



# TOWARDS ACCELERATED AGEING PROTOCOLS FOR SERVICE IN HOSTILE CONDITIONS

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## Abstract

*This paper examines the combined effect of stress, temperature and chemical exposure on the mechanical and physical properties of polymer matrix composites. It considers a number of accelerated ageing regimes, including artificial weathering, and exposure to aqueous and alkali environments at ambient and elevated temperatures. The paper considers both continuous and intermittent exposure testing. Mechanical and physical properties considered include residual stiffness and strength, life expectancy, moisture content, glass transition temperature and surface reflectance and hardness. The results clearly demonstrate that suitable non-invasive test methods are available that can provide reliable and accurate quantitative data relating the degree of surface degradation to mechanical performance. These test methods make it possible to produce measurable criteria for performance testing for determination of chemical resistance of composite materials. Analytical and semi-empirical relationships can be also used to determine the degree of material property degradation with the level of degrading agents.*

## 1 Introduction

Polymer matrix composites (PMCs) are increasingly being used in a wide range of engineering applications where long-term service in hostile environments (e.g. gas pipelines, chemical storage vessels, bridges and off-shore installations) is required. As a consequence, there is growing demand for manufacturers to guarantee the life expectancy of their products, particularly where inspection and/or maintenance can be difficult or failure catastrophic. Effective use of composite structures for combined stress, thermal and chemical

environments hinges on the availability of validated data applicable for the entire service life of the structure, which may be 20 to 50 years, or longer. User companies are seeking to gain increased confidence in the use of PMCs through better testing and predictive methodologies, with a consequently increased demand for data and support information from the material producers.

Currently, there is concern regarding the relationship of the results of accelerated ageing tests for PMCs and the actual service performance. There are few test methods or standards that predict life expectancy with confidence. For example, the drafting of Part 4 of the new pultrusion specification standard BS EN 13706 [1], by working group CEN TC249/SC2, is in abeyance due to the problems of identifying the linkage between test data and guaranteed life in order to provide mandated minimum requirements as in Part 3 for short-term properties.

Whilst mechanical properties and dimensional stability are quantitatively measurable quantities, a number of the appearance criteria (i.e. colour, gloss, crazing, fibre prominence, blister formation, loss of surface resin, etc) tend to be assessed in qualitative terms, and hence the question arises as to the reliability of the assessment [2]. A major challenge is to ensure that performance testing for determination of chemical resistance (level of degradation) is based upon a set of "quantitatively" measurable criteria; avoiding qualitative or subjective assessment.

This paper presents experimental results and observations from a number of case studies conducted on PMCs under different environmental conditions including artificial weathering, continuous and intermittent immersion in alkali solution at ambient and elevated temperatures, and continuous exposure to hot/humid environments (including steam autoclave).

The paper considers the combined effect of stress, thermal and chemical exposure on bulk and surface properties of PMCs. Mechanical and physical properties considered include residual stiffness and strength, life expectancy, moisture content, glass transition temperature, surface reflectance and hardness. The results are interpreted using a number of analytical and semi-empirical relationships in order to determine the degree of material property degradation with the level of degrading agents.

## 2 Artificial Weathering

Accelerated weathering procedures [3-4] generally involve cyclic exposure to a combination of salt spray, elevated and/or sub-zero temperatures, and ultraviolet (UV) radiation. This section presents the results of 6 months exposure of glass fibre-reinforced polyester rectangular beam sections to artificial weathering. Mechanical and physical measurements were conducted on composite rods after 0, 1, 2, 3, 4 and 6 months exposure in order to assess surface and bulk property degradation. Surface and bulk properties measured include:

- Barcol hardness
- Gloss
- Spectral reflectance (colour)
- Glass transition temperature ( $T_g$ )
- Moisture content (wt %)
- Flexural properties

Accelerated weathering was performed using rectangular composite rods 8 mm wide, 9 mm thick (nominal) and 230 mm long cut from a pultruded glass fibre-reinforced polyester enclosure curved panel section. The composite panels, supplied to the project by Fibreforce Composites Limited, were identical to the material used in a footbridge cladding system. The composite laminate contains several layers of 0°/90° stitched mat and unidirectional roving of varying areal weight with a surface veil on the top and bottom top surfaces. Solids (i.e. glass fibre and mineral filler) content: was  $72 \pm 1$  wt% (measured by burn-off using ISO 1172 [5]).

