JOURNAL OF MATERIALS SCIENCE 18 (1983) 1059–1071

The environmental stress corrosion cracking of glass fibre-reinforced laminates and single E-glass filaments

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The environmental stress corrosion cracking of epoxy/glass fibre crossply, unidirectional coupons and single E-glass filaments have been compared. At initial applied strains $> 0.15\%$ the resin does not protect the fibres as shown by their equivalent failure times. The failure occurs in the environment and planar fractures occur because of the localized stress in the load bearing plies adjacent to a transverse crack in the 90° ply of the 0°/90°/0° coupons. These transverse cracks result from stress corrosion of the glass/resin interface, which leads to a reduction of the transverse cracking strain. At applied strains $< 0.15\%$ fracture occurs within the unexposed half of the coupons and is thought to be caused by rapid transport of glass corrosion products where they crystallize within the coupon. This phenomenon is also responsible for the progressive transverse cracking that occurs in both the 0° and 90° plies of the unimmersed half of the crossply coupon under zero load.

1. Introduction

With increasing use of glass fibre-reinforced plastics (GRP) for the fabrication of structures such as chemical plants and pipelines, which are subjected to aggressive environments, the effects of chemical degradation and particularly that of stress corrosion has become increasingly important if efficient and economic designs are to be attained. Whereas the resins are normally resistant to acidic environments [1] glass fibre-reinforced polyester pipes have been shown to be susceptible to cracking under the combined influence of a stress and the environment [2–7]. It is now well established that environmental stress corrosion failures have essentially planar fracture surfaces which show little evidence of fibre pull-out and occur normal to the load-bearing reinforcements [5]. A comparison of the stress corrosion of unimpregnated and resin impregnated glass fibre bundles lead to an explanation of the planar fracture in terms of the stress transfer length between fibre and matrix and the increase in the rate of growth of flaws near to fractured fibres [8]. Planar fractures have also been explained by localized acid attack on the fibres at the stress corrosion crack tip [6]. Hogg and Hull have identified four distinct stages to stress corrosion and have also shown that stress and strain corrosion are essentially the same phenomenon [7].

Recently [9, 10] however, the usual matrix resins have been shown to be only partial barriers to water and aqueous acids, so that the retained strength of GRP correlates closely to that of E-glass fibres [11]. Therefore the susceptibility of E-glass to both chemical and stress corrosion [12–14] places the onus onto the matrix resin to provide for chemical durability of the laminates. However crossply laminates are susceptible to transverse cracking and Jones *et al.* [15] showed that these can nucleate catastrophic failure of polyester coupons and 4-point bend. Furthermore, the magnitude of the tensile thermal strains in the 90° ply can significantly reduce the threshold for transverse cracking [15]. In addition, under tensile loading the polyester coupons were susceptible to edge-cracking at low strains but at higher strains the transverse cracks did not apparently promote a rapid stress corrosion fracture [16].

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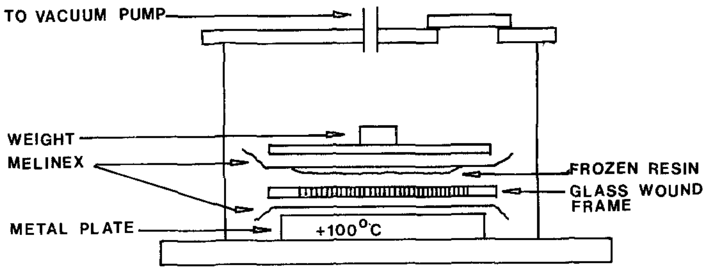

Figure 1 Schematic representation of the vacuum impregnation technique.

Methylendimethylenetetrahydrophthalic-anhydride (NMA) cured bisphenol 'A' epoxy resins and chemical resistant unsaturated polyesters have similar molecular structures so that a glass laminate from the former, whose failure has been extensively studied previously [17, 18], could be considered to be a model system. There is also a lack of published information available on the stress cracking of epoxy GRP. In this paper therefore, we report a detailed study of the stress corrosion cracking phenomena of model epoxy resin glass fibre laminates.

2. Experimental procedures

2.1. Laminate preparation and characterization

The laminates were prepared by using a winding machine to wind Silenka 051P 1200 TEX E-glass roving onto preformed steel frames. This ensured a uniform distribution of glass and consistent volume fractions. The wound frames were impregnated with degassed resin of formulation: Epikote 828 cured with 80phr Epikure NMA and catalysed with 1.5phr benzyldimethylamine (BDMA) (Shell Chemicals Ltd) using the following technique, shown schematically in Fig. 1. The resin was frozen by pouring it onto a precooled metal plate at −18° C covered in "Melinex" sheet. The release film with the adhered resin was inverted and placed with the glass fibres on top of a heated metal plate. For good impregnation it was essential that the resin did not soften prior to evacuation.

After 30 min the resin impregnated frame was removed, inspected and the "Melinex" sheets replaced with a silicone treated "Melinex" to prevent the cured resin adhering to the untreated film. The laminate was cured for 3 h at 100° C under a load of 300 kg m^{-2} in a circulating air oven, removed from the frame and cut into coupons of dimensions 25 cm × 2 cm using a water cooled diamond wheel. These were post-cured at 150° C for 3 h. For testing, etched aluminium end-tags were attached using "Araldite" cold setting epoxy adhesive. Additionally, coupons for tensile testing had resistance strain gauges bonded to them.

Each laminate was characterized by tensile testing for the initial Young's modulus. For the cross-ply laminates the multiple cracking behaviour was recorded photographically. The softening point of the laminates was found to be 140° C from the onset of curvature, on cooling coupons cut from a 0°/90° unsymmetrical laminate [19].

2.2. Stress corrosion experiments

The stress corrosion of coupons was carried out on creep machines at constant load. Physical changes and the mode of failure were recorded by time-lapse photography. Two different types of environmental cell were used, both constructed from borosilicate glass and are illustrated in Fig. 2.

