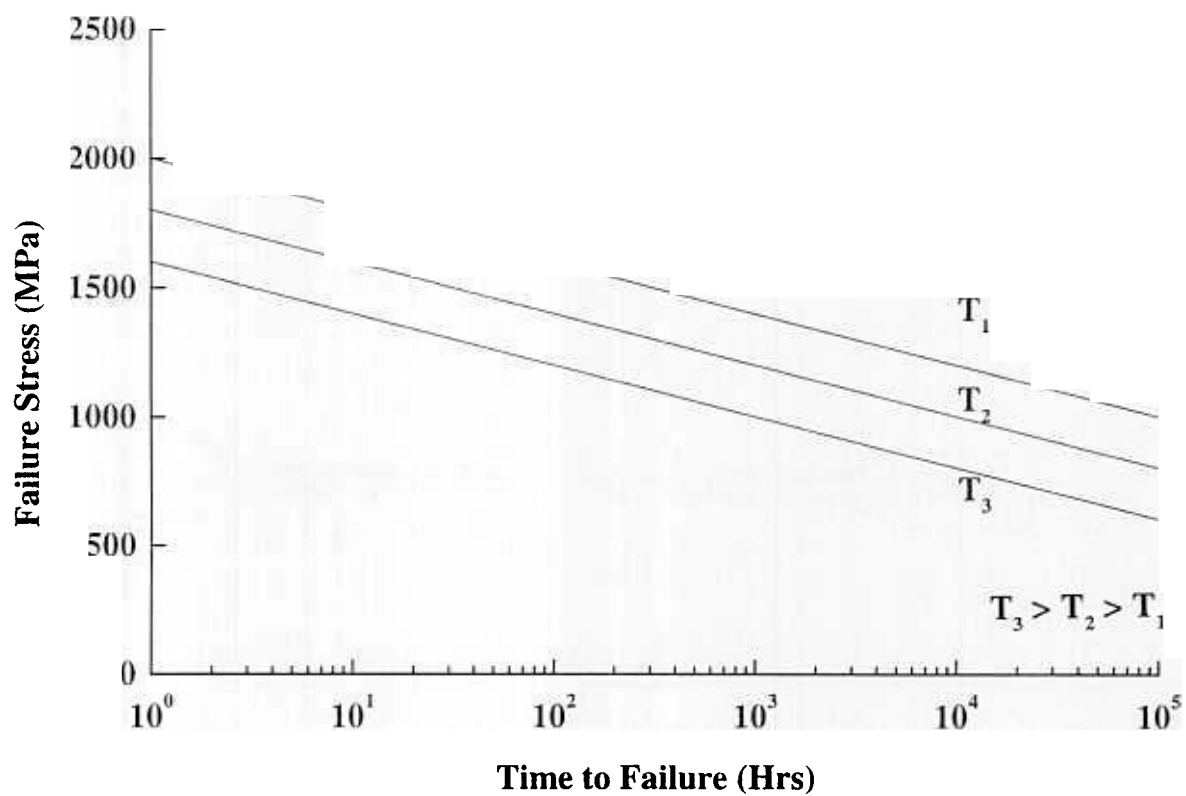


Figure 2 Log strength versus ageing time.

