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High temperature acidic stress corrosion of glass fibre composites

Part I *Effect of fibre type*

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The stress corrosion characteristics of uniaxial glass fibre reinforced thermosetting resin composites have been examined in hydrochloric acid at 80 °C. A simple technique based on linear elastic fracture mechanics (LEFM) is presented for characterizing crack growth in these materials subjected to hostile acidic environments. The environmental stress corrosion cracking is investigated both for different types of resin and different types of glass fibre reinforcements. Two matrices were used: DERAKANE* 411-45 epoxy vinyl ester resin (based on Bisphenol-A epoxy resin) and DERAKANE 470-30 epoxy vinyl ester resin (based on epoxidized novolac resin). Two glass fibre types were employed: standard E-glass fibre and **ECR**GLAS®, a special type of E-glass with superior acid resistance. Model experiments using a modified double cantilever beam test with static loading have been carried out on unidirectional composite specimens in 1 M hydrochloric acid solution at 80 °C. The rate of crack growth in the specimen depends on the applied stress, the temperature and the environment. Consequently, the lifetime of a component or structure made from glass fibre reinforced plastics (GRP) subjected to stress corrosion conditions, could be predicted provided the dependence of crack growth rate on stress intensity at the crack tip is known. Scanning electron microscope studies of the specimen fracture surfaces have identified the characteristic failure mechanisms. The most important finding of this work is that the selection of DERAKANE epoxy vinyl ester resins reinforced with **ECR**GLAS® fibre exhibited superior resistance to crack growth at 80 °C compared to similar E-glass reinforced composites at room temperatures.

1. Introduction

Few construction materials can safely be used in prolonged contact with aggressive chemicals, especially at elevated temperatures, without corroding, swelling, cracking or dissolving. Glass fibre reinforced plastics (GRP) in general perform well in this respect, but it is not true that GRP materials are *totally* inert to chemical attack. They tend to perform well in dilute acids and alkalis and certain other environments, where traditional materials such as steel, brass, zinc and aluminium encounter severe corrosion.

When a GRP composite undergoes degradation due to the effects of environment, the causes may involve; (i) loss of strength of the reinforcing fibres due to stress-corrosion, (ii) loss of adhesion and interfacial bond strength from degradation of the fibre–matrix interface, (iii) chemical degradation of the matrix material, (iv) dependence of the matrix modulus and strength on time and temperature, and (V) accelerated degradation caused by combined action of temperature and chemical environment [1].

Environmental stress corrosion cracking (ESCC) is the delayed brittle fracture of a stressed material under the influence of an environmental agent [2]. Stress corrosion has been reported for E-glass reinforced plastics based both on thermosetting [3, 4] and thermoplastic [5, 6] matrices in acidic environments. Many of the practical problems occurred typically in chemical plants and related anti-corrosion applications. Although failures of GRP process equipment (such as storage tanks, for example) were not uncommon in the past, improvements in both resin and

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glass fibre technology, together with improved design knowledge have greatly increased the reliability of the GRP equipment in these applications.

The increase in environmental awareness over the past few years has led to an expansion in the use of gas cleaning systems both in power generation and waste disposal/energy recovery. Corrosion resistant GRP composites are excellent candidates for use in the corrosive conditions found in such plants, but the high operating temperatures justify a fresh look at ESCC resistance in order to demonstrate the material's suitability in these applications.

Under ESCC conditions at room temperature, failure occurs by a process of slow crack propagation from a stress raiser, resulting in a planar fracture surface typical of brittle behaviour. Eventually, ESCC of fibres results in a crack of a critical length, where the crack tip stress intensity exceeds the fracture toughness of the composite and this can result in catastrophic failure [7, 8]. The behaviour of composites in acid at room temperatures has been studied extensively and the role of fibre and matrix on crack growth rates has been determined [9]. However, the effect of raising the ESCC test temperatures to levels more representative of flue gas and waste incineration applications has not previously received adequate study.