Accelerated weathering was conducted at RAPRA in accordance with ISO 20340 [6]. This standard combines French AFNOR (NFT 34-600) and Norsok standards [3]. ISO 20340 consists of the following stages:

- 72 hrs exposure to UV radiation and water in accordance to ISO 11507 [7]. Alternating between:

- 4 hrs exposure to UV (UVA 340 nm) at 60 °C; and
- 4 hrs exposure to moisture condensation at 50 °C
- 72 hrs exposure to salt spray at 35 °C in accordance with ISO 7253 [8]
- 24 hrs exposure at -20 °C

Standard exposure procedures generally exclude the effect of sub-zero temperatures often experienced in practice or in external exposure testing. ISO 20340 was selected because it includes a freeze cycle in an attempt to produce more realistic results (see [3]). In order to simulate actual topside service conditions, only one surface was exposed to salt spray and UV radiation. Surface measurements were restricted to exposed surfaces (i.e. topside).

### 2.1 Performance Testing Results

This section presents the results of performance testing conducted on the conditioned specimens to determine the degree of degradation with exposure time.

#### 2.1.1 Moisture Content and $T_g$

The moisture content (wt%) was measured before and after artificially weathering (see Table 1). The moisture content of the “as-received” material was approximately 0.23 wt% - equivalent to approximately one month of artificially weathering. Prior to artificially weathering, the rods were dried to a constant weight in an oven at 50 °C.

Table 1. Moisture content and  $T_g$  for accelerated weathered E-glass/polyester

Material	Moisture Content (wt %)	$T_g$ (°C)
As-received	$0.23 \pm 0.01$	94.7
Dry	0	97.0
<u>Exposure (Months)</u>		
1	$0.27 \pm 0.02$	92.2
2	$0.34 \pm 0.04$	95.5
3	$0.56 \pm 0.04$	92.3
4	$0.39 \pm 0.10$	95.2
6	$0.54 \pm 0.02$	96.3

The moisture content reached an equilibrium level of ~0.55 wt%. Variability in moisture content was most probably a result of partial drying occurring during transportation of samples to NPL.  $T_g$  remains relatively constant with moisture content.

Glass fibre-reinforced polyester systems when fully immersed in water at room temperature, or higher, has been observed to absorb far higher levels of moisture within the same timescale [9]. The increase in moisture content is also generally commensurate with a reduction in  $T_g$ . The results shown in Table 1 may be indicative of moisture being mainly limited to the outer layers of the composite.

2.1.1 Barcol Hardness

The Barcol hardness was measured at ten locations on the surface of unconditioned material and on the topside surface of the weathered specimens. Measurements were carried out using a Colman GYZJ 934-1 hand-held portable hardness tester. The results shown in Fig. 1 indicate that hardness decreases with exposure time; asymptotically approaching a constant value after 3 - 6 months (see also Table 2).

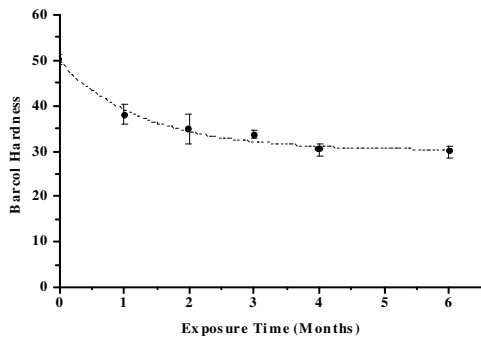


Fig. 1. Barcol hardness versus exposure time

2.1.3 Gloss

A Novo-Gloss meter was used to measure surface reflectivity of the weathered and “as-received” materials at a fixed angle of 60°. This instrument projects a collimated beam of white light (filtered to give a spectrum response similar to that of the human eye) onto the target surface at a specific angle and measures the amount of specular reflected light. Surface degradation (e.g. micro-cracking, loss of surface resin and fibre prominence, etc.) causes the incident light to be scattered at other angles, such that the scatter increases with the level of degradation. Table 2 presents the 60° angle gloss measurements for different periods of artificial weathering. The gloss measurements clearly show a strong relationship between gloss and exposure time (Fig. 2).

Gloss decreases rapidly with exposure time. After 6 months of artificial weathering, gloss has been reduced by a factor of almost 10.

