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LIFE ASSESSMENT AND PREDICTION

Environmental Degradation of Unidirectional Composites

W.R.Broughton, M.J.Lodeiro and D.R. Mulligan

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ABSTRACT

Test methods have been developed within the *Composites Performance and Design Project - Life Assessment and Prediction (CPD2)* for determining the residual strength and remnant life of glass and carbon fibre-reinforced composites exposed to aggressive environments and/or static loads. This report considers convenient methods for obtaining basic strength-time data for unconditioned and chemically conditioned fibre bundles and continuous aligned fibre-reinforced rods. These test methods require shorter conditioning times and less manufacturing and testing costs in comparison with current practices.

The report considers: (i) tensile testing of moisture and chemical (i.e. sulphuric acid and sodium hydroxide solutions) pre-conditioned fibre bundles and fibre-reinforced rods; and (ii) creep rupture behaviour of moisture and chemical conditioned fibre bundles and fibre-reinforced rods. Moisture pre-conditioning involved immersion in deionised water at temperatures ranging from 23 °C to 70 °C for up to 42 days, whereas chemical conditioning involved exposure of fibre bundles to either sulphuric acid or sodium hydroxide solutions at room-temperature for periods no longer than 2 weeks.

The principal conclusions that can be drawn from the results are: (i) fibre tows and small diameter (i.e. 1.5 mm) composite rods are suitable for accelerated testing under combined static loads and hostile environments, with fibre tows offering a relatively rapid method for evaluating fibre sensitivity to chemical attack; (ii) carbon fibres are relatively impervious to moisture, whereas E-glass fibres are highly sensitive to chemical attack; and (iii) results from narrow laminate strips cannot be extrapolated to larger coupon specimens. The report includes recommendations on preparation and testing of fibre bundles and composite rods.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,

Head of Centre for Materials Measurement and Technology.

CONTENTS

1. INTRODUCTION

This report provides an assessment of test methods developed for characterising the tensile properties of fibre bundles and impregnated strands (i.e. composite rods) under combined aggressive environments and static loading conditions. These test methods require relatively short conditioning times (i.e. suitable for accelerated testing) and relatively low manufacturing and testing costs in comparison with current practices. Tests have been carried out on both glass-fibre and carbon-fibre reinforced systems to obtain basic strength-time data for unconditioned and environmentally conditioned fibre bundles and continuous aligned fibre-reinforced rods. The research described in this report forms part of the DTI funded project *Composites Performance and Design - Life Assessment and Prediction (CPD2)* directed towards the development and validation of test methods and predictive models that can be used for characterising the long-term performance of polymer matrix composites (PMCs).

The report is divided into five sections including this section. Section 2 describes the materials used to validate the test methods in the programme, together with specimen geometry and specimen preparation. Tensile test results for moisture and chemical (i.e. sulphuric acid and sodium hydroxide solutions) pre-conditioned fibre bundles and composite rods are presented in Section 3. Section 4 considers creep rupture behaviour of fibre bundles and composite rods in aggressive environments. Conclusions are presented in Section 5. **Throughout this report, statements of particular importance or relevance are highlighted in bold type**.

2. MATERIALS CHARACTERISATION

2.1 MATERIALS DESCRIPTION

The materials used to develop and validate the test methods in this programme are listed below:

(a) E-glass (1,878 g/km or Tex) and Grafil 34-700 carbon (779 Tex) fibre tows supplied by RBJ.

(b) Pultruded glass fibre-reinforced polyester rods (glass/polyester). The composite consisted of axially aligned E-glass fibres impregnated with Scott Bader Crystic 196 orthophthalic polyester. Rods of varying diameter, ranging from 1.5 mm to 5.0 mm, were obtained from RBJ. The rods were post cured for 3 hours at 80 ^{o}C.

(c) Pultruded carbon fibre-reinforced vinylester (carbon/vinylester) rods. The composite consisted of axially aligned Grafil 34-700 carbon fibres impregnated with Norpol Dion 9100. Norpol Dion 9100 is a non-accelerated, Bisphenol-A epoxy based vinylester resin. Rods of varying diameter, ranging from 1.5 mm to 5.0 mm, were obtained from RBJ. The rods were post cured for 3 hours at 80 ^{o}C.

(d) Autoclaved continuous unidirectional glass fibre-reinforced epoxy prepreg sheet (E-glass/Fibredux F922). Laminates were manufactured at the National Physical Laboratory (NPL). The laminates were autoclaved and post-cured to Hexcel Composites specifications.

(e) Autoclaved continuous unidirectional glass fibre-reinforced epoxy prepreg sheet (E-glass/Fibredux 913). Laminates were manufactured at NPL to Hexcel Composites specifications.

(f) Autoclaved continuous unidirectional carbon fibre-reinforced epoxy prepreg sheet (Torayca T300/Fibredux 924). Laminates were manufactured (including post curing) at NPL to Hexcel Composites specifications.

On delivery, all rods and panels were visually inspected for evidence of damage or processing defects.

2.2 FIBRE CONTENT AND COMPOSITE DENSITY

Fibre volume fraction, $\mathbf{V_f}$, fibre weight fraction, $\mathbf{W_f}$, and composite density, ρ_f, were measured for all materials (Table 1). Composite density measurements were carried out using method A (zeroed pan immersion) specified in ISO 1183 [1]. Fibre volume and weight fraction measurements for the glass fibre-reinforced rods and laminates were carried out according to the ISO 1172 standard [2], which uses a resin burn-off technique. In this technique, the composite is dried to constant mass, then subjected to 600 ^{o}C in a furnace for at least an hour to remove all traces of resin. The fibre volume and weight fractions of carbon-fibre/epoxy panels and carbon/vinylester rods were determined according to BS ISO 11667 [3]. Resin removal was achieved using concentrated sulphuric acid and hydrogen peroxide. This process was carried out using a Prolabo Microdigest 401 digester.