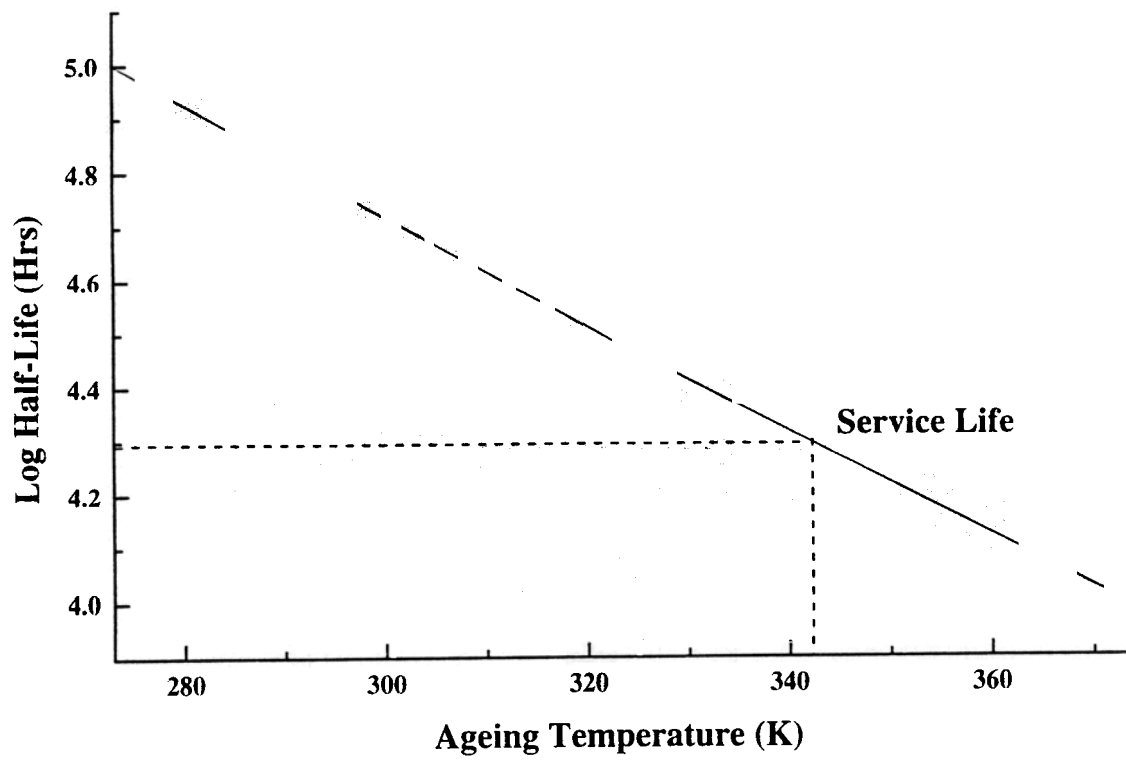


Figure 3 Schematic of strength half-life versus ageing temperature.

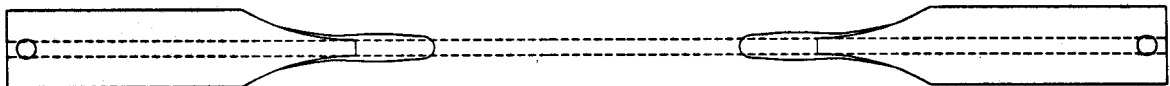


Figure 4 Fibre bundle test specimen.

APPENDIX I - ENVIRONMENTAL TEST METHOD STANDARDS

Table 1 ISO Standard Environmental Test Methods for Plastics/ Composites

NUMBER	TITLE	PURPOSE
ISO 62	Plastics - Determination of water absorption.	Moisture absorption
ISO 75	Plastics - Determination of temperature of deflection under load.	HDT
ISO 175	Plastics - Determination of the effects of liquid chemicals, including water.	Chemical resistance
ISO 291 prEN 2743	Plastics - Standard atmospheres for conditioning and testing.	Atmospheric conditioning
ISO 483	Plastics - Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value.	Relative humidity
ISO 554	Standard atmospheres for conditioning and/or testing - specification.	Standard atmospheres
ISO 846	Determination of behaviour under the action of fungi and bacteria - Evaluation by visual examination or measurement of change in mass or physical properties.	Biological degradation
ISO 877	Plastics - Methods of exposure to direct weathering, to weathering using glass-filtered daylight, and to intensified weathering by daylight using Fresnel mirrors.	Weathering
prEN 2378	Fibre reinforced plastics - Determination of water absorption by immersion in demineralised water.	Water absorption
prEN 2489	Fibre reinforced plastics - Determination of the action of liquid chemicals.	Chemical resistance
ISO 2578	Plastics - Determination of time-temperature limits after prolonged exposure to heat.	Elevated temperature
prEN 2823	Fibre reinforced plastics - Determination of the effect of exposure to humid atmosphere on physical and mechanical characteristics.	Moisture degradation
ISO 3025	Preferred test temperatures.	Preferred temperatures
ISO 4582	Plastics - Determination of changes in colour and variations in properties after exposure to daylight under glass, natural-weathering or artificial light.	Photodegradation
ISO 4589	Plastics - Determination of resistance to environmental stress cracking (ESC) - Bent strip method.	Environmental degradation
ISO 4600	Determination of environmental stress cracking (ESC) - Ball or pin impression method.	Environmental degradation

Table 1 ISO Standard Environmental Test Methods for Plastics/ Composites (Cont.)