Table 2. Barcol hardness and gloss measurements for accelerated weathered E-glass/polyester

Material	Barcol Hardness	Gloss (%)
As-received	50.0 ± 1.0	22.1 ± 0.1
<u>Exposure (Months)</u>		
1	38.0 ± 2.1	17.8 ± 0.4
2	34.8 ± 3.1	9.7 ± 0.1
3	33.6 ± 0.9	7.1 ± 0.3
4	30.3 ± 1.4	3.5 ± 0.3
6	29.8 ± 1.5	2.6 ± 0.1

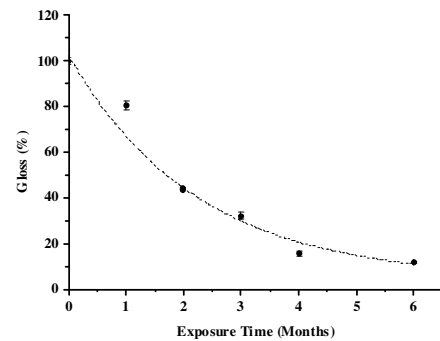


Fig. 2. Gloss versus exposure time

2.1.4 Colorimetry

The spectral reflectance of conditioned and “as-received” materials was measured using a Datalog Spectraflash 500 spectrophotometer. The spectrophotometer exposes a 10 mm diameter circular area on the surface to a light source with a daylight colour temperature and compares the percentage reflectance within the visible spectrum (360-750 nm wavelength) to that of reference white and black colour tiles [10].

The reflectance of the sample was measured at 1 nm wavelength intervals over the spectral range 380 nm to 780 nm. Two sets of measurements were made on separate occasions on the test specimens and the average recorded. The degree of reflectance decreases with exposure time (Fig. 3), mirroring the changes in gloss. Fig. 4 shows a normalised plot of total reflectance over the spectral range 380 nm to 780 nm. The total reflectance data for each specimen (i.e. exposure time) was normalised with respect to the total reflectance data obtained for the unconditioned material.

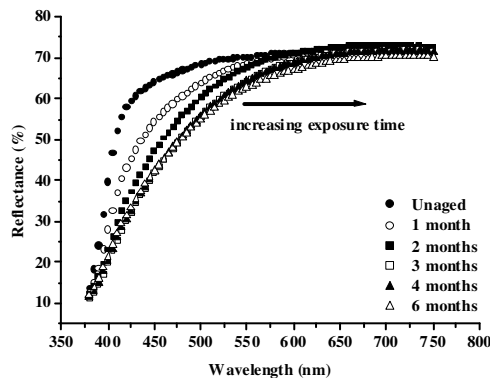


Fig. 3. Spectral reflectance versus exposure time

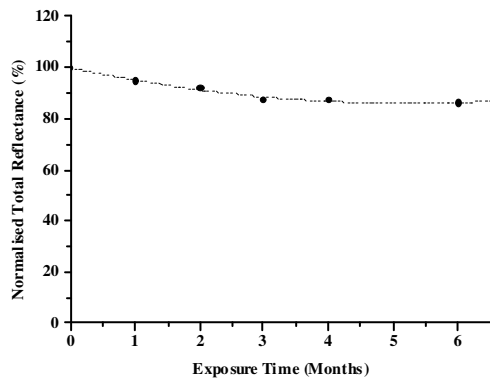


Fig. 4. Total reflectance of artificially weathered specimens normalised with respect to the “as-received” material

The spectral reflectance results presented in Fig. 3 also show that the composite discolours as a result of artificial weathering. The discolouration (i.e. yellowing) observed with exposure is due to a reduction in spectral reflectance over the spectral range 380 nm to 600 nm (i.e. blue component), which can be mainly attributed to exposure to UV radiation (UVA 340 nm).

### 2.1.5 Flexural Properties

Four-point flexure tests were carried out on the unconditioned and artificially weathered composite rods, which were 8 mm wide, 9 mm thick (nominal) and 230 mm long. Although non-standard specimen geometry was employed, testing generally conformed to BS EN ISO 14125 [11].

The geometry was selected for convenience of conditioning and to enable direct comparison with other flexure data obtained for GRP pultruded rods of similar dimensions exposed to deionised water and alkali solution. The specimens were tested using an Instron 4507 screw-driven test machine at a crosshead displacement rate of 5 mm/min under standard laboratory conditions (23 °C and 50% relative humidity (RH)). Load and displacement were recorded during the tests. The inner and outer span lengths were 100 mm and 200 mm, respectively. The support and loading rollers had a diameter of 10 mm. Displacement at the beam mid-section was measured using a linear voltage displacement transducer (LVDT).