Table 1 Composite Density, Fibre Volume Fractions and Fibre Weight Fractions

Material	Composite Density (kg/m^3)	Volume Fraction (%)	Weight Fraction (%)
E-glass/polyester	1,862 ± 21	50.56 ± 1.10	69.50 ± 0.56
Carbon/vinylester	1,484 ± 2	50.68 ± 1.15	61.49 ± 1.34
E-glass/F922	2,122 ± 53	65.41 ± 4.46	78.84 ± 3.01
E-glass/913	1,883 ± 39	50.95 ± 0.19	69.26 ± 0.26
HTA/F922	1,562 ± 17	58.64 ± 2.21	66.41 ± 1.79
T300/924	1,555 ± 10	61.49 ± 1.44	68.48 ± 1.24

2.3 SPECIMEN GEOMETRY AND PREPARATION

This section describes the test methods and specimen geometries used for testing fibre tows, composite rods and unidirectional laminates. Details of specimen preparation for each method are also covered.

2.3.1 Fibre Bundles

Tensile and cyclic fatigue (tension-tension) tests were carried out on unimpregnated E-glass and Grafil 34-700 carbon fibre tows using the specimen geometry shown in Figure 1.

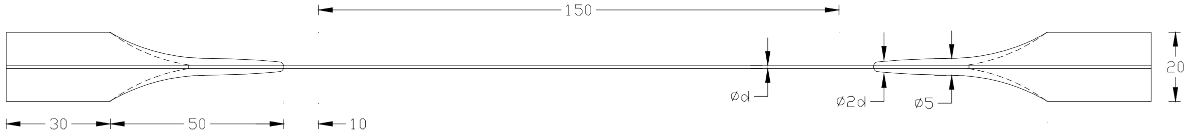

Figure 1 Schematic of fibre tow test specimen (mm).

Specimen preparation was carried out according to BS ISO 9163 [4]. A cold-curing epoxy system based on Araldite® LY 5052/hardener HY 5052 (Ciba Speciality Chemicals) was 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**. Figure 2 shows a photograph of a fibre tow specimen.

Figure 2 Fibre tow test specimen.

The end tabs were cast in silicon moulds, using BS ISO 9163 specifications as a guide, and cured at 80 °C for 20 minutes. The silicon moulds were produced using a two-part, low-shrinkage, room-temperature curing silicon elastomer. **Care was taken to ensure that the mould was free of air bubbles (degassed) and the internal surfaces of the mould were smooth and free of defects**. Moulds tend to split and break due to constant handling and therefore need to be replaced on a regular basis (5-6 castings per mould). A schematic diagram showing the dimensions of the template used for producing the moulds is presented in Figure 3.

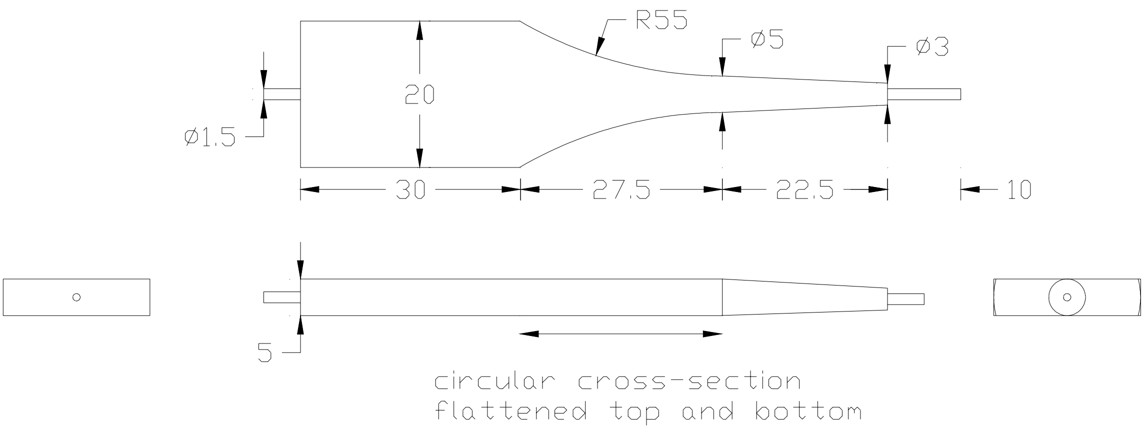

Figure 3 Schematic of moulding template used for producing end tabs (mm).

2.3.2 Composite Rods

The test geometry specified in BS ISO 9163 proved unsatisfactory for measuring the tensile strength of composite rods with diameters of 1.5 mm or greater. It was found that the interface between the composite rod and epoxy resin end tabs yielded/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 the composite rods. Figure 4 shows a schematic of the composite rod specimen and end tab used for testing glass/polyester and carbon/vinylester pultruded rods with a nominal diameter of 1.5 mm.

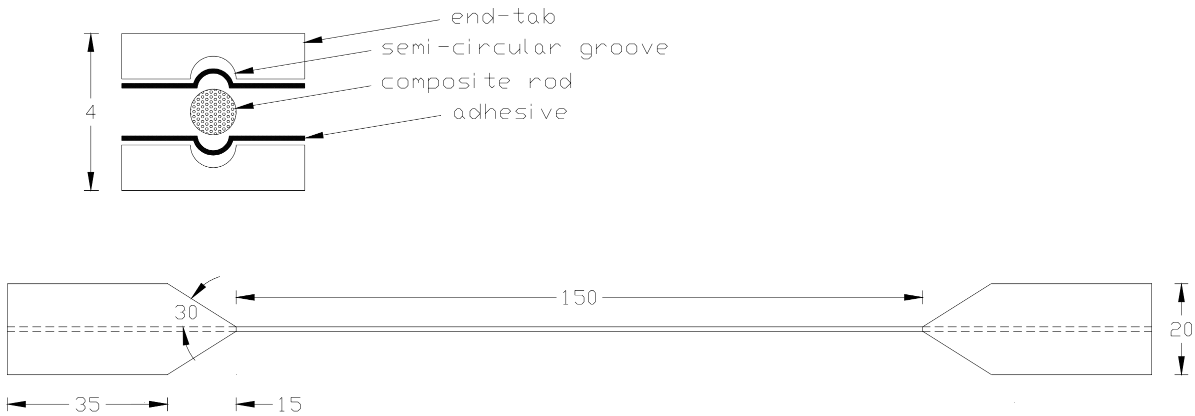

Figure 4 Schematic of composite rod specimen and end tab (mm).