NUMBER	TITLE	PURPOSE
ISO 4607	Plastics - Methods of exposure to natural weathering.	Natural weathering
ISO 4611	Plastics - Determination of the effects of exposure to damp heat, water spray and salt mist (on optical and colour properties).	Environmental conditioning
ISO 4892	Plastics - Methods of exposure to laboratory light sources.	Photodegradation
ISO 6252	Plastics - Determination of environmental stress cracking (ESC) - Constant tensile-stress method.	Environmental degradation
ISO 6914	Determination of ageing characteristics by measurement of stress at a given elongation.	Ageing
ISO 9370 (draft)	Guide for instrumental determination of radiant exposure in weathering tests.	Weathering
ISO TR 9673	Solar radiation and its measurements for determining outdoor weather exposures.	Photodegradation
ISO 11,248	Plastics - Thermosetting moulding materials - Evaluation of short-term performance at elevated temperatures.	Heat ageing
ISO CD 11,359-2	Plastics - Thermomechanical analysis (TMA). Part 2: Determination of linear thermal expansion coefficient and glass transition temperature.	Thermal analysis
ISO CD 11,359-3	Plastics - Thermomechanical analysis (TMA). Part 3: Determination of softening temperature.	Thermal analysis
ISO 11,403-3 (draft)	Part 3: Environmental influences on properties.	Environmental degradation
ISO/CD 13,760-2	Method for approximating the maximum allowable stress for pipes transporting fluids under pressure.	Stress allowables

Table 2 BSI Standard Test Methods for Environmental Testing of Plastics/ Composites

NUMBER	TITLE	PURPOSE
BS 2011	Environmental testing procedures and severities of tests designed to assess the durability of electrotechnical products under conditions of use, transport and storage.	Environmental testing
BS 2782: Part 5: Method 551A	Plastics - Determination of the effects of exposure to damp heat, water spray and salt mist.	Salt spray
BS 4618	Recommendations for the presentation of plastics design data. Part 4 Environmental and chemical effects.	Environmental testing
BS 4994	Design of construction vessels and tanks in reinforced plastics.	Design allowables
BS 5480: Part 2	Specification for glass reinforced plastics (GRP) pipes and fittings for use for water supply and sewerage. Part 2: Design and performance requirements.	Sewerage
BS 5691	Thermal endurance properties of electrical insulating materials.	Thermal - dry heat
BS EN 60068	Environmental testing - Tests A. Cold/Test B. Dry Heat.	Environmental testing

Table 3 ASTM Standard Test Methods for Environmental Testing of Plastics/ Composites

NUMBER	TITLE	PURPOSE
ASTM B 117	Salt spray (fog) testing	Salt spray
ASTM C 272	Standard test method for water absorption of materials for structural sandwich constructions.	Moisture absorption
ASTM C 481	Standard test method for laboratory aging of sandwich constructions.	Accelerated ageing
ASTM C 581	Standard practice for determining chemical resistance of thermosetting resins in glass fibre reinforced structures intended for liquid service.	Chemical resistance
ASTM D 543	Standard practice for evaluating the resistance of plastics to chemical reagents.	Chemical resistance
ASTM D 570	Standard test method for water absorption of plastics	Moisture absorption
ASTM D 618	Standard practice for conditioning plastics and electrical insulating materials for testing.	Environmental conditioning
ASTM D 756	Standard practice for determination of weight and shape changes of plastics under accelerated service conditions.	Accelerated ageing.
ASTM D 1042	Standard test method for linear dimensional changes of plastics under accelerated service conditions.	Dimensional stability
ASTM D 1435	Standard practice for outdoor weathering of plastics.	Weathering
ASTM D 1499	Standard practice for operating light and water exposure apparatus (carbon arc type) for exposure of plastics.	Equipment - operation
ASTM D 1870	Standard practice for elevated temperature aging using a tubular oven.	Heat ageing
ASTM D 1975	Standard test method for environmental stress crack resistance of plastic injection moulded open head pails.	Environmental resistance
ASTM D 2288	Standard test method for weight loss of plasticisers on heating..	Heat ageing
ASTM D 3045	Standard practice for heat ageing of plastics without load.	Heat ageing
ASTM D 3681	Standard test method for chemical resistance of fibreglass (glass fibre reinforced thermosetting resin) pipe in a deflected condition.	Chemical resistance
ASTM D 3753	Standard specification for glass fibre reinforced polyester manholes.	Chemical resistance
ASTM D 3826	Standard practice for determining degradation end point in degradable polyethylene and polypropylene using tensile tests.	Degradation
ASTM D 4019	Standard test method for moisture in plastics by coulometric regeneration of phosphorus pentoxide.	Moisture absorption
ASTM D 4097	Standard specification for contact - moulded glass-fibre reinforced thermoset resin corrosion - resistant tanks.	Chemical resistance
ASTM D 4102	Standard test method for thermal oxidative resistance of carbon fibres.	Thermal oxidation

Table 3 ASTM Standard Test Methods for Environmental Testing of Plastics/ Composites (Cont.)