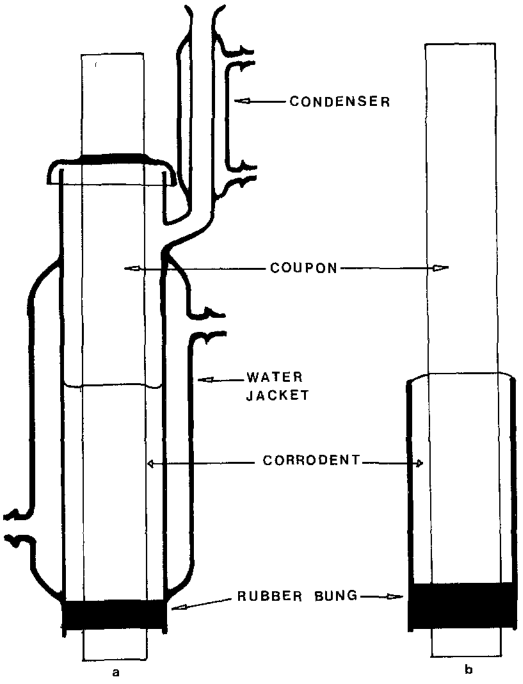

Figure 2 Types of environmental cell used for ESCC studies under constant tensile loads: (a) closed-cell and (b) open-cell.

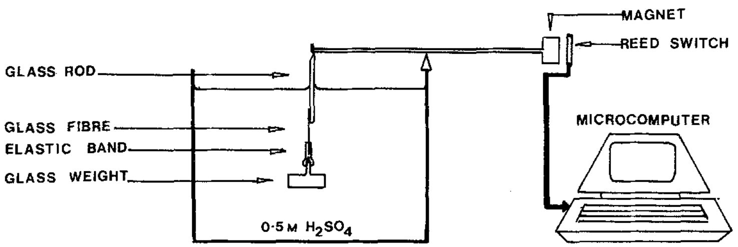

Figure 3 The experimental method for the study of the stress corrosion of single-glass filaments.

The open-cell was simply a tube supported on the coupon. The closed-cell allowed control of both temperatures by the circulation of thermostatted water, and the atmosphere if desired by purging with an appropriate gas. Both types of cell were supported on the coupon by a split rubber bung with silicone grease to provide a leak-free seal.

The experiments on single glass fibres (Silenka 051P E-glass) were carried out in the apparatus shown diagramatically in Fig. 3. Eight fibres could be tested at any one time. The single fibres were separated from the roving and their diameters determined by forward light scattering [20]. They were then glued to the glass rods and elastic bands with "Evostik" high impact adhesive. The fibres were loaded whilst immersed in the environment to prevent prestressing. All the failures were examined to ensure that they had occurred away from the adhesive.

3. Results

3.1. Laminate characterization

The model 0°/90°/0° coupons used for this study have been described previously and we have made use of the transverse cracking phenomenon for the control of the laminates [17, 18].

Table I gives the properties of the laminates used in this study. The softening point of the resin was found to be 140° C.

3.2. 0°/90°/0° crossply laminates in 0.5 M H_2SO_4

The stress corrosion of the load bearing plies may occur either within the aqueous acidic environment, and this we have termed a Mode I type of failure, or outside of the environment, termed a Mode II. The mode of failure is dependent upon the type of environmental cell, the nature of the environment and the initial applied strain. The two types of failure are illustrated in Figs. 4 and 5.

TABLE I The properties of the epoxy/E-glass fibre laminates used in this study. V_f, E and ϵ_{tlu} are the glass fibre volume fractions, Young's modulus and transverse cracking strain, respectively. The crossply laminates had 90° plies twice that of the 0° plies

Laminate	V_f (%)	E (GPa)	ϵ_{tlu} (%)
0°/90°/0°	57 ± 2	26.9 ± 1	0.29 ± 0.03
0°	56 ± 1	41.8 ± 2	–

Mode I failures occur at strains greater than $\simeq 0.15\%$ in both environmental cells, whereas at lower strains Mode II occurs only in the open-cells. Mode II failure has not been observed in the closed-cells at any applied strain.

Above $\simeq 0.15\%$ strain the times-to-failure of the coupons in the open- and closed-cells are shown in Fig. 6 to be identical, whereas at strains less than 0.15% the Mode II failures occur earlier than the Mode I failures.

A comparison of the fracture surfaces using scanning electron microscopy (SEM) from Mode I and Mode II failures revealed no significant differences, other than the presence of more crystalline deposits in the latter.

3.3. 0° unidirectional laminates in 0.5 M H_2SO_4

Failure of unidirectional coupons may also be either by Mode I or Mode II (Figs. 7 and 8). In order to compare the results from 0° unidirectional and the 0°/90°/0° crossply coupons, the maximum strain in the longitudinal plies of the crossply coupons has been calculated (i.e. the stress in the longitudinal plies in the same plane as a transverse crack). The result of such a calculation is given in Fig. 9. Above $\simeq 0.15\%$ the Mode I failure times for both laminate configurations are found to be identical. At lower strains, the Mode II failure times are also similar but fall on a line of different slope.

As shown in Fig. 10, the fracture surfaces of 0° coupons subjected to initial applied strains greater than 0.15% (i.e. Mode I) are considerably stepped in appearance in contrast to the planar fractures of the crossply coupons. At $\simeq 0.1\%$ strain and below they are as planar as the crossply failures. Examination of the fracture surfaces