The end tabs were manufactured from a plain woven glass fabric reinforced epoxy laminate (1.6 mm thick) with the fibre axes of the fabric set at ±45° to the specimen axis. A high elongation adhesive was used to bond the end tabs to the specimen. **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, reducing adhesive loss. Specimen preparation was also relatively clean in comparison to paste adhesives**. The semi-circular groove, shown in Figure 4, was cut with a 1.5 mm thick diamond slitting wheel (water lubricated). **It is essential that the end tabs are dried before bonding to remove moisture, which can compromise the adhesive bond.** The test geometry shown in Figure 5 is limited to rods with diameters of 1.5 mm or less. Details of a method for testing larger diameter rods is given in reference [5].

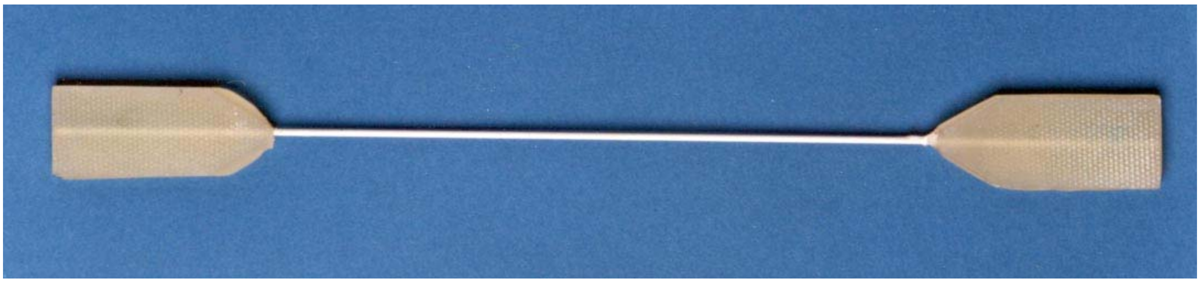

Figure 5 Composite rod specimen with bonded end tabs.

2.3.3 Unidirectional Laminates

Comparative tests were carried out on narrow (i.e. 3 mm wide) and standard (i.e. 15 mm wide) unidirectional laminates to determine the suitability of using narrow width specimens for accelerated test purposes. Specimen preparation of unidirectional laminates was carried out to BS EN ISO 527-5 [6] specifications. Standard specimens (Figure 6) were 250 mm in length, 15 mm wide and 1 mm (i.e. 8 plies) thick. The overall gauge-length (i.e. region between grips) was 150 mm. End tabs (50 mm long), manufactured from a plain woven glass fabric/epoxy laminate (1.6 mm thick) with the fibre axes of the fabric set at ±45° to the specimen axis, were adhesively bonded to the specimens. The tab angle was 90° (i.e. not tapered). An epoxy film adhesive with a cure temperature of 120 °C was used to bond the end tabs to the specimen. The narrow laminate strips were 3 mm wide and 2 mm (i.e. 16

plies) thick. Apart from the differences in width and thickness, all other aspects of specimen geometry and preparation were the same as that for the standard test specimen.

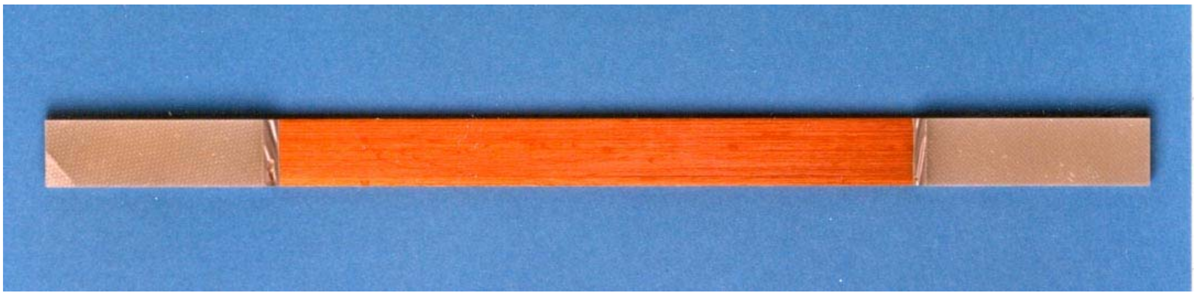

Figure 6 Continuous unidirectional glass fibre-reinforced laminate.

3. TENSILE TESTS

This section presents the results from tensile tests that were carried out to evaluate the effects of exposure to aggressive environments on the residual strength of E-glass and Graphil 34-700 fibre tows, and glass/polyester rods. Moisture pre-conditioning involved immersion in deionised water at temperatures ranging from 23 °C to 70 °C. Batches of specimens were withdrawn at selected intervals over a 3 week period for the fibre tows and over a 6 week period for the composite rods and then tested within an hour of removal from the water bath. The 6 week (≈ 1,000 hours) period was considered representative for the development of an accelerated ageing test programme. Fibre tows were also immersed in either sulphuric acid or sodium hydroxide at room-temperature for periods no longer than 2 weeks.

Testing was carried out under standard laboratory conditions (23 °C, 50% RH) at a constant displacement rate of 2 mm/min using an Instron 8501 servo-hydraulic test machine fitted with servo-hydraulic grips. Instron Series IX software was used to control the servo-hydraulic test machine and to collect the test data. Tests were conducted on at least 5 specimens per condition.

3.1 FIBRE BUNDLES

3.1.1 Water Resistance

The tensile stress at break of E-glass and Grafil 34-700 carbon fibre tows was determined using BS ISO 9163. Displacement was determined directly from the cross-head movement. The tensile results for unconditioned fibre tows are shown in Tables 2 and 3.

Table 2 Tensile Results for Unconditioned 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 3 Tensile Results for Unconditioned Grafil 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

The uncertainty in the tensile breaking stress and strain measurements for the carbon fibre tows was noticeably less than for the E-glass fibre tows. This is to be expected as glass fibres are more susceptible to damage induced through handling and moisture degradation. Two batches of glass fibre tows selected from different sections (widely separated) on the same reel, were found to have significantly different values of breaking stress and breaking strain (see Table 2). Figure 7 shows that the cumulative distribution of failure events for tests conducted on the glass and carbon fibres is essentially linear.

