

Environmental and Fatigue Testing of Fibre Bundles and Impregnated Strands

Currently, there is no generic approach to accelerated testing of polymer matrix composites (PMCs). Two contrasting methods for accelerating environmental degradation have been identified. The first employs severe test conditions to induce an accelerated response in standard composite specimens or sub-components, whereas the second uses fibre bundle specimens which allow rapid conditioning under more realistic temperatures, stresses and concentrations reflecting actual service conditions.

Test methods have been developed for characterising the tensile properties of fibre bundles and impregnated strands under static and cyclic (tension-tension) loads, and/or aggressive environments. These test methods require shorter conditioning times and less manufacturing and testing costs compared with current practices. The methods have been validated for a range of test conditions using glass and carbon fibres and their composites. The use and limitations of these methods for determining fibre sensitivity and composite resistance to chemical attack, and static and cyclic fatigue loading conditions are discussed. Supporting test data, including environmental stress corrosion data from tests conducted on specimens aged in water, sulphuric acid and sodium hydroxide solution are presented. Details of specimen geometry, dimensions, preparation and testing are provided for both fibre bundle and impregnated strands, along with a description of test apparatus for conducting long-term environmental testing.

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Introduction

A review of test methodologies and standards [1] revealed that the general approach to accelerated ageing of PMCs is 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 cases far longer (3 to 5 years), and may not be fully loaded during this time. Conditioning at 70°C and 85% relative humidity (RH) is frequently used in aerospace/defence applications, however, this may produce greater degradation of properties compared with longer tests under moderate and more realistic conditions.

The use of fibre bundle specimens (loose and impregnated) allows rapid conditioning (i.e. suitable for accelerated testing) under more realistic temperatures, stresses and concentration levels reflecting actual service conditions. Static fatigue and stress-rupture tests have been carried out on fibre bundles [2-3] in order to determine resistance to the combined effect of chemicals (including water), elevated temperatures and stress. Although successful, there are no national or international standards specifically related to determining static fatigue and creep-rupture of fibres and laminated coupons.

Test methods suitable for standardisation have been developed at NPL for characterising the tensile properties of fibre bundles and impregnated strands under static and cyclic (tension-tension) loads, and/or aggressive environments [4-5]. These test methods require shorter conditioning times and less manufacturing and testing costs than current practices.

Standards

Several test methods exist for measuring the tensile strength and longitudinal modulus of single fibre filaments and fibre tows or rovings (i.e. bundle of continuous parallel filaments). These methods are usually intended for yarns having a diameter of less than 2 mm (typically 0.5 to 0.8 mm), or a linear density lower than 2,000 tex (weight per unit length in g/km). Fibre tow methods include testing of both unimpregnated (i.e. loose or dry) and impregnated

rovings. ISO DIS 3341 [6] specifies a method for the determination of the breaking force and failure strain of unimpregnated glass fibre rovings, whereas BS ISO 9163 [7] allows for both unimpregnated and impregnated fibre tows. ASTM D 4018 [8] and ISO 10618 [9] specify test methods for carbon fibre tows. Manufacturers will often use in-house quality assurance methods for determining fibre tensile properties.

Fibre Tow Testing

Tensile and cyclic fatigue (tension-tension) data for loose fibre bundles (tows) can be obtained using the specimen geometry shown in [Figure 1](#). Specimen preparation is carried out according to BS ISO 9163. End tabs, although not mandatory, are recommended to avoid mechanical damage to the specimen ends and to ensure failure occurs within the gauge length. For cyclic loading, the use of end tabs minimises the possibility of fibre fretting/wear within the gripped region. BS ISO 9163 specifies a method for moulding end tabs. A cold-curing epoxy system is used to produce the end tabs. It is important that the resin system has low viscosity to ensure full impregnation of the fibres within the tab region and good mechanical and dynamic properties. Poor impregnation can result in non-uniform loading of the fibres within the fibre bundle resulting in low strength values with large uncertainties.

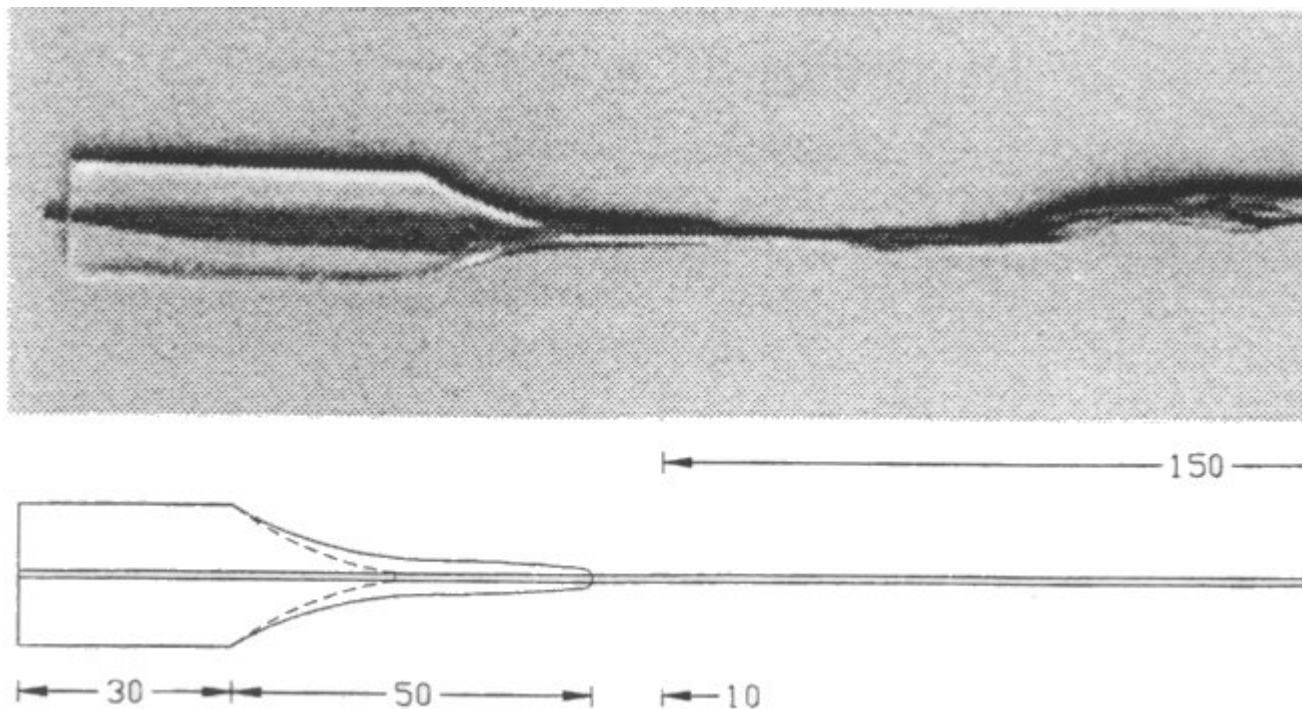


Figure 1: Fibre tow test specimen (mm).

The end tabs are cast in silicon moulds, which can be produced using a two-part, low-shrinkage, room-temperature curing silicon elastomer. Care needs to be taken to ensure that the mould is free of air bubbles (degassed) and the internal surfaces of the mould are smooth and free of defects. Moulds tend to split and break due to repeated handling and therefore need to be replaced on a regular basis (5 to 6 castings per mould). A schematic diagram showing the dimensions of the template used for producing the moulds is shown in [Figure 2](#).