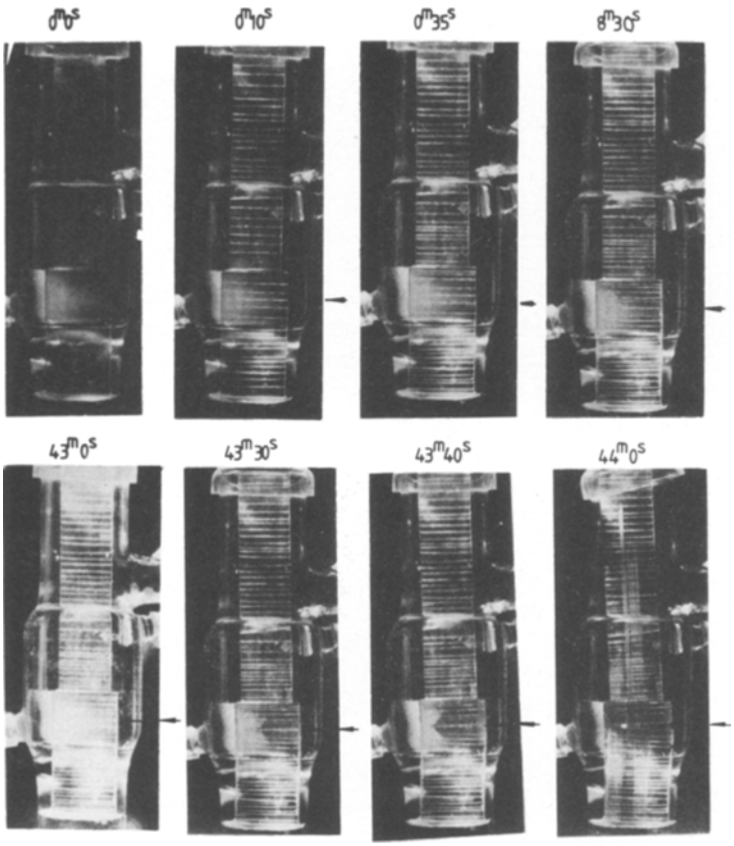

Figure 4 Mode I failure of a 0°/90°/0° coupon in 0.5 M H_2SO_4 at 23° C. $\epsilon_a = 0.49\%$. The transverse crack adjacent to the failure is arrowed.

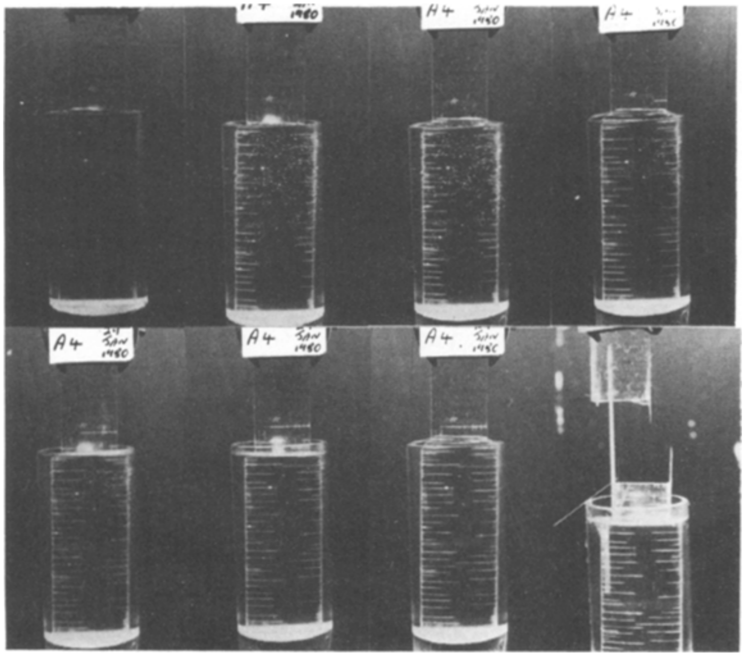

Figure 5 Mode II failure of a 0°/90°/0° coupon in 0.5 M H_2SO_4 at 23° C. $\epsilon_a = 0.1\%$.

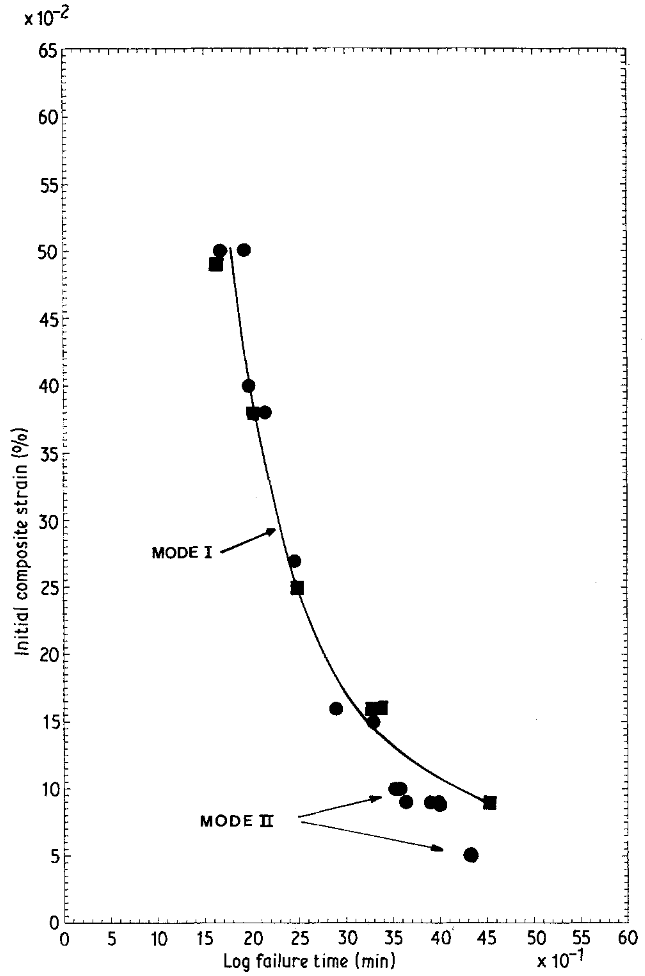

Figure 6 ESCC failure times of epoxy 0°/90°/0° coupons in open (●) and closed-cells (■). The applied strain is that on the composite obtained from the stress–strain curve of the characterization coupon for each laminate.

by SEM did not reveal any significant differences with those of the crossply coupons.