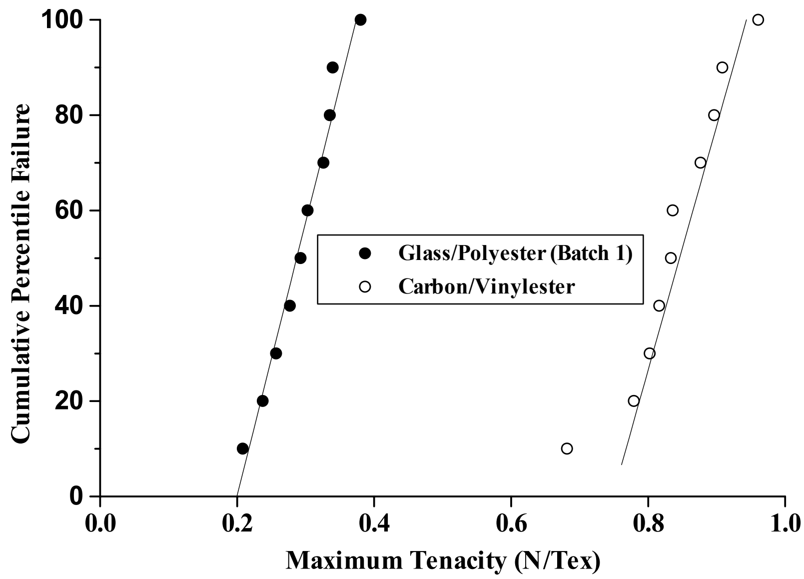

Figure 7 Cumulative distribution of failure events.

The results in Table 4 and Figure 8 (Batch 1) indicate that pre-soaking in deionised water in the absence of stress had a severe effect on the subsequent short-term strength of the E-glass fibres. The maximum load at failure, as shown in Figure 8, has been normalised with respect to the ultimate tensile load of unconditioned fibre tows (i.e. 0.31 ± 0.04 N/Tex).

Table 4 Rupture Stress (N/Tex) for Moisture Pre-Conditioned E-Glass Fibre Tows

Condition	**Pre-Conditioning Temperature (°C)**			
	23	**40**	**60**	**70**
Water immersion				
dry	0.31 ± 0.04	-	-	-
7 days (wet)	0.26 ± 0.02	0.27 ± 0.03	-	0.23 ± 0.02
14 days (wet)	0.21 ± 0.02	0.17 ± 0.01	0.18 ± 0.02	0.21 ± 0.03
14 days (dried)*	-	-	0.21 ± 0.04	-
21 days (wet)	0.16 ± 0.01	0.15 ± 0.01	-	0.13 ± 0.02

* Specimens have been immersed in water for 2 weeks then dried and tested.

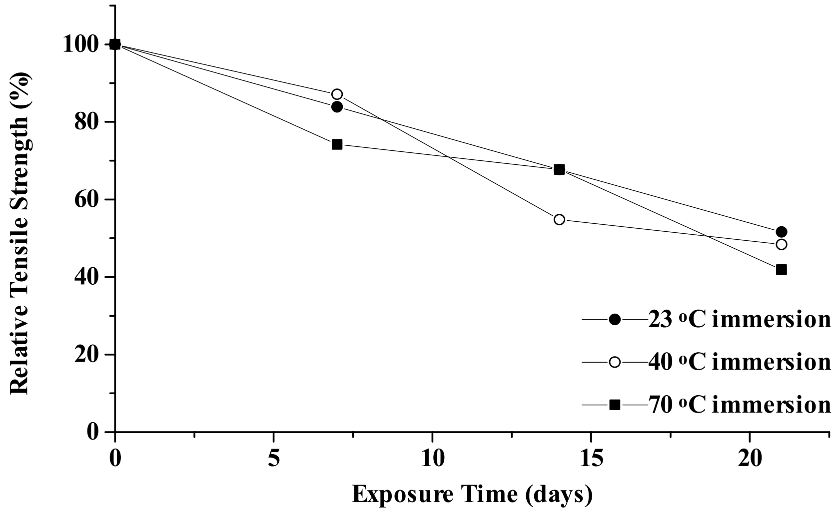

Figure 8 Residual strength for moisture pre-conditioned E-glass fibre tows.

Degradation of the E-glass fibres can be mainly attributed to leaching of alkali oxides (sodium or potassium oxide) from the fibre surface resulting in the formation of surface micro-cracks. A check was carried out to determine if the loss of tensile strength was reversible. Tensile tests were conducted on specimens that had been immersed in water at 60 °C for 2 weeks and then dried in an oven in an attempt to remove all surface moisture. Drying the fibres had no reversible effect on tensile strength (Table 4). **Loss of strength can be expected to be permanent at all conditioning temperatures and exposure times.**

A batch of specimens, which had been exposed to boiling water for 24 hrs prior to testing, showed a far greater loss of strength than that observed for specimens that had been immersed in water for 21 days at 70 °C. The maximum load at failure after 24 hours exposure in boiling water was 0.08 ± 0.02 N/Tex, a 75% reduction in strength compared with the dry samples (so-called zero values).

A number of observations can be made in relation to the environmental resistance of E-glass and carbon fibre tows exposed to the combined effect of temperature and moisture.

- Carbon fibres exhibit excellent moisture resistance and can be expected to remain unaffected by exposure to water at elevated temperatures.
- Tensile strength of E-glass fibres decreases linearly with exposure time, with a 50% reduction in strength occurring within 3 weeks.
- Degradation mechanism of E-glass fibres is temperature independent over the temperature range 23 °C to 70 °C.
- Exposure of E-glass fibres to boiling water for short periods (i.e. 24 hours) results in a dramatic loss of strength (75% strength reduction compared with dry samples).
- Tensile modulus of E-glass fibre tows remains unaffected by water immersion at elevated temperatures (i.e. 23 °C to 70 °C). A 10 % reduction in modulus was observed after 24 hours immersion in boiling water.
- Loss of strength of E-glass fibres is irreversible.

NB. Glass fibres are susceptible to damage induced through handling and moisture degradation and therefore care should be taken to avoid these problems. Glass fibres should be stored in a dry (i.e. low humidity) area.