Four-point flexure modulus and strength data measured for the “as-received”, dried and artificially weathered material is presented in Table 3. Additional flexural tests were conducted on 15 mm wide specimens (as specified in BS EN ISO 14125) for the “dry” material in order to check that edge effects were minimal for the narrow beam specimens (i.e. no reduction in flexural properties).

Table 3. Flexural properties of accelerated weathered E-glass/polyester

Material	Modulus (GPa)	Strength (MPa)
As-received	22.5 ± 0.5	442 ± 23
Dry	23.8 ± 0.5	490 ± 11
<u>Exposure (Months)</u>		
1	23.3 ± 1.3	416 ± 38
2	22.7 ± 1.3	404 ± 56
3	21.7 ± 1.3	377 ± 23
4	22.1 ± 1.2	377 ± 24
6	21.3 ± 0.8	371 ± 35

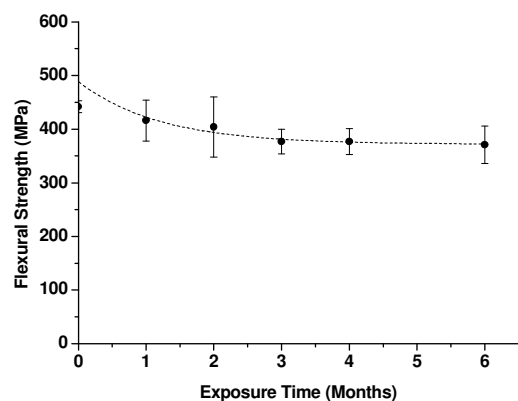


Fig. 5. Flexural strength versus exposure time

The flexure modulus remains relatively unaffected by artificial weathering. In contrast, flexure strength decreases with exposure time as shown in Fig. 5. Tensile initiated failure was observed to occur for the majority of flexure tests. A decrease in flexural strength with exposure time is to be expected as any degradation involving the outer layers of the composite will have an amplified effect on load-bearing capacity under flexural loading conditions. The influence of layers on flexural strength increases proportionally to the cube of the distance of the layer from the neutral axis of the beam.

The reduction in flexural strength is commensurate with the reduction in surface hardness, gloss retention and spectral reflectance observed. The results shown in Table 3 also indicate that the flexural strength decreases with increasing moisture content. The question arises as to the use of non-destructive surface measurements, such as Barcol hardness, gloss retention and spectral reflectance, as possible indicators of mechanical property (i.e. flexural strength) reduction.

It is possible to estimate the flexural strength of the composite based on reflectance data. Fig. 6 shows that flexural strength tends to decrease linearly with a reduction in normalised total reflectance (increased degradation). The curve incorporates flexural strength and reflectance data for the “as-received” material and not the “dry” material. The correlation coefficient for the linear curve fit to the flexural strength and normalised total reflectance is 0.9985. Linear relationships can also be derived between flexural strength and both gloss and surface hardness.

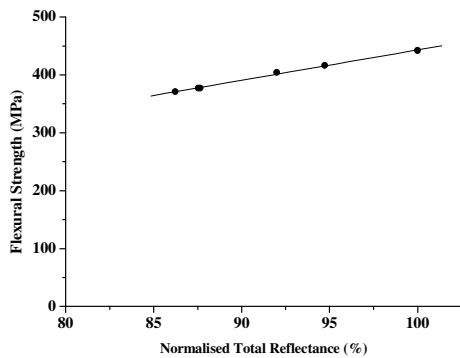


Fig. 6. Flexural strength versus normalised total reflectance total reflectance for weathered and “as-received” material (— linear curve fit)

### 3 Immersion Tests

#### 3.1.1 Continuous Exposure to Moisture

Unidirectional E-glass/Fibredux 913 and T300/924 (Torayca carbon fibre/Fibredux 924) epoxy specimens were conditioned by immersion in deionised water at a temperature of 60 °C. The specimens were 2 mm thick. Dynamic mechanical analysis (DMA) measurements were performed on dry and aged specimens to determine the changes in  $T_g$  as a function of moisture content (Fig. 7).