3.4. Crossply coupons in various acids under zero load

Crossply coupons partially immersed in 0.5 M sulphuric acid quickly develop damage in the unimmersed half of the laminate, whilst the immersed part showed no sign of damage (Fig. 11). This damage, termed Mode III, consists of longitudinal splits and transverse cracks, and has not been observed in longitudinal coupons. If the acid is replaced by hydrochloric or nitric acid of similar concentration, then no damage occurs, (hydrochloric acid gave very slight damage after 2 weeks), whereas phosphoric acid produces damage in the immersed half also, but after a considerably longer time than the sulphuric acid.

A crossply coupon totally immersed in 0.5 M H_2SO_4 for 15 min showed no tendency to longitudinal split or tranverse crack, the attack is apparently limited to the exposed surfaces only.

3.5. Stress corrosion of single glass fibres and tows

At strains of 0.15% and above single glass fibres failed in similar times to those of both the unidirectional and crossply coupons (Fig. 12). At lower strains the fibres failed more rapidly than the laminate under Mode II conditions. Glass fibres stored in 0.5 M H_2SO_4 became embrittled, and spontaneous spiral cracking of the outer sheath occurred in the majority of fibres of the tow.

Tows of fibres always failed within the acid under applied strains of 0.1% in an open-cell experiment. Under these conditions the laminates would have failed by Mode II.

4. Discussion

4.1. Glass fibres

Fig. 12 shows that, above an applied strain of

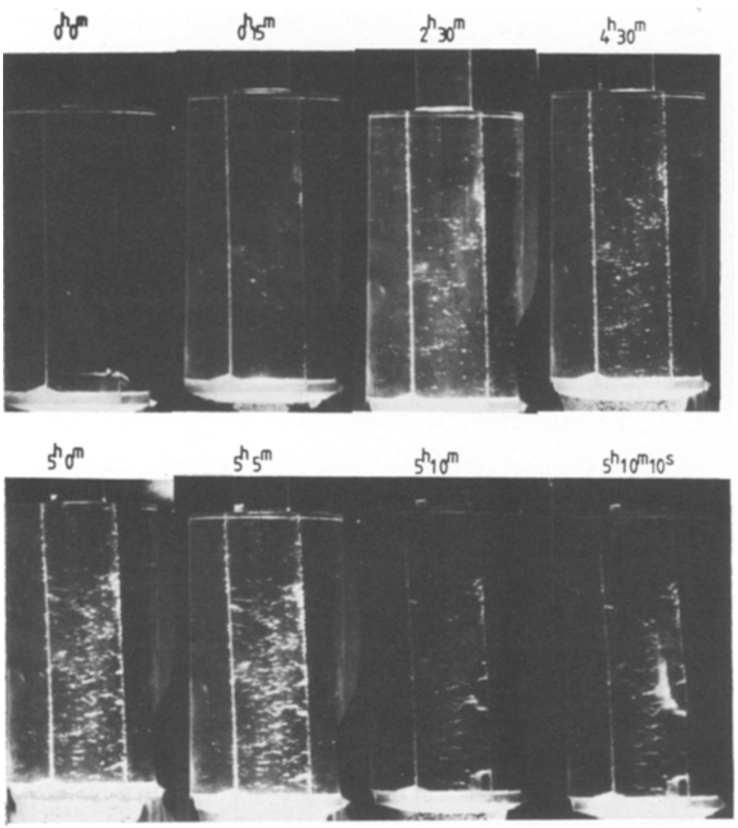

Figure 7 Mode I failure of a 0° unidirectional coupon in 0.5 M H_2SO_4 in an open-cell with an initial applied strain ϵ_a of 0.34%.

0.15%, the times-to-failure of single glass fibres and laminates are similar, demonstrating that the acid rapidly reaches the fibres in the laminates. Below 0.15% the laminates take longer than the glass fibres to fail.

Relatively few spiral-cracked fibres have been found in the laminate fracture surfaces compared with the number in unstressed tows. This suggests that spiral cracks are only formed when the fibres are not stressed and therefore their role in the stress corrosion of laminates may be unimportant. The fracture surfaces of single fibres were shown to be smooth and similar to those reported by Charles [21] and to those found in stress corroded

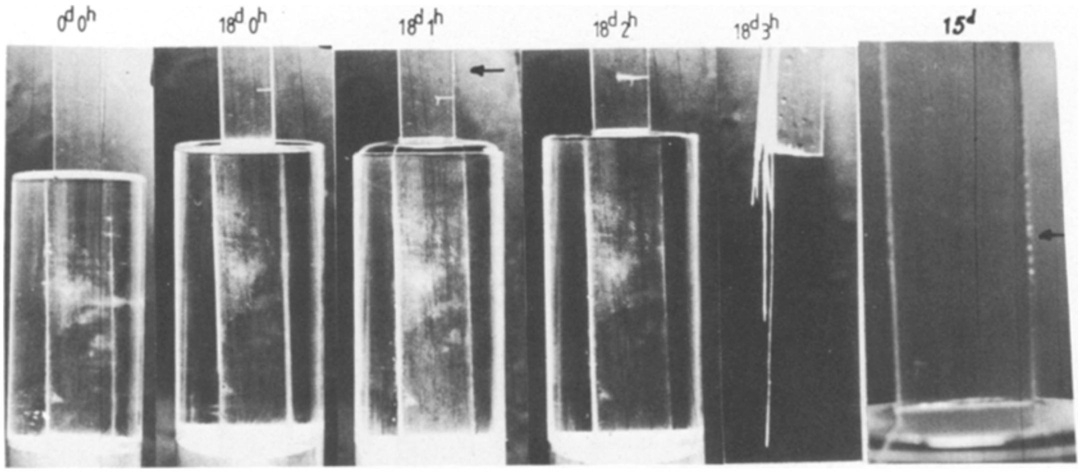

Figure 8 Mode II failure of a 0° unidirectional coupon in a 0.5 M H_2SO_4 in an open-cell at an applied strain ϵ_a of 0.09%. Note the crystalline deposit on the cut edge above the growing crack.