3.1.2 Chemical Resistance

Fibre bundles were exposed to either sulphuric acid or sodium hydroxide solutions at 23 °C in an unstressed state for 2 weeks. Exposure to a 0.5 M (or 1 N) solution of sulphuric acid (1 M concentration of H^+ ions) resulted in severe corrosion of the E-glass fibres. A one normal (1 N) solution contains one gram-equivalent weight of a particular substance dissolved in 1 litre of solution. The strength retention was effectively zero after 2 weeks of immersion in the acidic solution. This reduction in strength probably occurred in 4 to 5 days, or less [7]. Immersion in a 1 M (1 N) sodium hydroxide solution (1 M concentration of OH^- ions) for the same period was far less damaging to the E-glass fibres, although more severe than that experienced when immersed in water for the same period (see Table 4). The maximum load at failure after 2 weeks exposure in the sodium hydroxide solution was 0.10 ± 0.02 N/Tex, a 68% reduction in strength compared with the dry samples. Tensile modulus was reduced by 15% in the same timescale. The pH of the solutions was measured daily and, although there was some variation, these were within the expected scatter of measurements.

Note: The results presented in Section 3.1 clearly demonstrate that tensile testing of fibre tows offers a relatively quick and cost effective means of evaluating fibre sensitivity to chemical attack.

3.2 COMPOSITE RODS

A series of tensile tests were conducted on moisture pre-conditioned 1.5 mm diameter glass/polyester rods according to BS ISO 9163. The results, shown in Table 5 and Figure 9, indicate that, without applied stress, the rate of reduction in tensile strength of the glass/polyester rods increases with pre-conditioning temperature. The tensile strength, as shown in Figure 9, has been normalised with respect to the ultimate tensile strength of unconditioned (i.e. dry) fibre tows measured at the same displacement rate (i.e. 1057 ± 16 MPa). The tensile modulus was found to be constant with temperature and exposure time.

Table 5 Tensile Strength (MPa) for Moisture Conditioned Glass/Polyester Rods

Exposure Time (days)	Pre-Conditioning Temperature (°C)			
	23	40	60	70
0	1057 ± 16	-	-	-
7	958 ± 10	914 ± 21	629 ± 9	574 ± 79
14	869 ± 9	722 ± 82	524 ± 25	523 ± 12
21	798 ± 51	656 ± 24	460 ± 12	496 ± 24
42	751 ± 26	538 ± 19	468 ± 27	478 ± 27

The glass transition temperature, $\mathbf{T_g}$, of the Crystic 196 polyester resin decreases with increasing moisture content, with the rate of reduction of $\mathbf{T_g}$ increasing as the conditioning temperature is elevated (Figure 10). The glass-transition temperature of the polyester resin decreases by approximately 9 °C for each 1 wt% of moisture gain. **Glass/polyester systems, as with all PMCs, should not be subjected to temperatures in excess of $\mathbf{T_g}$, nor should glass/polyester structures be immersed in water or exposed to humid environments for prolonged periods without suitable protection (e.g. resin gel coat).** The maximum operating temperature often recommended is ($\mathbf{T_g}$ - 20 °C), which for the fully conditioned glass/polyester system is close to room-temperature.

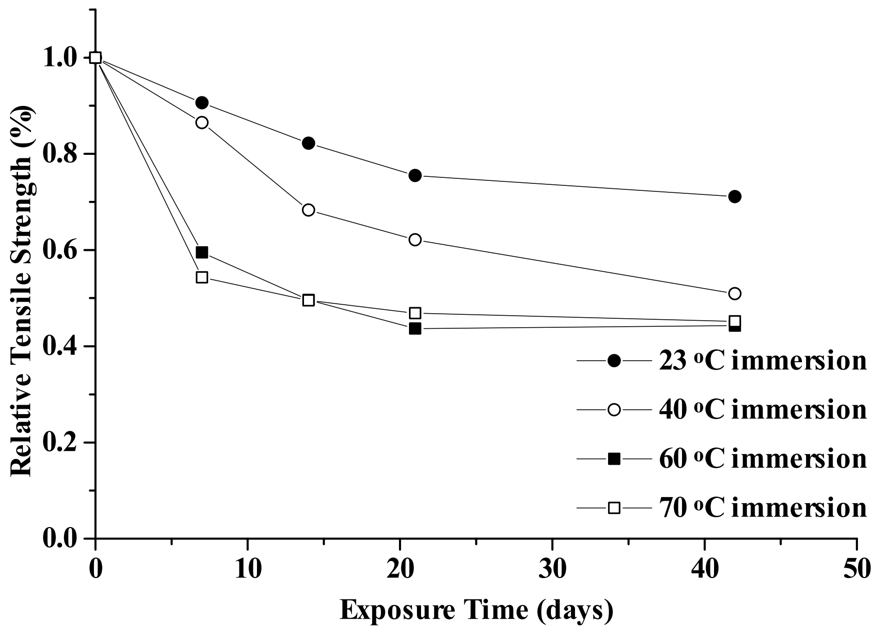

Figure 9 Residual strength for moisture pre-conditioned E-glass/polyester rods.

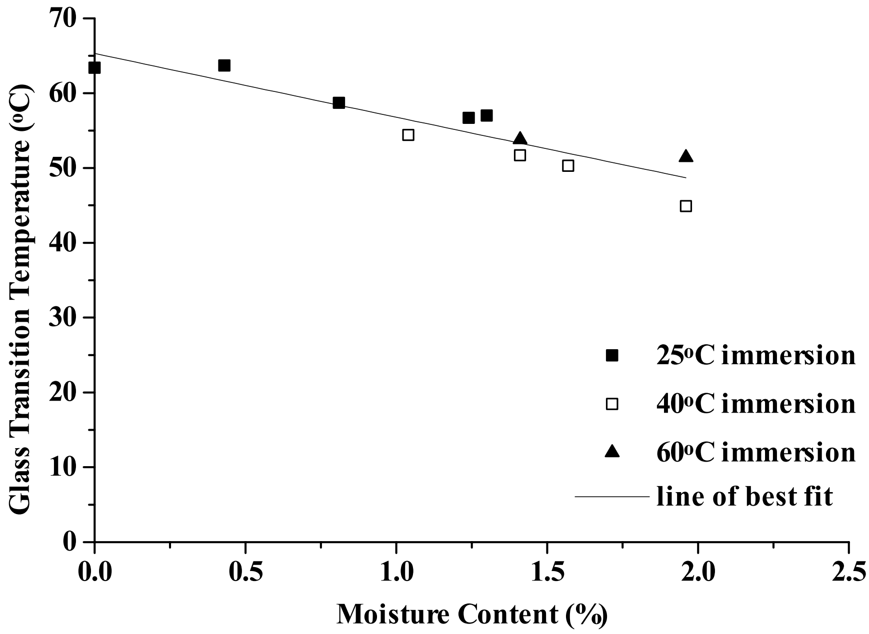

Figure 10 Glass transition temperature for moisture conditioned Crystic 196 polyester.