NUMBER	TITLE	PURPOSE
ASTM D 4329	Standard practice for operating light and water apparatus (fluorescent UV and condensation type) for exposure of plastics.	UV radiation
ASTM D 4364	Standard practices for performing accelerated outdoor weathering of plastics using concentrated natural sunlight.	Accelerated weathering
ASTM D 4398	Standard test method for determining the chemical resistance of fibreglass reinforced thermosetting resins by one side panel exposure.	Chemical resistance
ASTM D 4459	Standard practices for operating an accelerated lightfastness Xenon arc type light exposure apparatus for the exposure of plastics for indoor applications.	Photodegradation
ASTM D 4508	Standard test method for chip impact strength of plastics.	Environmental degradation
ASTM D 4585	Standard practice for testing water resistance of coatings under controlled condensation.	Water resistance
ASTM D 4674	Standard test method for accelerated testing for colour stability of plastics exposed to indoor fluorescent lighting and window filtered daylight.	Accelerated ageing
ASTM D 4762	Guide for testing of automotive/industrial composite materials.	Automotive test methods
ASTM D 5071	Standard practices for operating Xenon arc type exposure apparatus with water for the exposure of photodegradable plastics.	Photodegradation
ASTM D 5208	Standard practice for operating fluorescent ultraviolet and condensation apparatus for exposure of photodegradable plastics.	UV radiation
ASTM D 5229	Standard test method for moisture absorption and equilibrium conditioning of polymer matrix composite materials.	Moisture absorption
ASTM D 5272	Standard practice for outdoor exposure testing of photodegradable plastics.	Weathering
ASTM D 5419	Standard practice for environmental stress cracking (ESCR) of threaded plastic closures.	Environmental degradation
ASTM D 5437	Standard practice for weathering plastics under marine floating exposure.	Weathering/marine
ASTM D 5510	Standard practice for heat ageing of oxidatively degradable plastics.	Heat ageing
ASTM E 698	Standard test method for Arrhenius kinetic constants for thermally unstable materials.	Activation energies (DMA)
ASTM F 1164	Standard test method for evaluation of transparent plastics exposed to accelerated weathering combined with biaxial stress.	Accelerated weathering
ASTM G 90	Standard practice for performing accelerated outdoor weathering of non-metallic materials using concentrated natural sunlight.	Photodegradation

Table 4 BS Standards Relating to Environmental Conditioning

Standard	Subject/ Comments	Materials	Conditions	Specimen
BS 2011: Part 2.1Ca: 1977 (= IEC 68-2-3)	Specifies test conditions under steady state damp heat environment	Not specified	Test at 40±2°C, 93±2%/-3% RH for 4, 10, 21 or 56 days	Not specified
BS 2011: Part 2.1Cb: 1990 (= IEC 68-2-56)	Specifies test conditions under steady state damp heat environment (primarily for equipment)	Electrotechnical products	Increase temperature by 1°C/min to the test temperature then increase humidity to required level in 2 hrs max, to one of the following test conditions: A - 30±2°C, 93±3% RH B - 30±2°C, 85±3% RH C - 40±2°C, 93±3% RH D - 40±2°C, 85±3% RH	Not specified
BS 2011: Part 2.1Db: 1981 (= IEC 68-2-30)	Specifies test conditions under cyclic damp heat environment	Not specified	Cycle between 25±3°C and 40°C (12 hr + 12 hr cycle) for 2, 6, 12, 21 or 56 cycles, or between 25±3°C and 55°C for 1, 2 or 6 cycles	Not specified
BS 2011: Part 2Ka (= IEC 68-2-11, JIS C 0023, NFC 20-711)	Comparison between specimens of the resistance to deterioration from salt mist NOT suitable as general salt corrosion test or for evaluation of individual specimens - QA only	Not specified	5% by weight salt solution, containing <0.1% sodium iodide, <0.3% impurities Test conditions: 35±2°C, min 85% RH Duration: 16 hrs, 24 hrs, 2 days, 4 days, 1 wk 2 wks or 4 wks	Not specified

Table 4 BS Standards Relating to Environmental Conditioning (Cont.)