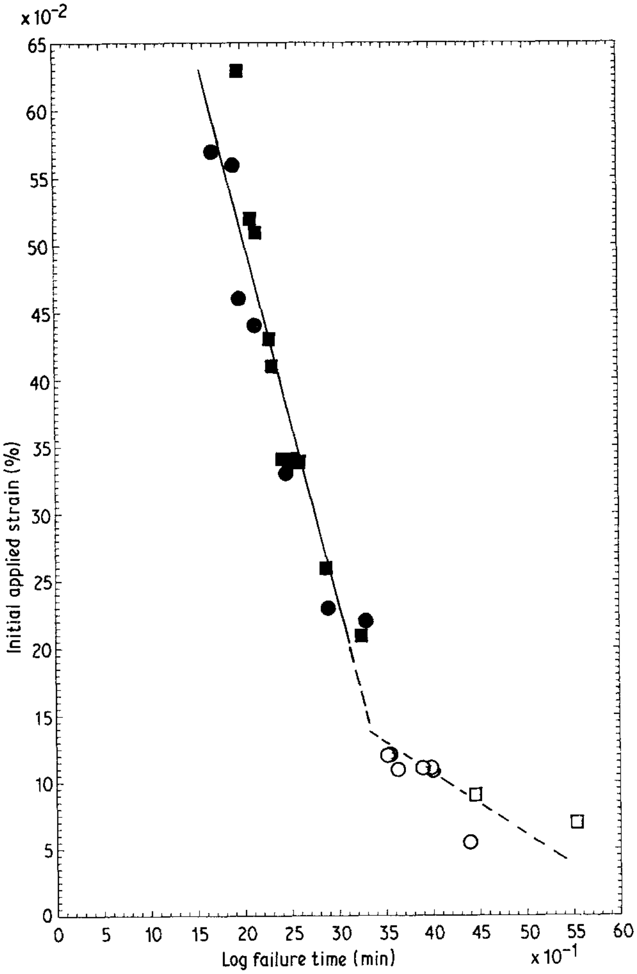

Figure 9 A comparison of the Mode I and Mode II failure times of 0°/90°/0° (●, ○) and 0° (■, □) coupons in open-cells. The open points are Mode II failures. The initial applied strain in the former has been calculated assuming the 90° is not load bearing.

laminates [2–7]. Some indication of a core-sheath structure has been observed, but it is believed to be caused by corrosion after stress corrosion fracture. Few core sheath structures have been observed in laminate fracture surfaces. Glass tows failed within the acid when tested in open-cells, so that the Mode II fracture can be considered to be a property of the laminates.

4.2. Mode I type failure

Mode I failure of crossply laminates occurs by fracture of the longitudinal plies adjacent to an initial transverse crack in the 90° ply. This is shown in Fig. 4 where the crack which leads to failure is arrowed. These failures can be explained as follows: when the transverse ply of a crossply coupon fractures, the load that is supported is redistributed onto the longitudinal plies. The modified shear lag theory of Garrett and Bailey [22] describes how this additional load on the longitudinal plies is transferred back into the transverse ply as a function of distance, and predicts a transfer length of less than 2 mm for 90% of this additional load.

Thus the longitudinal ply adjacent to a transverse crack experiences a greater stress than at a short distance away. Therefore, stress corrosion will be most rapid in this area, and failure will occur adjacent to the initial transverse crack. The effect of this localized highly stressed region is that crossply coupons that fail in this manner have planar fracture surfaces, whereas those from longitudinal coupons, which have no localized laminate stress concentrations, have a stepped topography (Fig. 10).

The failure of 0° coupons at strains greater than 0.2% begins by the initiation and growth of individual stress corrosion cracks randomly over the immersed half of the coupon, as shown in Fig. 13. These cracks occur normal to the applied load and on the microscale are seen to cross individual fibres normal to their axes. With time, these