Note: The results demonstrate that small diameter rods (e.g. 1.5 mm) enable rapid uptake of aggressive agents, with any degradation effects on the composite being quickly manifested (within 7 days) in terms of strength reduction.

4. STATIC FATIGUE

This section presents results from static fatigue tests that were carried out on E-glass and carbon fibre tows, glass/polyester and carbon/vinylester rods, and unidirectional E-glass/F922 laminates (3 mm and 15 mm wide). The test results presented in this report are confined to ambient conditions only. Tests were conducted to evaluate the combined effect of environment and applied static tensile stress. 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 described in Section 3.1.2. Figure 11 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.**

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 manually adjust the 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**.

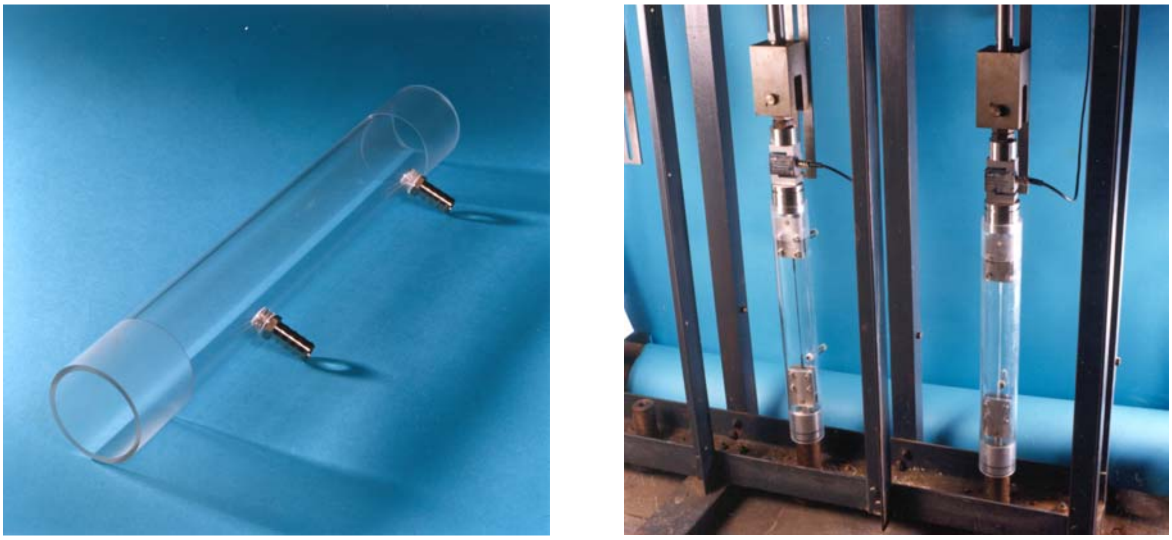

Figure 11 Long-term environmental fatigue apparatus.

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 12):

$$\sigma_{APP} / \sigma_{UTS} = 1 - k\log t_f \qquad (1)$$

where $\boldsymbol{\sigma_{APP}}$ is the applied load (or stress), $\boldsymbol{\sigma_{UTS}}$ is the maximum short-term strength of the unconditioned material, **k** is the slope and $\mathbf{t_f}$ 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.

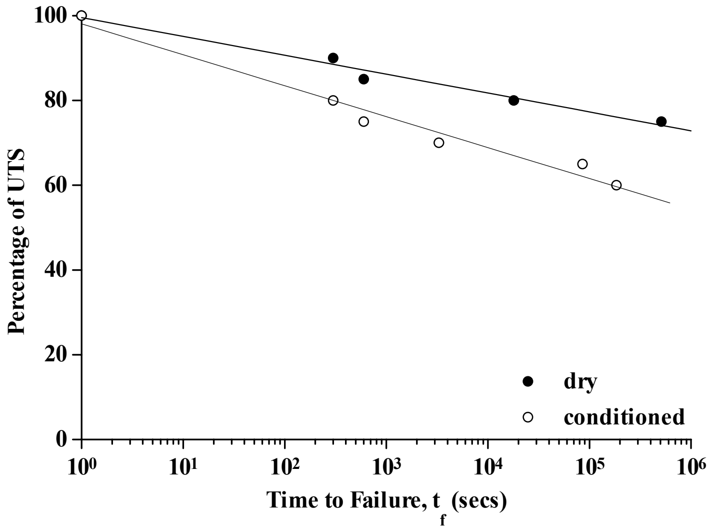

Figure 12 Stress rupture of E-glass fibres in air and water.

The rate of strength reduction of the E-glass fibre tows (Figure 12) in water (25% strength loss in less than an hour) was relatively high compared with air (25% strength loss in 5.5 days). In contrast, carbon fibres can maintain static loads approaching the UTS of the fibre (~90% UTS) indefinitely in both air and water.