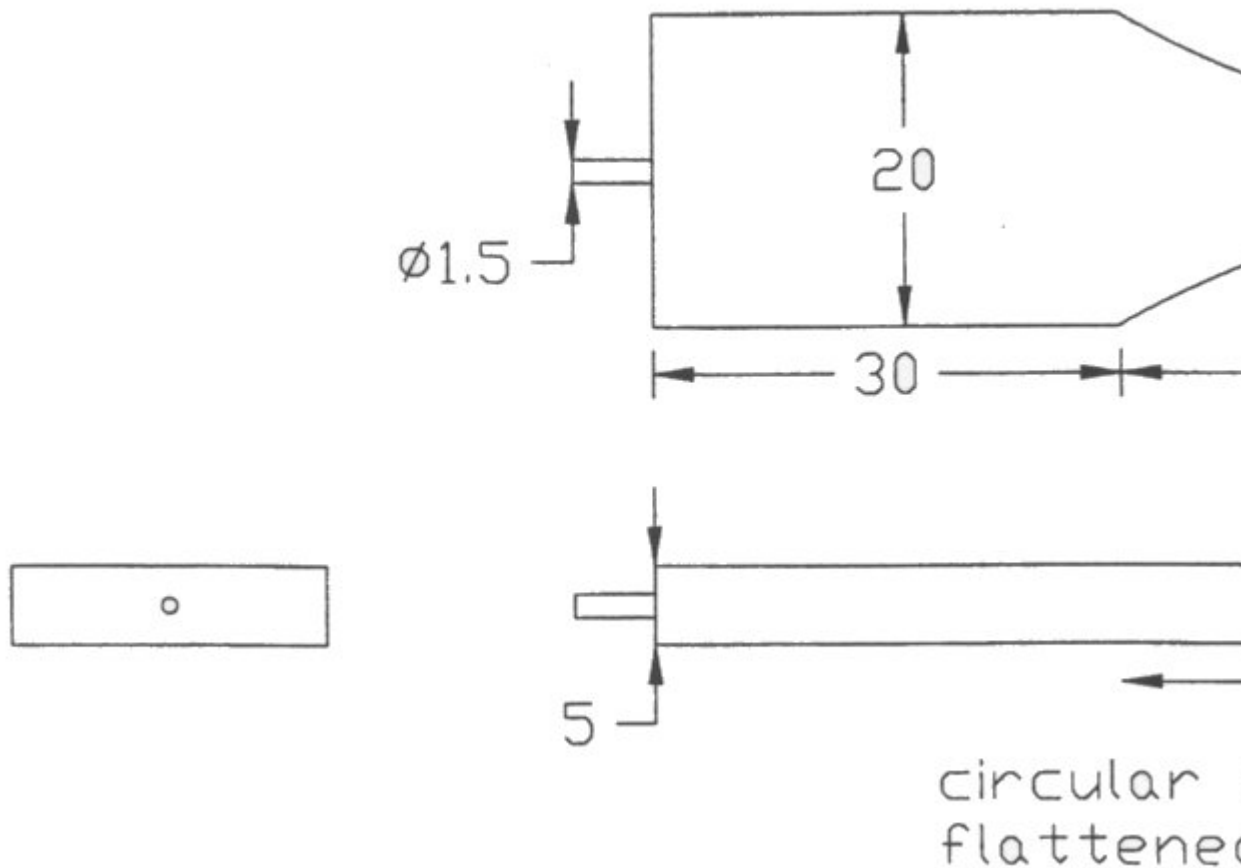


Figure 2: Schematic of moulding template used for producing end tabs (mm).

The end tab length (110 mm) specified in BS ISO 9163 exceeds the grip length of most manual or servo-hydraulic gripping systems, and hence, specimen tab lengths need to be shortened to match the grip length of the test equipment (typically 50 mm).

Initial strength and strength retention of fibres can be significantly reduced due to friction between fibres. An approach used by industry to protect conditioned or unconditioned glass fibres against abrasion is to impregnate the fibre tow with a rosin beeswax mixture. The effect of the protective coating on tow stiffness, although minimal, can be determined from comparative data obtained from unimpregnated material. Lubricating agents in the glass sizing formulation are introduced to protect fibres from abrasion.

Fibre bundle testing is relatively straightforward using low capacity mechanical or servo-hydraulic loading frames. Testing consists of loading the specimen to failure at a constant displacement rate (< 250 mm/min). The load and displacement are monitored during the test. Modulus and strain to failure are measured using an extensometer (mechanical or non-contact). It is important that the gripping system maintains good axial alignment throughout the duration of the test. Failure should occur within the specimen gauge length otherwise the strength data are invalid.

Testing Of Composite Rods

A resin impregnated tow, when cured, produces a rigid specimen which is easier to handle and test than a loose bundle of fibres and also ensures uniform loading of the fibres in the bundle. The fibre tow is impregnated with resin and oven cured. It is important when feeding the fibre bundle through the resin bath that the speed and tension is sufficient to produce a uniformly impregnated test specimen. The resin must be capable of fulfilling that requirement. An automated strand preparation device equipped with a tension-regulating system (typically 0.2-20 N) is generally used. Specimen fabrication is fast and inexpensive, although considerable capital outlay is required to purchase fibre impregnation equipment. Specimens can be produced using a filament winding machine. Carbon and glass fibre-reinforced rods can be pultruded in a range of resin systems (e.g. polyester, vinylester and epoxy). The minimum diameter size that can be pultruded is typically 1.5 to 2.0 mm.

The test geometry specified in BS ISO 9163 proved unsatisfactory for measuring the tensile strength of composite rods with a diameter of 1.5 mm, or greater. The interface between the composite rod and epoxy resin end tabs failed prematurely, resulting in pull-out of the composite rod rather than tensile failure within the gauge length. As a result, a new method was developed for producing end tabs for composite rods. [Figure 3](#) shows a schematic diagram of a composite rod specimen with adhesively bonded composite end tabs that was successfully employed in static and fatigue testing glass/polyester and carbon/vinylester rods with a nominal diameter of 1.5 mm.