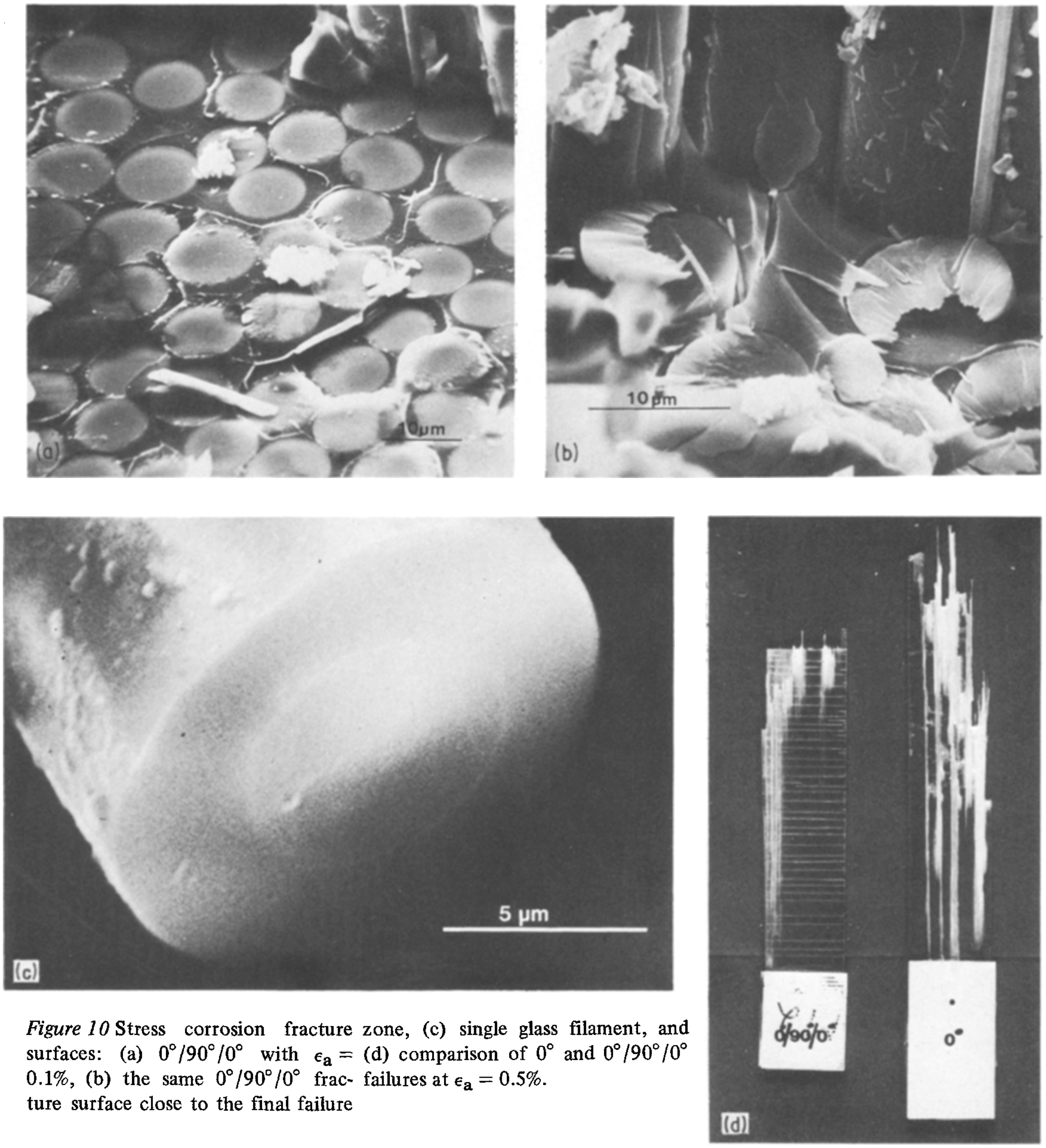

Figure 10 Stress corrosion fracture surfaces: (a) 0°/90°/0° with $\epsilon_a = 0.1\%$, (b) the same 0°/90°/0° fracture surface close to the final failure zone, (c) single glass filament, and (d) comparison of 0° and 0°/90°/0° failures at $\epsilon_a = 0.5\%$.

cracks increase in size and coalesce by longitudinal splitting, and are easily visible over the whole of the immersed surface, in Fig. 7. When the crack reaches a critical size, failure of the coupon results. At applied strains of less than that for transverse cracking of crossply laminates, instantaneous failure of the transverse ply does not occur on loading, but in the aqueous acidic environment there is a slow growth of "edge-cracks" into the specimen which are clearly seen in Fig. 5. These "edge-cracks" do not span the entire width of the coupon because of their mutual interference. However, we have shown by comparison of the transverse cracking of crossply coupons in air and in aqueous acids that they behave similarly to transverse cracks produced in air and may be considered to be identical [23, 24]. It appears therefore, that the transverse cracking strain of the crossply coupons is reduced under the combined influences of the stress and the acidic environment.

Experiments with polyester coupons, in bend, lead to the hypothesis that transverse cracks would cause a more rapid failure, of the longi-

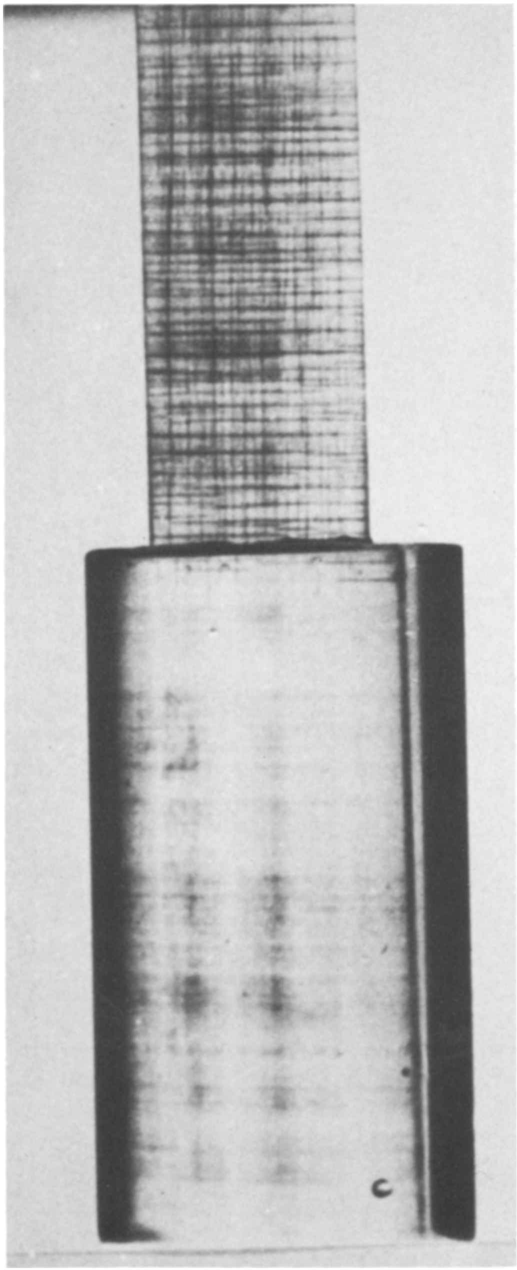

Figure 11 Mode III damage in the unexposed half of a 0°/90°/0° coupon without an external applied strain. The damage is initiated just above the 0.5 M H_2SO_4 after about 3 days and progresses up the coupon.

tudinal plies of a 0°/90°/0° laminate than those of a 0° laminate, since not only would they allow the acid to enter the laminate, but could also act as sharp notches where stress corrosion cracks would be initiated. Since the times-to-failure of unidirectional and crossply coupons above 0.15% are shown in Fig. 9 to be the same, it is concluded that neither factor significantly affects the fracture of the crossply coupons. This result is in agreement with experiments with polyester laminates under tensile loads where the failure times were also unaffected by the presence of transverse cracks [24, 25].