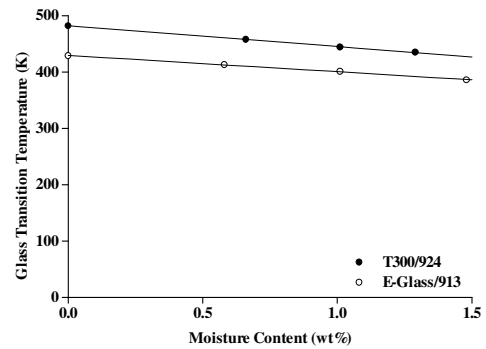


Fig. 7.  $T_g$  versus moisture content for hygrothermal aged unidirectional E-glass/913 and T300/924

The reduction in  $T_g$  for the two materials can be determined by the following relationship:

$$T_{gw} = T_{gd} - gM \quad (1)$$

$T_{gd}$  is the glass transition temperature of the dry material,  $T_{gw}$  is the glass transition temperature of the conditioned (or wet) material,  $g$  is the temperature shift (in K) per unit moisture absorbed and  $M$  is the amount of moisture absorbed (wt %). The value of  $g$  is 28.9 K and 36.8 K for E-glass/913 and T300/924, respectively. The corresponding values of  $T_{gd}$  are 430 K and 482 K.

Tests were also conducted to measure the diffusivity of T300/924 immersed in deionised water at 25, 40 and 60 °C for up to 216 days. Moisture absorption results for T300/924, shown in Fig. 8, obey Ficks law (saturation level is ~ 1.5 wt %). The diffusivity  $D$  is a function of absolute temperature  $T$  and is given by the Arrhenius relation (Fig. 9):

$$D = D_o \exp^{-E/RT} \quad (2)$$

$D_o$  is a constant,  $E$  is the activation energy of diffusion and  $R$  is the ideal gas constant. For T300/924,  $D_o$  is 0.327 mm<sup>2</sup>s<sup>-1</sup> and  $E/R$  is 4,669 K.

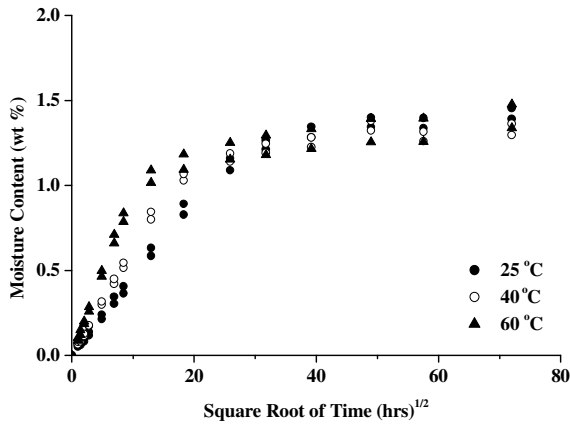


Fig. 8. Moisture absorption in T300/924

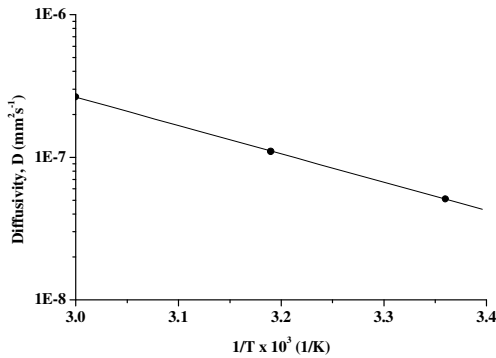


Fig. 9. Diffusivity versus temperature for T300/924

Four-point flexure tests were also conducted in accordance with BS EN ISO 14125 on moisture conditioned E-glass/913 and T300/924 specimens. The specimens were exposed for periods up to 6 weeks in deionised water at 23 °C and tested at temperatures ranging from 23 °C to 200 °C. Eq. 3 [12] given below was found to be applicable to the transverse flexure data (strength and stiffness) for both materials.

$$\frac{P}{P_o} = \left( \frac{T_{gw} - T}{T_{gd} - T_o} \right)^n \quad (3)$$

$P$  denotes the material property (usually strength) at the test temperature  $T$  and  $P_o$  is the initial property (un-aged) value of the dry material at room or reference temperature  $T_o$ . The exponent  $n$  is a constant empirically derived from experimental data. The relationship only provides a rational solution when  $T_{gd} > T_o$  and  $T_{gw} > T$ . The exponent  $n$  was estimated to have a value of 1 for E-glass/913 and a value of 0.3 for T300/924.

It was not possible to obtain consistent values of  $n$  for the longitudinal flexure properties – understandable as Eq. 3 was originally intended for use in estimating hygrothermally degraded properties of the resin matrix.