Standard	Subject/ Comments	Materials	Conditions	Specimen
BS 2011: Part 2Kb BS EN 60068-2-52 (= IEC 68-2-52, JIS C 0024)	Method of evaluating resistance of components to salt atmosphere	Not specified	5% by weight salt solution, containing <0.1% sodium iodide, <0.3% impurities Test conditions: 35±2°C, min 85% RH Spray for 2 hrs between 15°C and 35°C for one of the following tests: A - 4 spray periods (2 hrs each), humidity storage period of 7 days after each B - 3 spray periods (2 hrs each), humidity storage period of 20-22 hrs after each C - 1 test cycle (4 spray periods, humidity storage period of 20-22 hrs after each), followed by storage period of 3 days at 23±2°C, 50±5% D - as C, but with 2 test cycles E - as C, but with 4 test cycles F - as C, but with 8 test cycles	Not specified
BS 2011: Part 2Rc: 1990 (= IEC 68-2-18)	Specifies test conditions under water immersion: A - Water tank B - Pressurised water chamber	Watertight electrotechnical products	A - fresh tap water or sea water at 35°C max Water head: 0.15m, 0.4m, 1m, 2m or 5m Duration: 0.5, 2 or 24 hrs B - 35°C max Pressure: 20, 50, 100, 200, 500, 1000, 2000, 5000 or 10000 kPa Duration: 2, 24 or 168 hrs	Not specified
BS 2782: Pt 5: Method 551A	See ISO 4611			
BS 5350: Part BXX (= prEN 1239)	Describes method for evaluating the freeze-thaw stability of adhesives	Adhesives	Measure viscosity, freeze for 16 hrs in sealed container at -5°C, -10°C, -15°C or -20°C then thaw for 8 hrs. Repeat cycle twice more, then measure viscosity again.	N/A
BS 5350: Part C7: 1990	Describes application of test methods in which force is applied to adhesive bond to determine displacement under constant force	As appropriate for tests in BS 5350: Parts C1, C3, C5, C14 and C15	As appropriate to test method	As appropriate to test method

Table 4 BS Standards Relating to Environmental Conditioning (Cont.)

Standard	Subject/ Comments	Materials	Conditions	Specimen
BS EN 60068-2-1: 1993	Specifies test conditions for cold environments: Test Aa. Non-heat dissipating specimen with sudden change of temperature Test Ab. Non-heat dissipating specimen with gradual change of temperature Test Ad. Heat dissipating specimen with gradual change of temperature	Not specified	Temperature: -65°C, -55°C, -40°C, -25°C, -10°C, -5°C or +5°C Duration: 2 hrs, 16 hrs, 72 hrs or 96 hrs Test Aa - specimen at ambient temperature introduced into test chamber at test temperature Tests Ab and Ad - specimen and chamber both at ambient temperature to start, temperature decreased at 1K/min to test temperature, then increased at 1K/min after test duration	Not specified, but: Tests Aa and Ab - climate chamber with forced air circulation Test Ad - distance from specimen to wall = min 15 cm, volume of chamber = min 5X volume of specimen
BS EN 60068-2-2: 1993 (= IEC 68-2-2)	Specifies test conditions under dry heat: Test Ba - Non-heat dissipating specimen with sudden change of temperature Test Bb - Non-heat dissipating specimen with gradual change of temperature Test Bc - Heat dissipating specimen with sudden change of temperature Test Bd - Heat dissipating specimen with gradual change of temperature	Not specified	Temperature: 200°C, 175°C, 155°C, 125°C, 100°C, 85°C, 70°C, 55°C, 40°C or 30°C (all $\pm 2^\circ\text{C}$) Also allowed: 250°C, 315°C, 400°C, 500°C, 630°C, 800°C, 1000°C ($\pm 2^\circ\text{C}$) Duration: 2, 16, 72 or 96 hrs	Not specified

**Table 5 Standards Relating to Environmental Conditioning
Polymer Matrix Composites - Laminates and Sandwich structures**