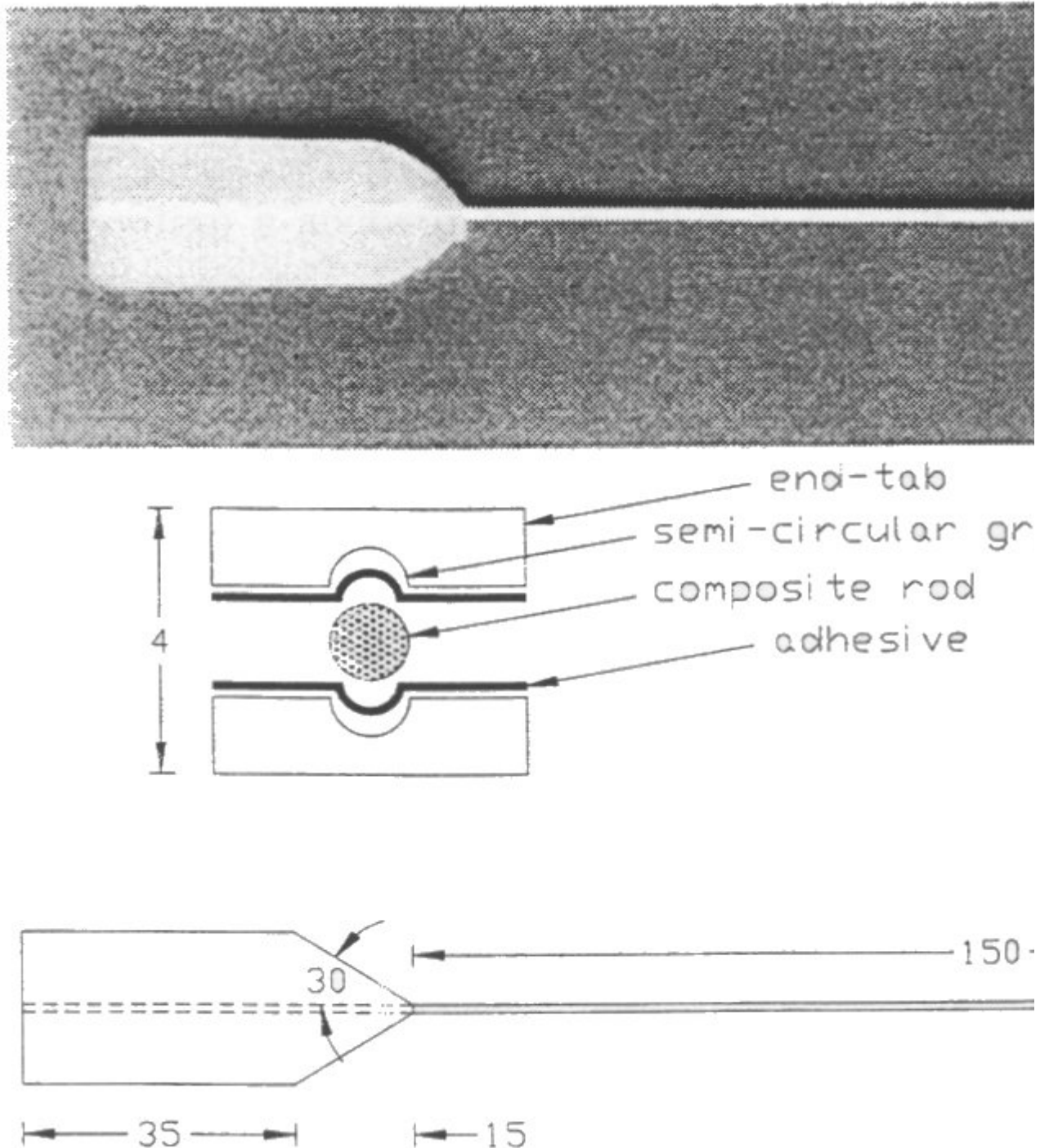


Figure 3: Composite rod specimen with adhesively bonded composite end tabs (mm).

End tabs are manufactured from plain woven glass fabric reinforced epoxy laminate (1.6 mm thick) with the fibre axes of the fabric set at $\pm 45^\circ$ to the specimen axes. The use of a film adhesive with carrier to bond the end tabs was found to reduce both preparation time and adhesive wastage. The carrier ensured good contact and constant bondline thickness. Specimen preparation is also cleaner in comparison with paste adhesives. The semi-circular groove to accommodate the specimen, shown in [Figure 3](#), can be cut with a diamond slitting wheel (water

lubricated). The thickness of the wheel should match the rod diameter. It is essential that the end tabs are dried before bonding to remove moisture, which can compromise the adhesive bond.

The use of composite end tabs, described above, is limited to rods with diameters of 1.5 mm, or less. Cylindrical moulded end tabs made with a high shear strength epoxy adhesive paste that were 1.25 mm thick and 50 mm long proved satisfactory for testing rods up to ~3 mm in diameter. Tensile failure consistently occurring within the gauge length. [Figure 4](#) shows a composite rod specimen with moulded end tabs.

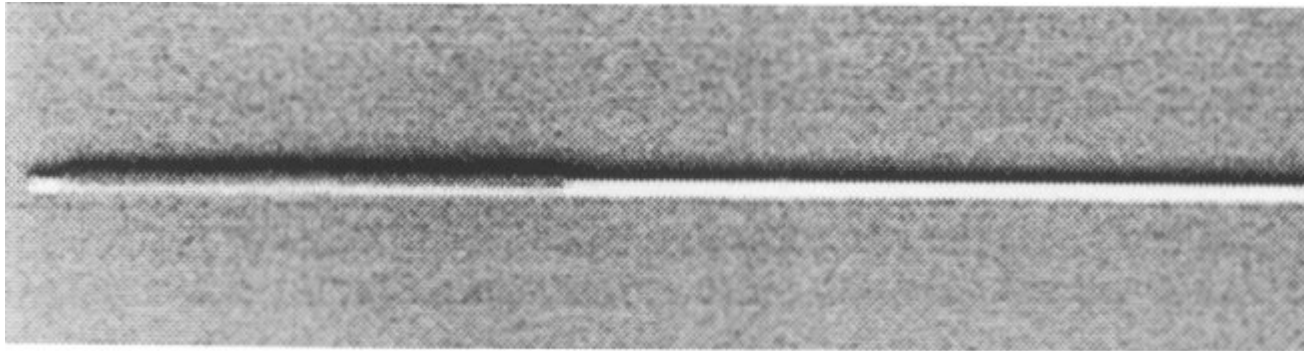


Figure 4: Composite rod specimen with moulded end tabs.

Larger diameter rod specimens tend to crush in the grips. This may be remedied by using thicker moulded end tabs and/or by adjusting the V-notch angle of the jaw face of the grips to ensure the points of contact are equidistant around the circumference. Problems may be encountered under cyclic loading conditions (e.g. possible fretting or wear of end tabs).

Fibre Tow Tensile Properties

Strength analysis assumes that all fibres in a tow have identical strength and therefore will break at the same strain. In practice, the level of variability will normally be wide, and thus the failure strength of a fibre tow of nominally identical fibres will most probably be less than the mean failure stress of individual fibres. This is because the load capacity of the fibre bundle gradually deteriorates as the weaker fibres fail with increasing load.

Tests conducted on E-glass and Graphil 34-700 carbon fibre tows (supplied by RBJ) showed that the uncertainty in the tensile breaking stress for the E-glass fibre tows ([Table 1](#)) was noticeably greater than for the carbon fibre tows ([Table 2](#)). Two batches of glass fibre tow taken from two widely separated sections of the same reel, were observed to have significantly different values of breaking stress and strain. Glass fibres are more susceptible to damage through handling and moisture degradation and therefore should be stored in a dry (i.e. low humidity) area. It is recommended that at least 10 specimens are tested per batch.