Unidirectional E-glass/polyester rods (1.5 mm diameter) were also immersed in deionised water at three temperatures (i.e. 25, 40 and 60 °C) and then tested to determine diffusivity and residual tensile strength as a function of immersion temperature. The tensile tests were carried out at room temperature in accordance with BS EN ISO 527-5 [13]. The calculated values of  $D_o$  and  $E/R$  were  $0.056 \text{ mm}^2\text{s}^{-1}$  and  $-2,300 \text{ K}$ , respectively.

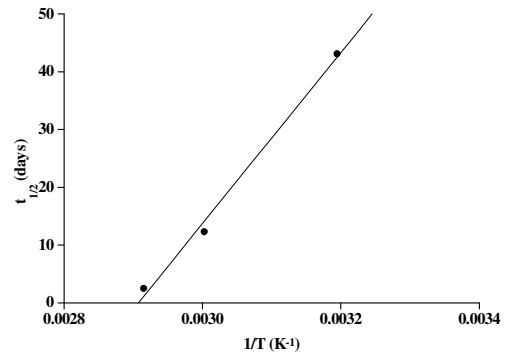


Fig. 10. Tensile strength (half-life) versus temperature for E-glass/polyester

The time required for the tensile strength to degrade to half its original value (half-life) at each temperature was calculated for the glass polyester rods using (see Fig. 10):

$$\ln t_{1/2} = A + k/T \quad (4)$$

$A$  and  $k$  are material constants determined experimentally, and  $T$  is the ageing (or conditioning) temperature. The estimated values for  $A$  and  $k$  were  $-430$  and  $1.48 \times 10^5$ , respectively.

### 3.1.2 Continuous Exposure to Alkali Solution

GRP pultruded 8 mm square bar (Eurocrete®) glass/vinylester, NFR (supplied by Fibreforce Composites Limited) were exposed to conditions representative of porewater in Portland cement for testing GRP rebars for concrete reinforcement [14]. The chemical requirements of the test solution are:

- $\text{Ca}(\text{OH})_2$  – 118.5 g
- $\text{NaOH}$  – 0.9 g
- $\text{KOH}$  – 4.2 g
- Deionised water – 1 litre
- pH of 12.6 – 13.0

The recommended test temperature for assessing static-fatigue (creep rupture) performance of the GRP rebar is 60 °C. The solution is supersaturated with Ca(OH)<sub>2</sub>, as a result CO<sub>2</sub> is continually released into solution, thus ensuring the pH level remains within the specified limits of 12.6 and 13.0.

Two sets of tests were conducted on the E-glass/vinylester rods. The first set of tests involved immersion of the GRP rods (230 mm in length) in porewater solution at 60 °C and 80 °C whilst subjected to four-point flexural loads (i.e. static fatigue). The GRP rods were dried to a constant weight before conditioning. The specimens were pre-loaded (or pre-stressed) at set percentages (i.e. 50 %, 40%, 30%, 25% and 20%) of the ultimate flexure strength  $P_{ULT}$  of the dried material as measured under ambient conditions. The results were compared to test data obtained at 40 °C by Fibreforce Composites Limited. Fig. 11 shows the self-stressing flexure fixture used for loading the GRP rods. The inner and outer spans of the loading fixtures were 100 mm and 200 mm, respectively. Load is achieved through compression of the two loading springs, which are pulled down towards the base when the loading bolts are tightened. The springs are calibrated by applying compression loads using a mechanical test machine to determine the load-displacement relationship (Hooke’s law). The load (spring displacement) was continuously monitored and adjusted to maintain constant load.

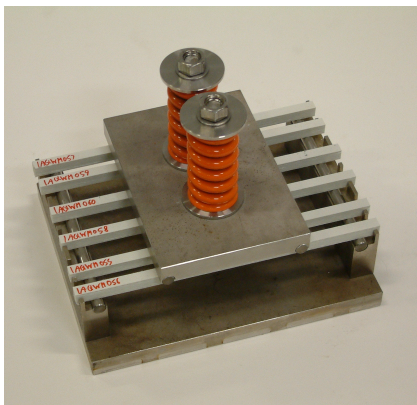


Fig. 11. Self-stressing flexure rig

The second set of tests involved immersion of the GRP pultruded rods in porewater solution and deionised water at four different temperatures ranging from 23 °C to 80 °C. The residual strength and stiffness (measured at ambient) and moisture content were measured over a period of 12 months.