Organic materials are generally less stable at elevated temperatures compared to metals and undergo degradation resulting from progressive chemical breakdown. Exposure of composites to elevated temperatures may influence ESCC behaviour through formation of differential thermal stresses between the fibre and matrix, the development of larger deformation zones and higher fibre pull-out lengths. Such mechanisms, coupled with possible changes in resin toughness at the elevated temperatures might be expected to *increase* the crack growth resistance with temperature. However, temperature effects also influence the rate of the chemical reaction and would tend to increase the rate of the fibre fracture, thereby *decreasing* the crack growth resistance of the material. The overall resistance of the composite at elevated temperatures is therefore likely to be governed by a combination of these opposing effects.

2. Experimental methods and materials

2.1. Materials

All the tests were performed on unidirectional glass fibre reinforced epoxy vinyl ester resin matrix composites using two commercially available glass fibre reinforcements. Standard E-glass roving COTECH® (E-LPb 567) was supplied by Tech Textiles International Ltd and ECRGLAS® by Owens-Corning Fiberglas Corporation. ECRGLAS® is from the "E" family of glasses with a broadly similar composition to that of standard E-glass, with low alkali content, no boron and fluorine and additions of zinc and titanium oxides, resulting in enhanced resistance to acid attack.

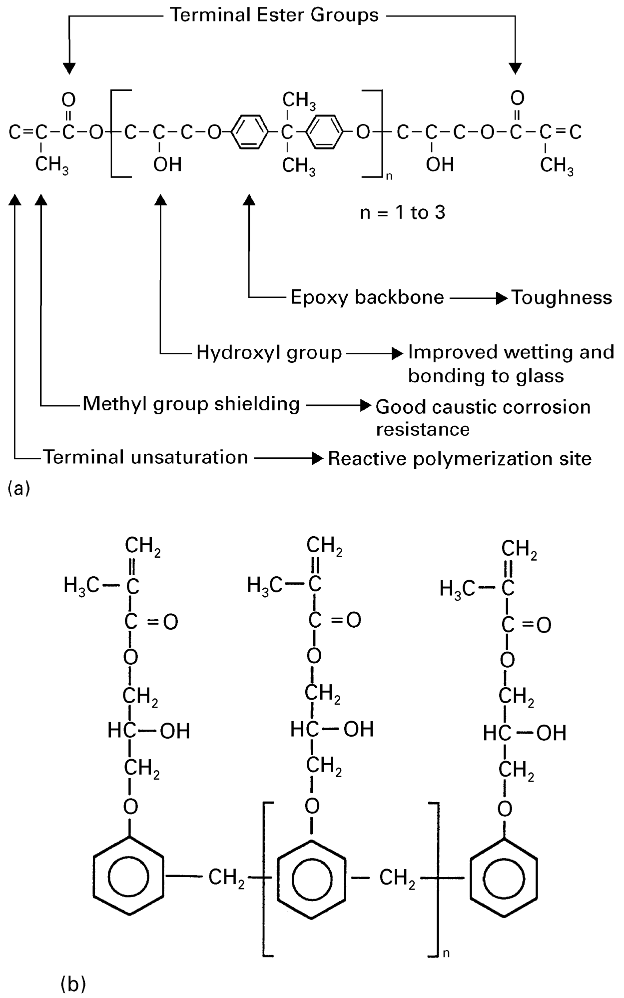


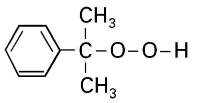
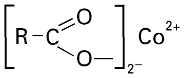
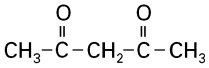
Figure 1 (*a*) Structural characteristics of DERAKANE 411-45 epoxy vinyl ester resin and (b) chemical structure of DERAKANE 470-30 epoxy vinyl ester resin.