Table 1: Tensile Results for E-glass Fibre Tows

Property	Measured Value
Breaking Stress (N/Tex)	
Batch 1	0.31 ± 0.04
Batch 2	0.38 ± 0.02
Breaking Strain (%)	
Batch 1	2.11 ± 0.14
Batch 2	3.01 ± 0.07
Tensile Modulus (N/Tex)	
Batch 1	18.3 ± 0.5
Batch 2	18.9 ± 0.5

Table 2: Tensile Results for Graphil 34-700 Carbon Fibre Tows

Property	Measured Value
Breaking Stress (N/Tex)	0.86 ± 0.05
Breaking Strain (%)	1.40 ± 0.05
Tensile Modulus (N/Tex)	71.1 ± 2.7

Environmental Corrosion Resistance

Carbon fibres exhibit excellent resistance and can be expected to remain unaffected by exposure to water, weak acids and weak alkalis at elevated temperatures. In contrast, the mechanical properties of E-glass fibres degrade in the presence of water, mineral acids, weak organic acids, salt solutions and alkalis (NB. hydrofluoric acid dissolves most glasses). For example, tensile strength of E-glass tows is reduced to 10%, or less, of its initial strength after less than 5 days immersion in 5% sulphuric acid (H_2SO_4) at 23°C [10]. This environmental conditioning regime is included in ASTM D 3681 [11] for strain-corrosion testing of pipe rings for underground sewer pipes. The reason being that in hot climates, concentrations of 5 wt%, or more, of H_2SO_4 have been detected in sewer pipes (NB. Hydrogen sulphide (H_2S), a commonly occurring gas in sewers, is oxidised to sulphuric acid by the Thiobacillus bacteria).

The loss of strength due to exposure to mineral acids is associated with a depletion of metallic ions near the fibre surface leading to surface micro-cracks, which act as stress concentrations. The effective diameter of the fibre is also reduced through leaching of minerals from the surface layer. Maximum degradation rate of E-glass fibres occurs in solutions of 1N to 4N for HCl and 1N to 8N for H_2SO_4 . (NB. A one normal (1N) solution contains one gram-equivalent weight of a particular substance dissolved in 1 litre of solution). Leaching of minerals from fibres had minimal effect on the solution's pH level.

Corrosion of E-glass fibres by strong alkalis (e.g. sodium hydroxide (NaOH)) involves a breakdown of the silica network by hydroxide (OH^-) ions resulting in dissolution of the silica network, and a gradual loss in weight and strength. Strength retention of E-glass fibre tows after 2 weeks immersion in a 1N solution of NaOH was approximately 30%. Tensile modulus was reduced by 15% in the same timescale.

E-glass fibres degrade in water with deionised water being much more aggressive than either tap water or sea water. Degradation of E-glass fibres can be mainly attributed to leaching of alkali oxides (sodium and potassium oxide) from the fibre surface resulting in micro-cracking in the surface layer. E-glass fibre strands were observed to degrade steadily in deionised water with the rate of degradation being temperature independent over the temperature range 23°C to 70°C (Figure 5). Within a 3 week period the tensile strength and modulus reduction was 50% and 15%, respectively. In contrast, carbon fibres remained unaffected when immersed in water. Immersion of E-glass fibres in boiling water for short periods (24 hours) results in a dramatic loss in strength (75% strength reduction compared with dry samples). Loss of strength can be expected to be permanent at all conditioning temperatures and exposure times. A thermostat controlled water bath is recommended for immersion tests.

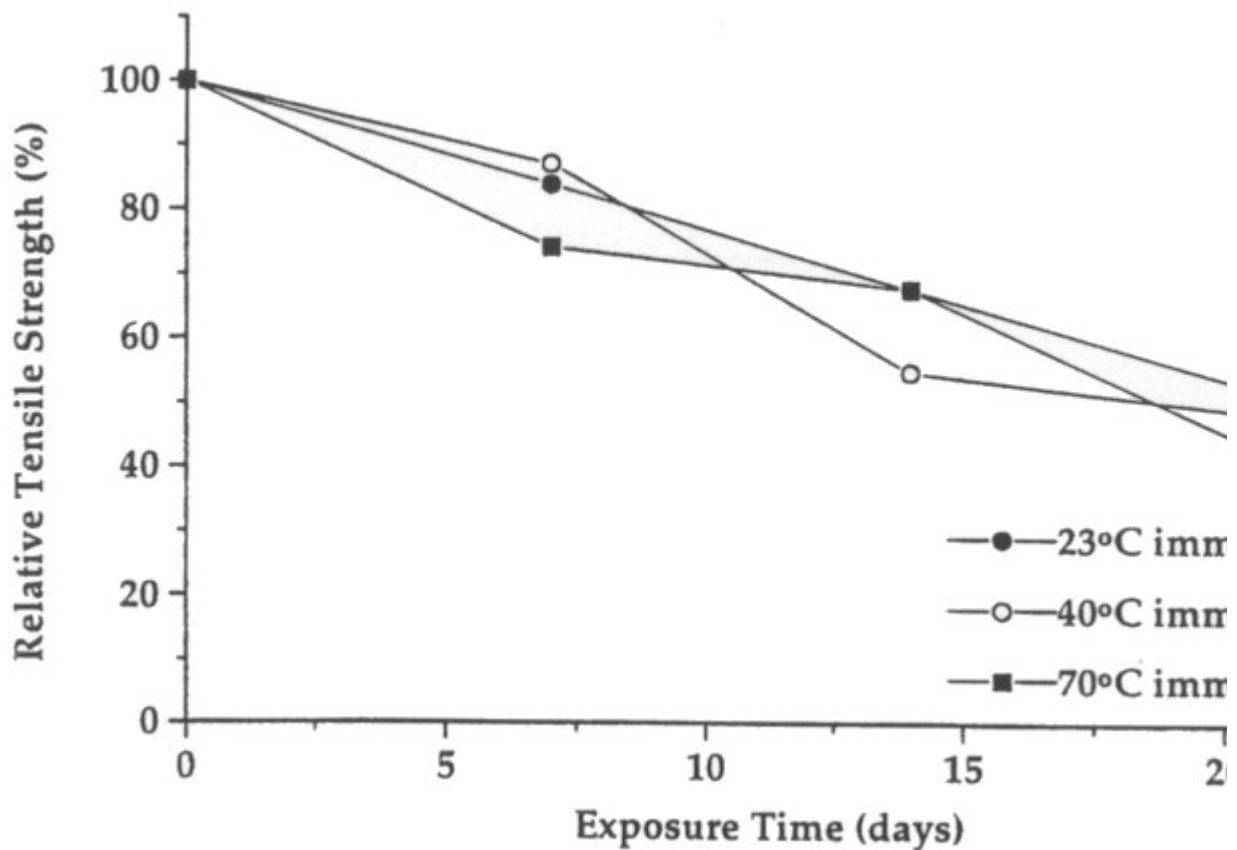


Figure 5: Strength retention of moisture conditioned E-glass fibre tows.

Moisture Conditioning

Most PMCs are capable of absorbing small, but potentially damaging amounts of moisture from the surrounding environments with the degree of degradation that occurs being linked directly with the amount of moisture absorbed. The absorbed water may adversely affect the material in a number of ways: (i) dimensional changes (swelling); (ii) reduction in the glass-transition temperature T_g of the resin; and (iii) reduction in mechanical properties. This will effectively lower the maximum service or operating temperature of the composite.

The two main types of basic moisture conditioning are: (i) fixed-time conditioning, where a test specimen is exposed to a conditioning environment for a specified period of time; and (ii) equilibrium conditioning, where a specimen is exposed until the material reaches equilibrium with the conditioning environment. The first technique is routinely employed for screening purposes. This approach results in non-uniform moisture distribution through the thickness of the test specimen. Test data obtained from specimens conditioned in this manner are only considered suitable for comparing different batches of the same material or for quality control tests.