It was difficult to generate reliable static fatigue data for short duration tests (i.e. static loads in excess of 50% of  $P_{ULT}$ ) due to stress relaxation that occurs in the initial stages of the test (increases with applied load). The static fatigue results showed rapid reduction in fatigue strength with exposure time for all three conditioning temperatures with the rate of degradation increasing with temperature. The fatigue strength seems to asymptotically approach a limiting value, which is temperature dependent. The strength-time data obtained for the 60 °C static fatigue tests is shown in Fig. 12.

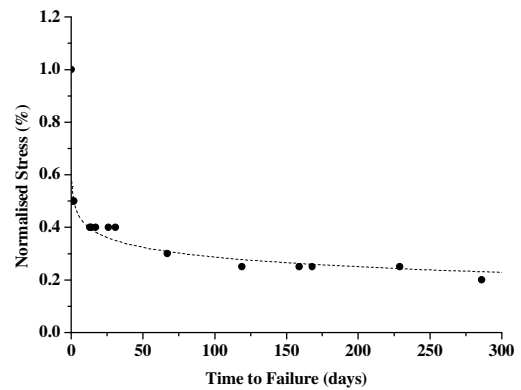


Fig. 12. Strength-time data for 60 °C

The linear-log plot of the strength-time data can be approximated using the following relationship:

$$P_{APP} / P_{ULT} = A - k \log t_f \quad (5)$$

$P_{APP}$  is the applied flexural pre-load (or pre-stress),  $P_{ULT}$  is the ambient failure load (or strength) and  $t_f$  is the time-to-failure.  $A$  and  $k$  are measured constants obtained from linear regression fits to the log-linear strength-time data (Table 4).

Table 4. Constants  $A$  and  $k$  for static fatigue tests of E-glass/vinylester

Conditioning Temperature (°C)	$A$	$k$
40	0.64	0.16
60	0.53	0.12
80	0.40	0.11

The value of  $A$  tends to be approximately four times the value of  $k$ .

The degradation mechanism at 80 °C seems to be different to that at the lower temperatures. The samples conditioned at the higher temperature discoloured and  $T_g$  tended to remain constant with exposure time. In contrast,  $T_g$  at 60 °C initially decreases and then remains steady with exposure time (i.e. moisture content) – see Table 5. The  $T_g$  results in Table 5 are for unstressed specimens.

Table 5. Glass Transition Temperature of chemically conditioned E-glass/vinylester

Exposure Time (Months)	60 °C	80 °C
Dry	118.2	
<u>Porewater</u>		
1	115.5	115.5
2	111.8	117.0
3	111.3	115.9
6	114.4	113.7
9	112.4	113.9
<u>Deionised Water</u>		
1	116.1	116.1
2	108.7	117.0
3	110.8	116.6
6	110.8	115.8
9	116.3	116.4

Exposure to aqueous and alkali solutions had minimal effect on the flexure modulus of the GRP rods. The conditioned material had a higher flexural modulus than the dried or “as-received” material. The flexural strength initially increases when exposed to either liquid (possibly post-curing). For the 23 °C exposure tests, flexural strength remained constant with exposure time for both liquids. After approximately a month exposure, flexural strength decreases with exposure time with the rate of degradation increasing with conditioning temperature. Immersion in alkali solution resulted in a slightly lower degradation rate, although the rate of moisture uptake tends to be slightly higher for specimens immersed in porewater. Table 6 compares the residual strength measured for GRP rods that have been conditioned at 60 °C.

Table 7 compares the residual flexural strength obtained for stressed (20%  $P_{ULT}$ ) and unstressed GRP rods conditioned in porewater at 60 °C. The rate of moisture uptake and degradation increases under load. It was difficult to relate the change in moisture uptake to changes in applied load due to localised yielding at the loading points and the complexity of 3-D diffusion associated with four-point flexure of the thick beam sections.