Two epoxy based vinyl ester resins were used; DERAKANE 411-45 resin based on Bisphenol-A epoxy resin and DERAKANE 470-30 resin based on epoxidized novolac resin, both supplied by Dow Europe. Fig. 1 represents the chemical structure of the two resins. A summary of the resin cure formulations used, together with the peroxide catalysts, accelerators and time gel retarder agents (supplied by AKZO) is given in Table I.

Unidirectional prepregs were made by passing the glass rovings through a resin bath and winding onto the mandrel of a drum-winding prepregging machine. Four strips of pre-preg, 300 mm by 370 mm, were cut and stacked together (with all fibres aligned in the same direction) to form the main test laminate. Due to the design of the test specimen and the geometric constraint resulting from the side grooving (see Fig. 2), the finished test specimens required additional reinforcement on their outer faces. Three layers of non-crimp E-glass fibre fabric (400 g per m^2 per direction) and 0°/90° fibre orientation (supplied by Flemings Laces Ltd.) were applied using a hand lay-up method to both sides of the main unidirectional prepreg layers.

COTECH® is a Registered Trademark of Tech Textiles International Ltd.

TABLE I Cure formulations used for manufacture of GRP laminates

	Curing agent	Accelerator agent	Time gel retarder
	Cumene Hydroperoxide	Cobalt carboxylate	Acetyl acetone
	 *TRIGONOX®239*[a]	 *Accelerator NL 51P*[a]	 $CH_3-\overset{O}{\overset{\|}{C}}-CH_2-\overset{O}{\overset{\|}{C}}-CH_3$ *PROMOTOR C*[a]
DERAKANE 411-45 resin	2 phr[b]	1 phr	0.3 phr
DERAKANE 470-30 resin	1.75 phr	0.5 phr	0.2 phr

(a) Supplied by AKZO.
(b) Parts (by weight) per hundred resin.

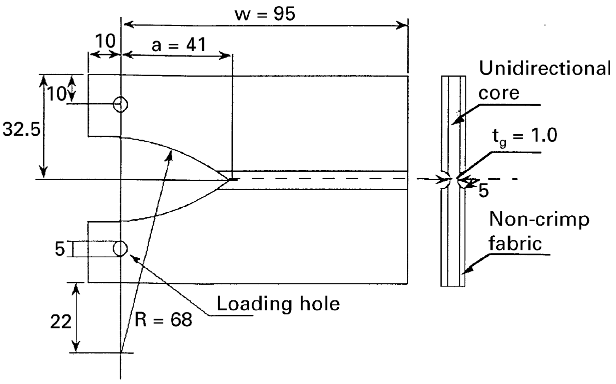


Figure 2 Tapered double cantilever beam specimen details (units in mm).

The thickness of the final laminate was controlled by compressing the material prior to cure, between steel plates with suitable spacers in order to produce a void-free laminate 5 mm thick. The laminate was cured for 24 h at ambient temperature under pressure followed by post-curing and ageing for 2 h at 115 °C and 24 h at 102 °C respectively. Post-curing and ageing of the finished laminates was carried between the steel plates to avoid the possibility of warping. Finished specimens were found to have glass content in the range of 0.48–0.55 vol % (ASTM D2584).