Ideally, comparative studies of water absorption properties of polymeric materials should be carried out only using the equilibrium moisture content method with identical exposure conditions. Comparisons between composite materials with different moisture absorption characteristics are possible if the materials are pre-conditioned to equilibrium. The thicker the material the longer the time required to reach equilibrium, hence the use of relatively thin specimens to determine the "through-the-thickness" moisture diffusion coefficient. The conditioning temperature should not exceed the glass transition temperature of the resin system.

BS EN ISO 62 [12] describes a procedure for determining the moisture absorption properties and/or diffusion coefficients in the "through-the-thickness" direction of flat and curved solid plastics. The method can be applied to humidity exposure and liquid immersion.

Moisture Conditioning of Composite Rods

Conditioning of composite rods consisted of exposing pre-dried specimens to a steady-state environment (i.e. constant temperature and constant moisture exposure level) and measuring the global moisture gain after a prescribed period of time. The amount of water absorbed by the test specimen was determined by measuring the change in mass (i.e. difference between initial mass and the mass after exposure).

Specimens were pre-dried in an oven maintained at $50 \pm 2^\circ\text{C}$ until the specimen weight reached a constant value. The temperature of the drying oven should not exceed the glass-transition temperature of the resin system. It should be noted that under standard laboratory conditions many composite materials absorb significant levels of moisture.

Pre-conditioned specimens were wiped with a clean cloth to ensure all surface water was removed prior to weighing. Damage may accumulate during long-term conditioning, and hence handling and monitoring of test specimens was kept to a minimum. Travellers were used to monitor specimen moisture content throughout the environmental history (i.e. manufacture, storage, pre-conditioning and testing). Traveller specimens had identical material properties, geometry and processing history as the test specimen. It is important that the moisture content be established prior to testing and that specimens be tested within an hour of removal from the conditioning environment in order to ensure that minimal water loss occurs.

Strength Retention of Composite Rods

Small diameter composite rods (i.e. 1.5 mm) are suitable for accelerated testing, enabling rapid uptake of aggressive agents, with any degradation effects on the composite being quickly manifested (within 7 days) in terms of strength reduction. The results presented in [Figure 6](#) demonstrate the rapid degrading effect that deionised water has on glass/polyester rods. The composite rods were immersed in deionised water for periods up to 6 weeks at temperatures ranging from 23°C to 70°C . Specimens were withdrawn at selected intervals and then tested within an hour of removal from a water bath. The 6 week period is considered representative for the development of an accelerated ageing test programme. The rate of degradation can be seen to increase at elevated temperatures.

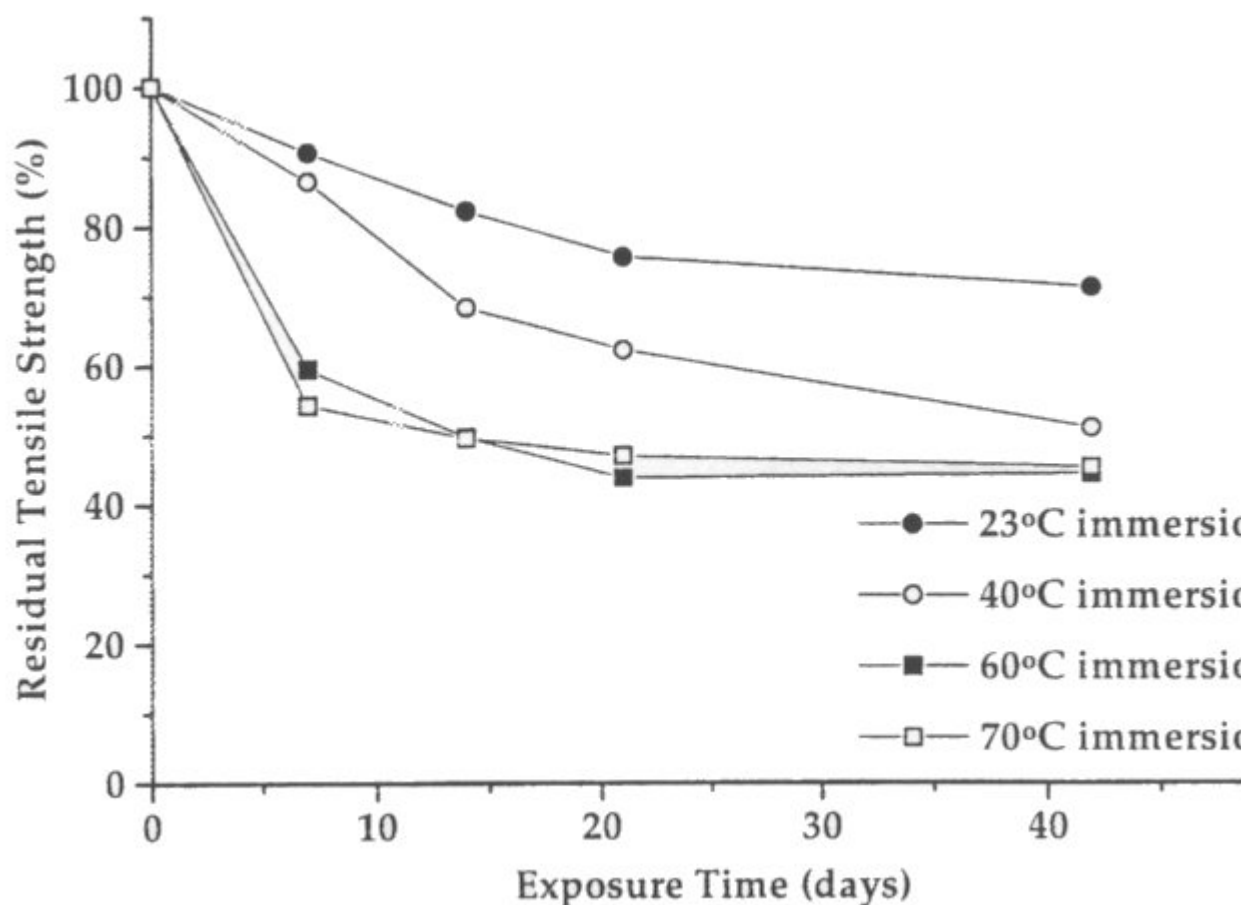


Figure 6: Strength retention of moisture conditioned E-glass/polyester rods.

Static Fatigue

Static fatigue tests were used to evaluate the combined effect of environment and applied static tensile stress on fibre tows and 1.5 mm diameter composite rod specimens. Tests were carried out using a bank of small creep machines, with the loading chain consisting of a screw jack in series with a load cell. The specimen was fully encapsulated in the liquid environment (i.e. deionised water, sulphuric acid or sodium hydroxide solutions). The acid and alkali solutions employed were identical to those previously described. [Figure 7](#) shows the environmental test apparatus used for long-term fatigue tests. The system developed allows for the liquid environment to be circulated. It is important that the test apparatus and all attachments (e.g. grips) are chemically resistant to the test environment.