Table 6. Flexural strength of E-glass/vinylester conditioned at 60 °C

Exposure Time (Months)	Deionised Water	Porewater
As-received	862 ± 48	
Dry	853 ± 39	
1	907 ± 14	929 ± 82
2	865 ± 31	922 ± 73
3	861 ± 28	964 ± 34
6	811 ± 24	876 ± 13
9	733 ± 27	791 ± 44
12	722 ± 12	778 ± 33

Table 7. Flexural strength of stressed and unstressed E-glass/vinylester conditioned in porewater at 60 °C

Exposure Time (Months)	Unstressed	Stressed
1	929 ± 82	847 ± 86
2	922 ± 73	845 ± 32
3	964 ± 34	627 ± 113
6	778 ± 33	378 ± 117

### 3.1.2 Intermittent Exposure to Alkali Solution

In an attempt to simulate chemical spillage, the E-glass/vinylester rods, described in Section 3.1.1, were intermittently exposed to porewater solution at 60 °C. Tests consisted of exposing a single side of the GRP rods for a short-time duration of 18 minutes to the alkali solution followed by 102 minutes of drying. The ratio of immersion/drying time is specified in a number of accelerated weathering and environmental testing standards. A motorized crane was used to lower and remove the rest specimens. The specimens were clamped in a manner that allows only the top surface to come into contact with the chemical solution.

The accelerated test cycle was repeated for up to 6 months. The accelerated tests were carried out by RAPRA. The results showed that after six months, flexural properties and  $T_g$  remained relatively unaffected by intermittent exposure. Specimens were observed to lose weight within a month of exposure (2.5 wt%) - possibly due to leaching. This loss gradually diminished with exposure time.



#### 4 Steam Autoclave Conditioning

Longitudinal tensile properties were measured for steam autoclave conditioned unidirectional E-glass/F922 and HTA/F922 (Tenax carbon fibre/Fibredux 922 epoxy) laminates. The tensile specimens were conditioned for periods ranging from 24 to 72 hrs at 2.2 bar pressure and a temperature of 136 °C and then tested under standard laboratory conditions. A Midas 40 bench top autoclave manufactured by Prior Clave Ltd was used to condition the specimens.

The tensile strength data for the dry and hot/wet conditioned material is shown in Table 8. The levels of moisture absorbed by the two materials within a 48 hour time period far exceed the levels of moisture recorded after 6 weeks at 70 °C and 85% RH. The tensile properties of HTA/922 remain unaltered after 72 hours in the steam autoclave. In contrast, the E-glass/F922 was highly sensitive to moisture ingress. The material showed a 50% strength loss within 48 hours. No further changes in tensile strength were observed after 48 hours. The loss in strength can be attributed mainly to fibre degradation (i.e. corrosion and leaching). Small blisters were observed on the glass/epoxy specimens. Tensile modulus was far less sensitive to environmental exposure. A 10-12% reduction in tensile modulus was observed for the hot/wet conditioned E-glass/F922 specimens.

Table 8. Tensile strength of hot/wet conditioned E-glass/F922 and HTA/922

Material	Moisture Content (wt %)	Tensile Strength (MPa)
<u>E-glass/F922</u>		
Dry	0.00	1087 ± 29
70 °C/85% RH (6 weeks)	1.00	763 ± 42
Steam autoclave (24 hrs)	2.09	654 ± 22
Steam autoclave (48 hrs)	2.94	579 ± 47
Steam autoclave (72 hrs)	2.83	585 ± 50
<u>E-glass/F922</u>		
Dry	0.00	1684 ± 132
70 °C/85% RH (6 weeks)	1.06	1728 ± 132
Steam autoclave (24 hrs)	2.12	1691 ± 91
Steam autoclave (48 hrs)	1.90	1725 ± 42
Steam autoclave (72 hrs)	2.20	1784 ± 94

#### 5 Conclusions

The artificial weathering results clearly demonstrate that suitable non-invasive test methods are available that can provide reliable and accurate quantitative data relating the degree of surface degradation to mechanical performance. These test methods make it possible to produce measurable criteria for performance testing for determination of chemical resistance of composite materials. Gloss and Barcol hardness measurements could possibly be used to monitor the detrimental effects of service environments on flexural strength. It may also be possible to use these techniques to quantify the level of degradation (i.e. a graduated scale) and provide realistic weighting factors (see [2]).

The results also indicate that in certain circumstances where degradation does not result in leaching of constituents to the surrounding environment that analytical and semi-empirical relationship may be used to relate the degree of material property degradation with the level of degrading agent, and thus enabling extrapolation for longer exposure times under more benign (realistic) conditions.

Experimental evidence suggests that steam autoclave conditioning is a viable option for inducing accelerated ageing, particularly for those systems possessing a cure temperature and a  $T_g$  in excess of 120 to 140 °C. The technique can be expected to prove too destructive for materials possessing low  $T_g$  values, such as polyester and vinylester resins. This approach could be useful for conditioning thick laminated sections and for providing a worst case-scenario as to material degradation.

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