Glass/polyester rods (Figure 13) and the 15 mm wide aligned E-glass/F922 specimens (Figure 14) are capable of sustaining a static load equivalent to 50% UTS under ambient conditions for at least 4 months. The resin matrix in each case acts as a chemical barrier to the corrosive effects of moisture/water on the glass fibres. The environmental moisture gains access to the glass fibres via diffusion or surface cracks. **It is advisable in manufacturing test specimens that a resin rich layer is present on the top and bottom surfaces of the specimen and that this layer is not breached, thus exposing the underlying fibres. Edge damage is unavoidable during machining and will provide a path for environment ingress.**

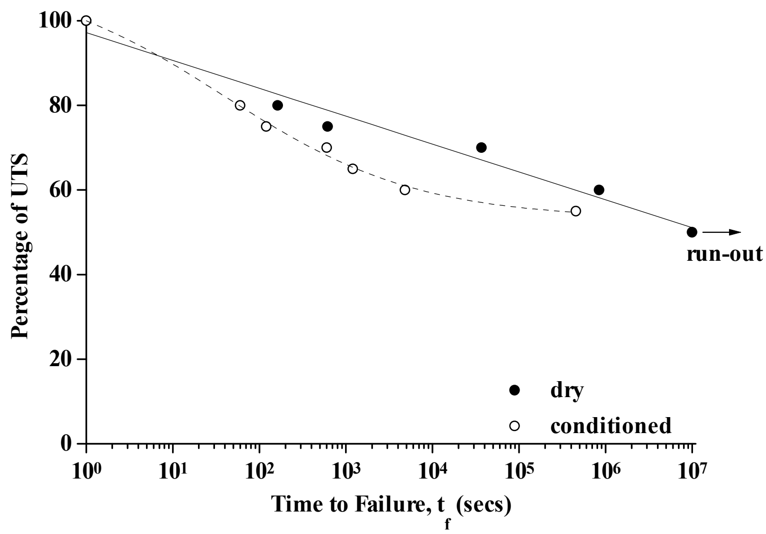

Figure 13 Stress rupture of E-glass fibres impregnated with polyester resin in air and water.

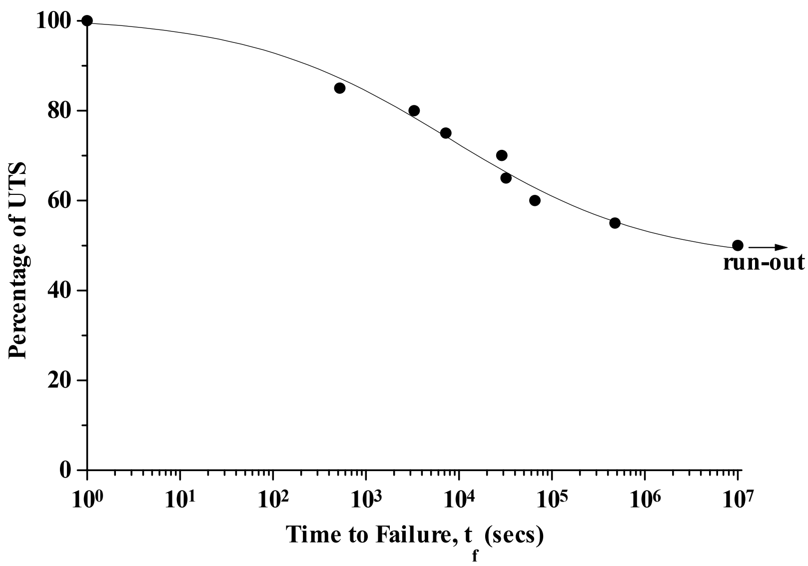

Figure 14 Stress rupture of 15 mm wide unidirectional E-glass/F922 laminate in air.

The normalised stress rupture curves for the E-glass/polyester rods (Figure 13) and 3 mm wide E-glass/F922 laminated strips (Figure 15) in air when plotted on a linear-log plot are essentially linear. The relationship between normalised applied stress and time to failure can be described by Equation (1) where the slope **k** of the line of best fit equals 0.069 and 0.090 for the two materials, respectively. The stress rupture curves of the 15 mm wide E-glass/F922 specimens in air and the E-glass/polyester rods in water were essentially "S-shaped" (sigmoidial) as shown in Figures 13 and 14. Similar curves have been observed by Aveston and Sillwood [8] for stress rupture of unidirectional glass fibre-reinforced laminates subjected to tensile loads whilst immersed in either distilled water or 1 N sulphuric acid solution. **The carbon/vinylester rods exhibit far superior environmental resistance than the glass/polyester material (time to failure > 12 days for 70% UTS).**

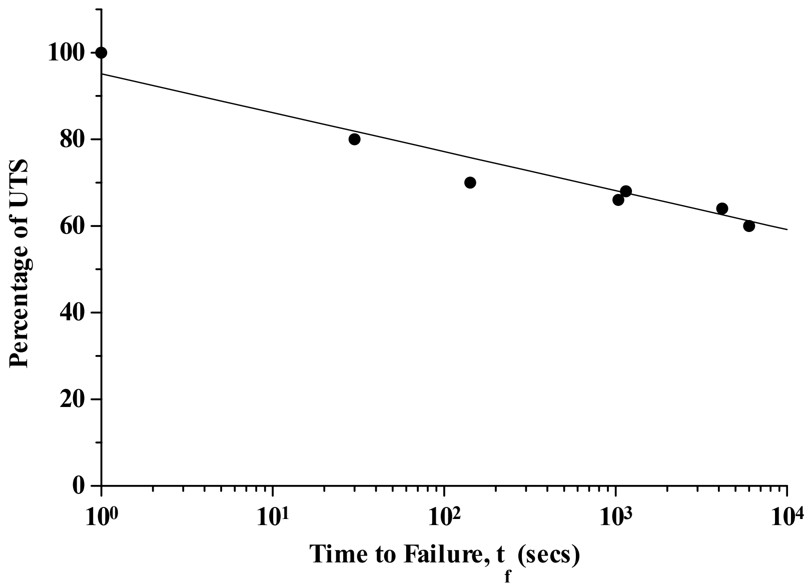

Figure 15 Stress rupture of 3 mm wide unidirectional E-glass/F922 laminate in air.

The reduction in strength of the 15 mm wide E-glass/F922 specimens was slower than that observed for the 3 mm wide specimens. Differences may be attributed to a number of factors including:

(i) A higher moisture content in the 3mm wide specimens compared with the wider specimens for the same exposure time;
(ii) Edge effects (including damage through machining) have a larger effect on the narrow specimens; and
(iii) A limited ability to redistribute load as a result of fibre breakage and matrix and interfacial cracking in smaller specimens.