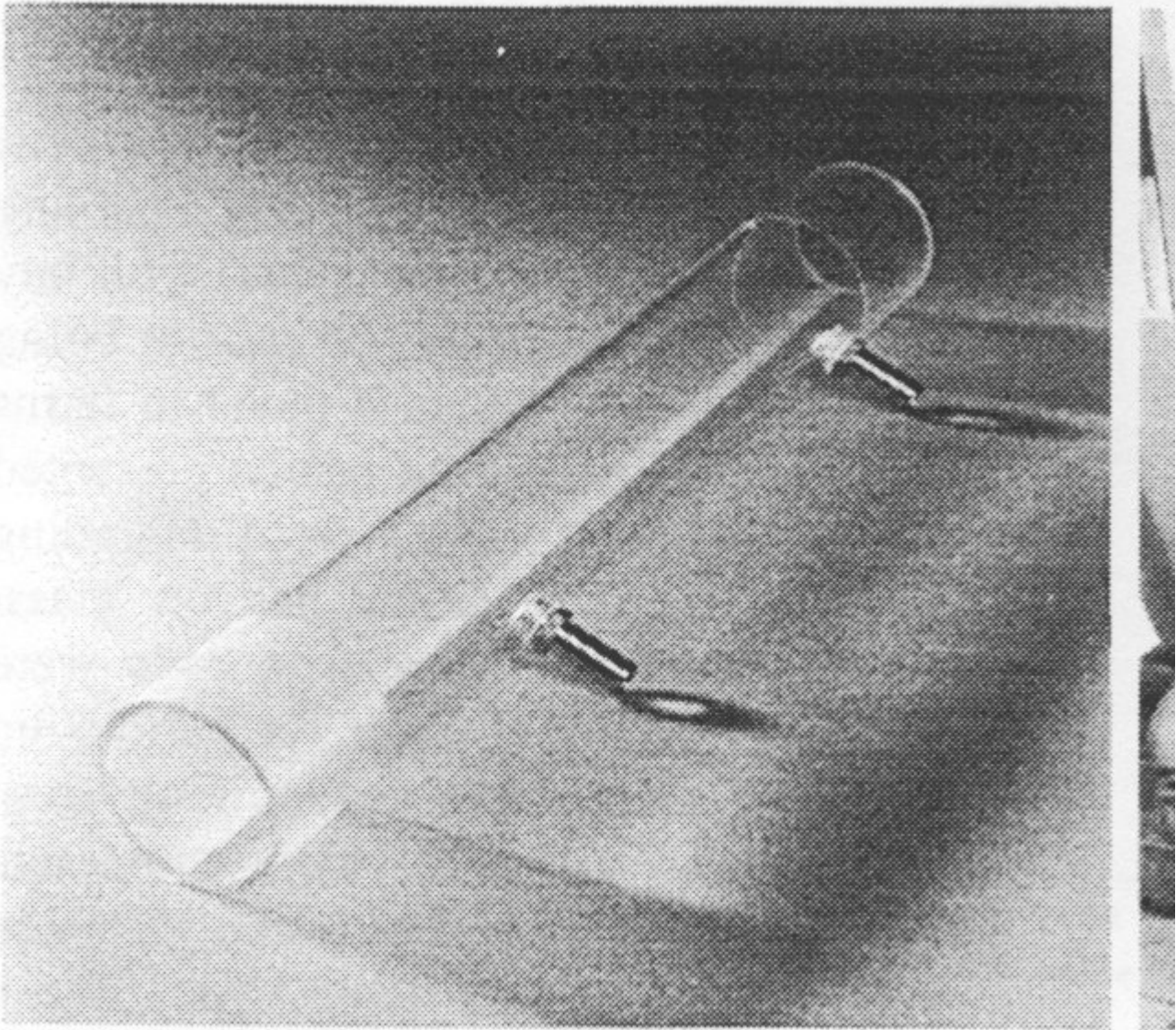


Figure 7: Long-term environmental static fatigue apparatus.

Most of the static tests under ambient conditions were carried out using Instron servo-hydraulic test machines. This is a relatively expensive option compared with the use of small stand alone test frames, but expedient due to the relatively short duration of most of the tests. For short duration tests (i.e. static loads close to the maximum load at failure) load relaxation occurs and it is therefore necessary to continuously adjust the manual screw jack on the creep frame in order to maintain a constant load. The use of servo-hydraulic controls avoids this problem. Manually operated systems are best suited to long-term testing where loads are relatively low and load relaxation is minimal.

The rate of strength reduction of the E-glass fibre tows ([Figure 8](#)) in water was relatively high compared with air (25% strength loss in 1 hour compared with 5.5 days). In contrast, carbon fibres can maintain static loads approaching the UTS (ultimate tensile strength) of the fibre (~90% UTS) indefinitely in both air and water.

The normalised stress rupture curves for E-glass fibre tows in air or water when plotted on a linear-log plot can be approximated by the equation (see [Figure 8](#)) [5]:

$$\sigma_{APP}/\sigma_{UTS} = 1 - k \log_{10} t_f$$

where σ_{APP} is the applied stress (or load), σ_{UTS} is the ultimate tensile strength of the unconditioned material, k is the slope and t_f is time to failure. The rate of strength reduction is accelerated in the presence of moisture. The value of k was 0.044 and 0.072, in air and water respectively.