The transverse cracking of this laminate system in air was shown previously to be preceded by debonding at individual fibres, which coalesce to form a transverse crack at a higher strain [18]. Since the transverse cracking strain, ϵ_{tlu}, is reduced by stress corrosion, it follows that the debonding strain will also be reduced. It can be shown that ϵ_{tlu} is reduced to 0.1% in short term environmental tests, and since for this laminate configuration where the inner ply thickness is twice the outer ply, the thermal strain in the longitudinal direction of the transverse ply is equivalent to that in the transverse direction of the longitudinal ply and that these strains do not decay significantly in the environment over the timescale of the experiments, then it follows that the 0° plies will also become debonded. Therefore, equivalent failure times of the single filaments and the 0°/90°/0° coupons can be understood in terms of the stress corrosion of the interfaces leading to rapid transport of the aqueous acid. Since the stressed 0° coupons, at applied strains $> 0.2\%$ also have similar failure times, it would appear that debonding of unidirectional coupons can also occur. Experiments with small cells clamped to the faces of these laminates gave similar results which demonstrate that diffusion through the surface resin to the interfacial region is important [26].

4.3. Mode II type failure

The Mode II failure of crossply coupons, unlike the Mode I failures, are not preceeded by transverse cracks, but rather the failure crack grows through both the transverse and longitudinal plies concurrently. At strain $< 0.15\%$ the Mode II type of failure occurs in shorter times than Mode I, but not so rapidly as single glass fibres. At higher strains when failure of single glass fibres occurs in similar time to that of laminates, Mode II failure has not been observed. Although stress corrosion may nucleate just above the aqueous acid in 0° coupons, these do not lead to final failure. Therefore, the Mode II mechanism only operates when resin is protecting the glass fibres and is suppressed in closed-cells when the atmosphere surrounding the unexposed

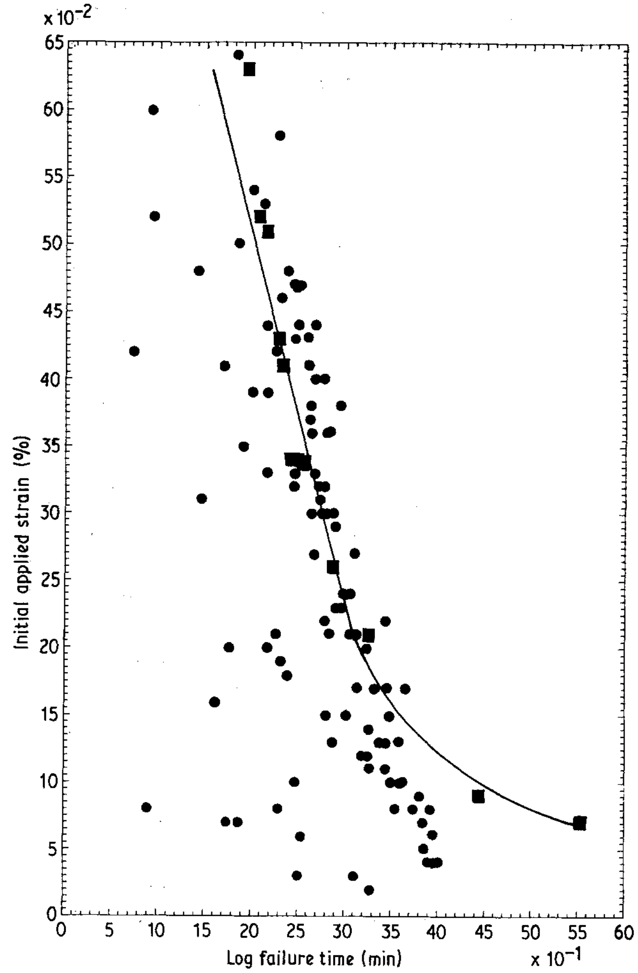

Figure 12 A comparison of failure times of 0° coupons in open-cells (■) with single glass filaments (●). Mode II occurs at $\epsilon_a < 0.1\%$. An error of ± 0.5 μm in the fibre diameter gives an error of ± 10% on applied strain for 10 μm single filaments.

half is moist. Enrichment of the atmosphere with oxygen had no effect on the time or mode of failure which eliminated oxidation as the mechanism.

Observations that the dry half of fractured crossply coupons became increasingly damaged when removed from the environment, which could not be accounted for by differential shrinkage stresses on drying out, led to the discovery of cumulative damage in the unexposed halves of partially immersed unstressed coupons. We have termed this Mode III (Fig. 11). There was however, a crystalline deposit always present on the unexposed edges and within the longitudinal splits.

X-ray analysis in the transmission electron microscope showed it to be rich in aluminium and sulphur with traces of calcium and potassium. Furthermore, Mode II was suppressed in hydrochloric and nitric acids but could form within the environment in the case of phosphoric acid (Table II). It is well documented [12] that acids leach the metallic ions from glass and for E-glass these are Al^{3+}, Ca^{2+}, and Na^{+}, and the trend in Table II appears to be related to the solubility of these salts. The apparently lower rate observed in phosphoric acid can be ascribed to its reduced acidity, as shown by a more positive acid dissociation constant, pK_a. In addition, as the initial applied strain in the laminate is decreased, additional Mode III damage, whose extent is increased, appears in the unexposed half of crossply coupons that fail by Mode II. It is therefore apparent that the mechanism responsible for stress corrosion Mode II failures is the same as that for Mode III damage within partially immersed coupons, under zero load.

The crystalline material found in Mode II fracture surfaces was found by microprobe analysis to be rich in calcium and sulphur with traces of aluminium, potassium and iron.