The results for narrow (i.e. 3 mm wide) unidirectional E-glass/F922 (Figure 15) indicated that these specimens were less robust. Efforts were made to minimise the possibility of machine induced damage at the specimen edges affecting the strength results, particularly for the narrow strips. Although the narrow specimens were cut oversized and then polished to the final width, damage generally initiated at the edges. **The results from the 3 mm wide E-glass/F922 strips cannot be extrapolated to larger coupon specimens, whereas the results from the glass/polyester rods doe not preclude this possibility.**

A combination of sustained tensile load and exposure to sodium hydroxide or sulphuric acid solutions resulted in rapid failure of E-glass fibre tows. Exposure to 1N sulphuric acid solution proved too deleterious to enable sufficient time for testing (i.e. data collection), even at moderate loads (50-60% UTS). Failure was almost spontaneous when the fibre tows were subjected to combined load and the acidic solution. Two methods, both of which proved unsuccessful, were tried in an attempt to test the E-glass fibres:

(i) Specimens were first loaded and then the chemical agent was introduced.
(ii) Specimens were exposed to the chemical agent and then loaded.

Note: Method (i) is the preferred method.

The situation was slightly better for stress rupture tests in the presence of 1 N sodium hydroxide (see Table 6). Short failure times were observed for applied loads as low as 45% UTS (~3 hours). Specimens exposed to loads in excess of 65% UTS were observed to fail within 1 to 2 minutes, insufficient time to stabilise load. Time-to-failure at stress levels lower than 45% UTS were similar to that observed for tests conducted in the range 45 to 55%. UTS.

Table 6 Stress rupture of E-glass fibres 1 N sodium hydroxide solution

% Ultimate Tensile Strength	Time-to-Failure (secs)
1	1
65	1,800
60	6,000
55	5,880
50	7,260
45	11,640

Glass fibre strands stress rupture under static loading conditions, whereas in the case of the composite rods and unidirectional laminates, failure was non-progressive (i.e. catastrophic) with failure consistently occurring within the gauge-length, although often near the end tabs. There was little indication on the load-displacement response that failure was imminent. Figure 16 shows a typical tensile failure for a glass/polyester rod specimen with fibre breakage and longitudinal splitting. This failure mode was observed for both static and cyclic fatigue loading.

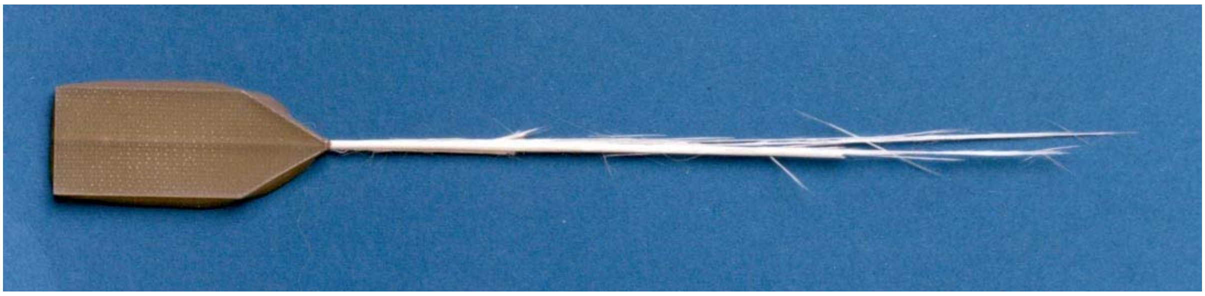

Figure 16 An example of static fatigue failure for a glass/polyester rod specimen.

The results of both the ambient and environmental exposure tests are to be compared with predictive analysis, which is being carried out in parallel with the test programme.

5. DISCUSSION AND CONCLUSIONS

Test methods have been developed for measuring the tensile properties of fibre tows and composite rods (≤ 3 mm diameter). E-glass fibre strands (i.e. bundles or tows) were sensitive to environmental attack from moisture/water, sulphuric acid and sodium hydroxide solutions. In contrast, carbon fibres were impervious to all three chemical agents even under tensile loading conditions. Immersion of E-glass fibres in either water, sulphuric acid or sodium hydroxide results in the loss of long-term strength. The ability to withstand continuous loads falls steadily when immersed in water, whereas failure times were far shorter in 1N solutions of sulphuric acid and sodium hydroxide. The E-glass fibres were particularly sensitive to sulphuric acid. The rate of degradation was considerably slowed by impregnating the E-glass fibres with either polyester or epoxy resin. A number of key points that have arisen from this work are summarised below.

Fibre Tows

- Carbon fibres exhibit excellent chemical 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 all three chemical agents investigated. The degradation process is accelerated under combined load and aggressive environment.
- E-glass fibres are susceptible to moisture degradation and therefore care should be taken to store fibre tows in a dry (i.e. low humidity) area. Tensile strength decreases steadily with exposure time in water (50% reduction within 3 weeks).
- Degradation mechanism of E-glass fibres due to water immersion is essentially temperature independent over the temperature range 23 °C to 70 °C. Loss of fibre strength is irreversible.
- Tensile modulus of E-glass fibre tows remains unaffected by water immersion at elevated temperatures (i.e. 23 °C to 70 °C). A 10 % reduction in modulus was observed after 24 hours immersion in boiling water.
- Fibre tows are suitable for accelerated testing under combined static loads and hostile environments, offering a quick and cost effective means of evaluating fibre sensitivity to chemical attack.

Composite Rods and Narrow Strips

- Small diameter (i.e. 1.5 mm) composite rods are suitable for accelerated testing under combined static loads and hostile environments. It may be possible to extrapolate static fatigue data to larger coupon specimens and to longer times (possibly a further decade of time).
- The data obtained from composite rods are suitable for comparative material studies, but not for engineering design purposes.
- Results from the 3 mm wide E-glass/F922 strips cannot be extrapolated to larger coupon specimens.

General

- The normalised stress rupture curves for E-glass fibre tows in air and water when plotted on a linear-log plot can be approximated by the equation (Equation (1)):

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

- The normalised stress rupture curve for both the 15 mm wide E-glass/F922 specimens in air and the E-glass/polyester rods in water was essentially "S-shaped" (sigmoidial). A straight line "best fit" could be used as a first approximation.
- Current standard (Part 10 of ISO 3597 [9]) for preparation and testing of composite rods requires revision to accommodate the determination of tensile strength.

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