Standard	Subject/ Comments	Materials	Conditions	Specimen
ASTM C 272-91	Moisture absorption	Sandwich constructions. Honeycomb, foam and balsa wood.	A - Water immersion $23 \pm 3^{\circ}\text{C}$, 24 hrs. B - $71 \pm 3^{\circ}\text{C}$, 85 $\pm$ 5%RH, 30days. C - Water immersion $23 \pm 3^{\circ}\text{C}$, 48 hrs+.	Length = 76.2 mm Width = 76.2 mm Thickness = 12.7 mm
ASTM C 481-62	Ageing (water) - Six complete cycles, < 30 minutes between cycles, followed by exposure at $23 \pm 1.1^{\circ}\text{C}$, 50 $\pm$ 2% RH until constant weight ($\pm 1\%$) reached.	Sandwich constructions.	Water immersion $49 \pm 3^{\circ}\text{C}$, 1 hr. Steam/vapour spray 71 or $93 \pm 3^{\circ}\text{C}$, 3 hrs. Store -40 or $-12 \pm 3^{\circ}\text{C}$, 20 hrs. Hot dry air 71 or $99 \pm 2^{\circ}\text{C}$, 3 hrs. Steam/vapour spray 71 or $93 \pm 3^{\circ}\text{C}$, 3 hrs. Hot dry air 71 or $99 \pm 2^{\circ}\text{C}$, 18 hrs.	Specimen geometry and dimensions are in accordance with the method or material standard specification used
ASTM D 5229-92	Through-thickness direction moisture absorption of flat or curved panels. Single phase Fickian moisture diffusion. Edges sealed - moisture absorption through faces.	Laminates. Not suitable for non-Fickian material behaviour.	121°C cure epoxies - $T_{\text{MAX}} = 70^{\circ}\text{C}$. 177°C cure epoxies - $T_{\text{MAX}} = 80^{\circ}\text{C}$. Other $T_{\text{MAX}} \leq T_g - 25^{\circ}\text{C}$. Water or humid air.	Length = 100 mm Width = 100 mm Width/thickness ≥ 100 Edges sealed with adhesively bonded stainless steel foil
CRAG 901	Determination of the moisture diffusivity characteristics.	Continuous unidirectional and 0/90 symmetric laminates. Not suitable for non-Fickian material behaviour.	120°C cure epoxies - $T_{\text{MAX}} = 45^{\circ}\text{C}$. 180°C cure epoxies - $T_{\text{MAX}} = 70^{\circ}\text{C}$. Humidity, 70%, 85% and 95%.	Length = 75 mm Width = 75 mm Thickness = 2mm
CRAG 902	Conditioning under hot/wet environments.	Continuous multidirectional laminates.	Drying and pre-conditioning 120°C cure epoxies - $T_{\text{MAX}} = 45^{\circ}\text{C}$. 180°C cure epoxies - $T_{\text{MAX}} = 70^{\circ}\text{C}$. 85% RH.	Length and width are in accordance with the method or material standard specification used Thickness 1-4 mm
ACOTEG ACO/TP/17	Determination of thermal spikes. Material is preconditioned, thermally spiked, post-conditioned and tested at ambient.	Unidirectional laminates.	Pre-conditioning 70°C , 85% RH (≥ 14 days). Thermal spike temperatures $90 - 300^{\circ}\text{C}$. Post-conditioning 70°C , 85% RH (≥ 14 days).	Specimen geometry and dimensions are in accordance with the method or material standard specification used

**Table 6 Standards Relating to Design Allowables and Endurance Calculations
(Plastics and Polymer Matrix Composites)**

Standard	Subject/ Comments	Materials	Conditions	Specimen
ISO 2578	Method for evaluating thermal endurance properties at elevated temperatures	Plastics	Constant temperature for > 5000 hrs	Specified in the method or material standard being used
ISO 11248	Determination of the relative performance temperature (RTP) at which a property value corresponds to 50% of a reference value	Thermosetting moulding materials	Short term exposure at elevated temperatures for 24 hrs	Specified in the method or material standard being used
ISO/CD 13760.2	Method for approximating the maximum allowable stress for pipes transporting fluids under pressure	Plastic pipes	Varying temperature and/or pressure	N/A
prEN 4238	Method for evaluating material property degradation due to thermal ageing	All fibre-reinforced plastics Laminates	Constant temperature Atmospheric pressure No mechanical load Heat-up rate 2 to 3°C/min.	Specified in the method or material standard being used Thickness = 2-3 mm