Fig. 8 shows that aluminium-rich crystals are

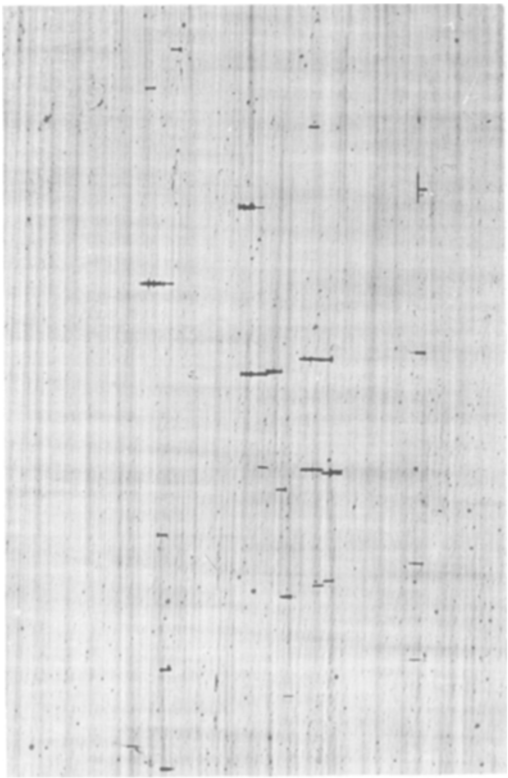

Figure 13 Micrograph of 0° coupon showing initiation and growth of individual stress corrosion cracks.

also deposited at the edge of these low strain coupons. Therefore, it is clear that, for aqueous H_2SO_4 the more insoluble calcium salts are precipitated within the laminate, whilst the more soluble aluminium salts are transported to the surface of the coupon, where precipitation occurs by evaporation of water. Acids, with soluble calcium and/or aluminium salts do not cause Mode III damage. Its absence in the immersed half of coupons in aqueous sulphuric acid shows that the glass degradation products can remain in solution and not cause damage. This is in contrast to phosphoric acid, whose calcium salts are more insoluble. Further support for the mechanism is given by the fact that, in 1 M HCl, at 0.1% strain, a delayed Mode I fracture occurs (in the environment), and that the fracture surfaces are devoid of crystalline deposits.

Thus the Mode II fractures and Mode III damage are caused by additional local stresses from the crystallization of the rather insoluble calcium salts. The glass corrosion products can clearly be transported to the unimmersed half by capillary action at the stress-corroded interfaces, since the thermal strains are sufficient to debond the fibres of a laminate whose transverse failure strain is reduced under the influence of the acid environment [23, 24]. The continued evaporation of moisture from the laminate surface, or diffusion from the interface into "dry" resin, effectively maintains a concentration gradient within the laminate which produces an additional driving force for acid transport. When distillation is not possible, as in the case of the closed-cells and fully immersed coupons, Mode II or Mode III behaviour is suppressed.

At applied strains at $< 0.15\%$ diffusion through the resin normal to the fibres is slower than along the stress-corroded fibre interfaces so that crystallization and hence the Mode II crack is initiated at the edge of the coupon, just above the environment where the fibres are not debonded. Crossply coupons develop thermal strains in the matrix which put the fibres into compression so that coupons under zero load are subject to cracking rather than fibre fracture. The stress state, associated with the Mode III cracks, causes further debonding and acid transport and the unexposed half becomes progressively damaged with time.

The planar fracture surfaces in the stress

TABLE II The time for onset of Mode III cracking (t_{III}) of 0°/90°/0° epoxy/glass coupons half-exposed to aqueous acids and its relationship with the acidity (pK_a) and solubility of the calcium salts

Acid	H_2O	1 M HCl	1 M HNO_3	0.5 M H_2SO_4	0.5 M H_3PO_4
pK_a	–	-7	-1.4	-3	2.1
t_{III} (days)	∞	∞	∞	3–7	> 14
Salt		*Solubility* (mol kg^{-1})*			
$CaCl_2$	5.36	> 5.36	–	–	–
$Ca(NO_3)_2$	6.2	–	> 6.2	–	–
$CaSO_4$	4.7×10^{-2}	–	–	4.4×10^{-2}	–
$Ca_3(PO_4)_2$	6.3×10^{-4}	–	–	–	3.5×10^{-5}
$Al_2(SO_4)_3$	0.91	–	–	–	–

*The solubilities in acidic environments are calculated from their solubilities in water and the appropriate solubility product [26, 27].

corrosion of GRP have been explained by Hogg and Hull [6], as localized acid attack upon exposed fibres at the crack tip. However, at applied strains $>0.15\%$ the equivalent failure times for crossply, 0° unidirectional and single glass filaments favour the explanation of Aveston *et al.* [8] that the rate of growth of flaws in fibres adjacent to the fractured filament is increased tenfold. At lower strains the resin provides some protection to the glass fibres so that the former mechanism of localized corrosion at the crack tip formed under the stresses generated by the crystallization process could also operate. The true reason for the planar fractures is therefore dependent on the detailed stress corrosion mechanism.

5. Conclusions

The prime cause of environmental stress corrosion of these epoxy glass fibre composites is the susceptibility of the glass fibres to stress corrosion. Therefore the main function of the resin is to protect the glass fibres from the environment. However, the glass/resin interface will stress corrode with the result that the acid penetrates the laminate, so that at initial applied strains greater than 0.15% the Mode I failure times are the same as for single filaments. At lower strains the resin protects the glass fibres and the laminates are more resistant to stress corrosion, but a second mechanism termed Mode II is available which leads to a stress corrosion fracture more rapidly than by Mode I failures at these strains. Mode II is associated with the crystallization of calcium-rich glass degradation products in the unexposed half of the coupons. In crossply coupons under zero load this results in Mode III cracking because the localized stresses are unable to overcome the compressive forces in the load bearing fibres with the result that the unimmersed half becomes progressively more damaged with time.

Thus the environmental stress corrosion resistance of GRP is not only controlled by the rate of diffusion of the acid through the resins to the glass fibres but by the degradation of the interface and the accumulation of crystallizable glass corrosion products in unexposed areas of the composite. Furthermore the change in mechanism at low strains shows that high strain control tests may not predict the long term-low strain life of a GRP structure.

Acknowledgements

We thank the Science and Engineering Research Council for a research studentship (JWR), and an equipment grant, and Silenka (UK) Ltd for the glass fibres.

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Received 22 July
and accepted 31 August 1982