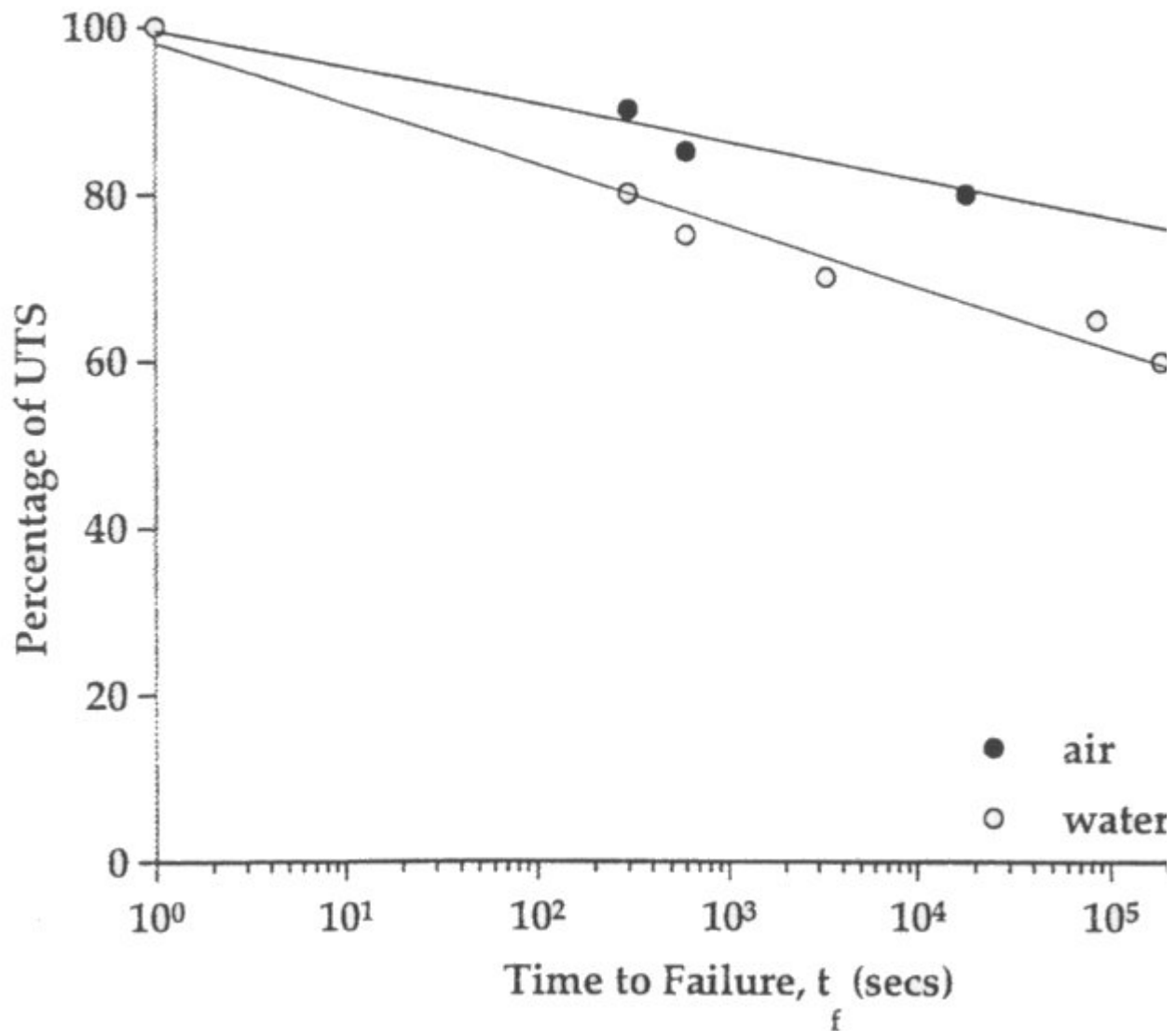


Figure 8: Stress rupture of E-glass fibres in air and water.

A combination of sustained tensile load and exposure to NaOH or H₂SO₄ results in rapid failure of E-glass fibre tows. Stress rupture (failure) was almost spontaneous when the fibres were subjected to combined load and 1N sulphuric acid solution. The preferred test method was to first load the specimen and then introduce the chemical agent.

Unidirectional glass/polyester rods exhibited good creep resistance. The composite rods sustained a static load equivalent to 50% UTS under ambient conditions for at least 4 months ([Figure 9](#)). The resin matrix acts as a chemical barrier to the corrosive effects of moisture/water on the glass fibres, and hence the extended life compared to loose fibre bundles. It is advisable in manufacturing glass fibre-reinforced structures that a resin rich layer is present on the outer surface(s) and that this layer is not breached, thus exposing the underlying fibres.

The stress rupture performance of carbon fibre-reinforced systems is far superior to that of glass fibre based systems. Carbon/vinylester rods can sustain a static load equivalent to 70% UTS under ambient conditions for at least 12 days, whereas glass/polyester rods can only sustain 70% UTS for 10 hours.

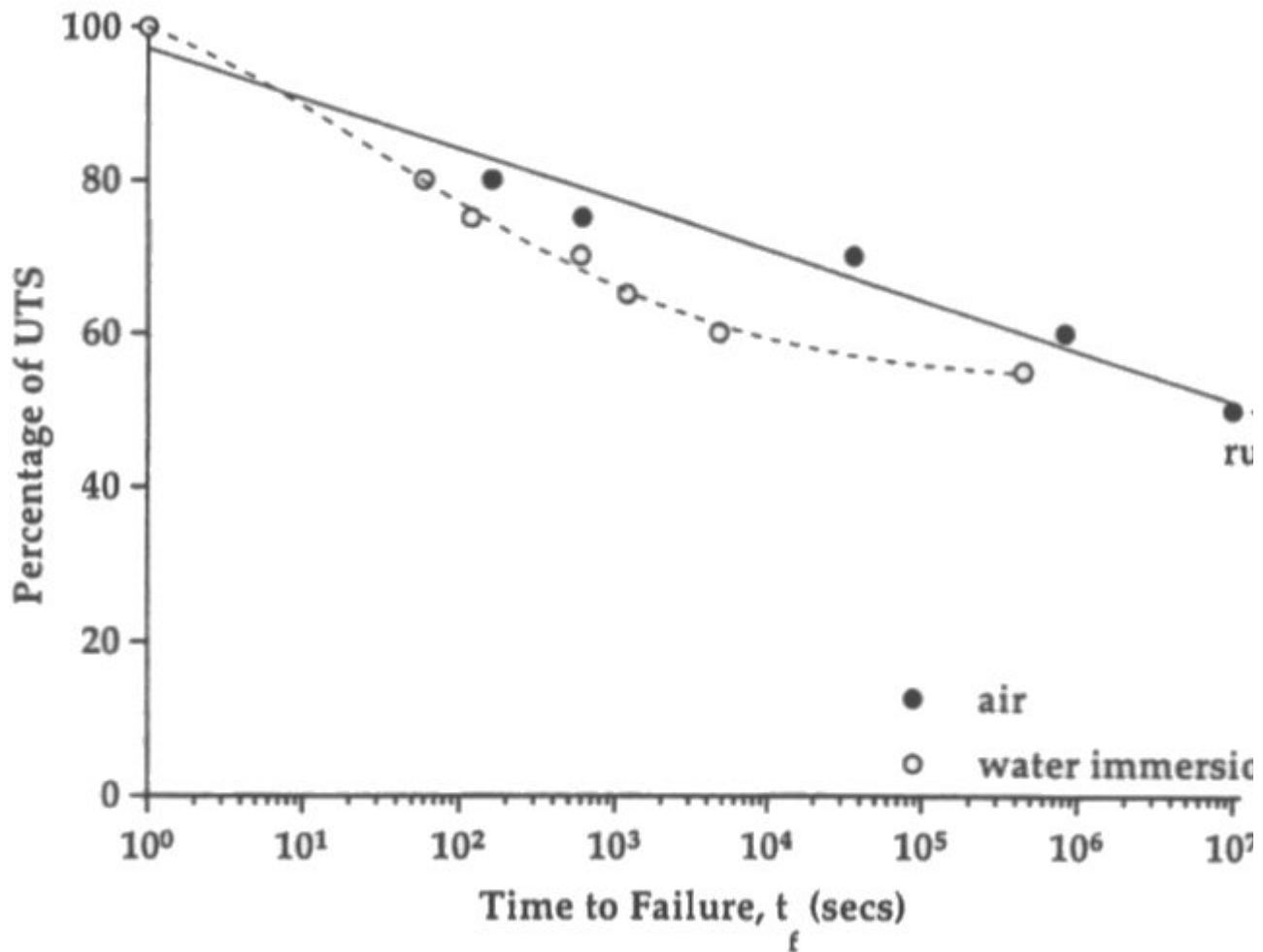


Figure 9: Stress rupture of E-glass/polyester rods in air and water.

Cyclic Fatigue

Unimpregnated glass-fibre and carbon-fibre tows exhibit excellent fatigue resistance under tension-tension cyclic loading. Fibre tow specimens can maintain applied stress levels approaching the UTS of the fibre bundle for at least 10^7 cycles. Accumulated damage in the form of broken fibres was observed to be minimal. Provided the applied stress is smaller than the UTS and falls outside the scatter band (i.e. 2 standard deviations), then the lifetime of the fibre tow will exceed 10^7 cycles.

Unidirectional carbon fibre composites are known to possess excellent fatigue resistance in comparison with metals and glass fibre composites. Fatigue tests conducted on composite rod specimens showed that fatigue resistance of carbon/vinylester was far superior to glass/polyester. In each case, 1.5 mm diameter composite rod specimens were subjected to constant amplitude (sinusoidal waveform) tension-tension cyclic fatigue.

The fatigue tests were carried out in load control using an Instron servo-hydraulic test machine at a frequency of 5 Hz. The stress ratio R ($\sigma_{\text{MIN}}/\sigma_{\text{MAX}}$) was equal to 0.1. All tests were carried out under standard laboratory conditions. Fatigue rated servo-hydraulic (wedge-action) grips were used to grip the composite rods.

Fatigue tests were conducted at five stress levels (i.e. 80%, 70%, 55%, 40% and 25% UTS) with specimens tested at each level. For inter-comparative purposes, fatigue strength data was normalised with respect to the ultimate tensile strength of identically conditioned specimens measured at the fatigue test loading rate. The normalised **S-N** data (**S** is applied stress and **N** is the number of cycles to failure) for tests carried out on glass/polyester rods

(Figure 10) can be represented by a linear relationship as follows [4]:

$$\sigma_{\text{MAX}}/\sigma_{\text{UTS}} = 1 - k \log_{10} N$$

where σ_{MAX} is the maximum applied load, σ_{UTS} is the ultimate tensile strength and k (the slope) is the fractional loss in tensile strength per decade of cycles. The value of k was approximately 0.1.

There was no detectable damage in the glass/polyester rods following 10^7 cycles at σ_{MAX} of 25% UTS. At higher applied stresses (i.e. 40% UTS, or greater), specimens failed catastrophically with failure occurring invariably within the gauge-length, although frequently near the end tabs. Non-progressive failure occurs due to insufficient material being available to accommodate local stress concentrations induced through fibre fracture. Fatigue performance of glass/polyester rods was poor in comparison with the standard test geometry.

The results from fatigue tests conducted on carbon/vinylester rods indicate a minimum fatigue life of 10^6 cycles for an applied stress level of 70% UTS. Technical difficulties associated with the presence of high stress concentrations near the tab ends prevented the successful completion of tests on the carbon composite rods at applied stress levels equal to, or higher than, 70% UTS.

Residual measurements carried out on glass/polyester rods showed that the longitudinal tensile modulus remains constant over the entire lifetime of the unidirectional composite. In contrast, tests conducted at an applied stress level of 40% UTS showed that tensile strength decreases rapidly within the first 25,000 cycles and then remains constant at 70% UTS for the remaining lifetime. Initial loss of strength is associated with damage in the end tab region.

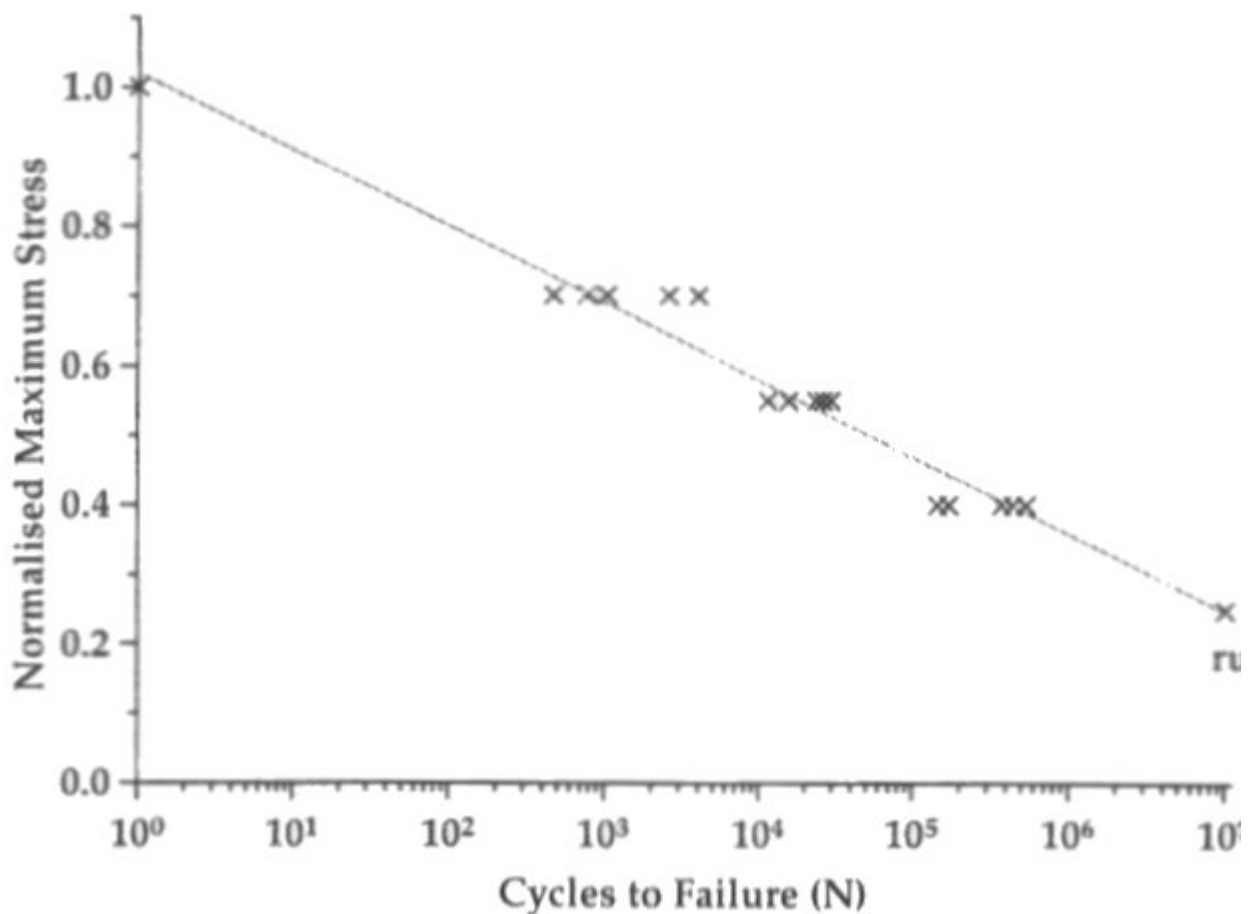


Figure 10: Normalised S-N curve for E-glass/polyester rods.

Conclusions

Test methods have been developed for measuring tensile properties of fibre tows and composite rods (≤ 3 mm diameter). It has been shown that these methods are suitable for generating basic strength-time and cyclic fatigue data for both glass and carbon fibres and their composites. The techniques can be used to determine resistance to the combined effect of water and other chemicals, elevated temperatures and stress.

The test methods require shorter conditioning times and less manufacturing and testing costs in comparison with current practices (i.e. laminate testing). Testing can also be conducted under more moderate and realistic environments, although testing of larger diameter rods would be required in order to verify the reliability of data extrapolation from small diameter composite rods to larger structures.

This Measurement Note demonstrates the sensitivity of E-glass fibre tows and glass/polyester to environmental attack. The rate of degradation is accelerated in the presence of stress and at elevated temperatures. Exposure to solutions of NaOH and H₂SO₄ with concentrations ≥ 1 N results in rapid deterioration/failure of E-glass fibres and their composites, particularly under tensile loads. In contrast, carbon fibres and their composites have excellent resistance to environmental stress corrosion.

Glass fibre and carbon fibre bundles, and continuous aligned carbon fibre-reinforced composites have excellent fatigue resistance. In comparison, the fatigue performance of glass fibre-reinforced composites is poor, although satisfactory for many industrial applications.

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