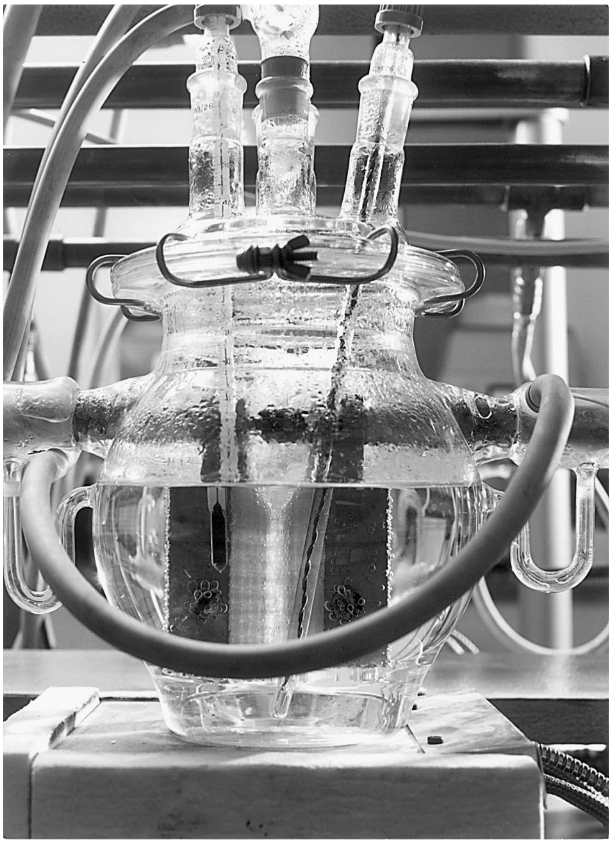


Figure 3 Measuring stress corrosion crack growth test at elevated temperatures.

2.2. Experimental methods

Stress corrosion crack growth tests have been carried out using a tapered double cantilever beam (TDCB) specimen designed by Price [2] as shown in Fig. 2. This specimen configuration was found to produce a stress intensity level which is independent of crack length for approximately the first 15–20 mm of crack extension. Specimens were tested under constant loading conditions using modified creep rigs fitted with a customized 2 l PYREX® glass flask, filled with the corrosive environment, as is shown in Fig. 3. The loading arms pass through two horizontal condensers in the flask wall. A vertical condenser was used to maintain the volume levels in the test cell and consequently the strength of the solution was assumed to remain unchanged over the duration of the experiments. A K-type thermocouple was immersed in the solution and connected to a temperature controller, which switched on/off automatically to maintain a constant solution temperature of 80 °C throughout the duration of the experiment.

Notches were machined into the test specimens using a computer controlled milling machine with a carbide cutter and then sharpened prior to testing by scoring the notch tip with a scalpel. The crack length was measured directly during the test using a travelling microscope with illumination from behind the specimen to highlight the crack tip position.

All tests were carried out using 1 M hydrochloric acid.

3. The application of a fracture mechanics approach

3.1. Fracture mechanics analysis

Several attempts have been made to apply fracture mechanics to composite materials and a useful review has been published by Kanninen *et al.* [10]. There are considerable problems encountered when applying linear elastic fracture mechanics (LEFM) to the in-plane propagation of cracks in composites. The problems result from the development of regions of microcracking or damage at or near the growing crack tip. The exact nature of the damage will depend on the form of the fibre reinforcement, i.e. mat, fabric or aligned stacks of plies. In some cases, microcracking can be linked to regions of plastic deformation such as occurs in ductile materials and whilst this can be considered to provide a toughening mechanism in the composite by reducing the effective crack tip stresses, it makes the job of measuring and analysing the crack growth behaviour more difficult as the process zone cannot be considered crack tip specific. A number of methods have been proposed to take account of damage related process zones at the crack tip when calculating crack tip stress intensities from the applied stress on the specimen and the measured crack length [13].

When a growing crack encounters fibres or fibre bundles aligned at high angles relative to the crack plane, extensive debonding may occur. The magnitude of such an effect is determined by the fibre–matrix bond strength. If debonding is extensive, the crack blunting effect may be such to render the concept of a crack tip stress concentration (and therefore the applicability of LEFM) inappropriate and this might be expected with the unidirectional glass fibre composites tested here. However, the environmental stress corrosion (ESC) failures of glass fibre reinforced composites, generated in acidic environments are characterized by sharp cracks with well defined tips (resulting in flat fracture surfaces) and an absence of fibre debonding. With this type of behaviour, a fracture mechanics approach would appear to be justified, as the crack growth process occurs local to the crack tip.

In theory, the resulting crack growth versus applied stress intensity data could be directly applicable for service lifetime predictions if sensible estimates of inherent crack sizes in composite structures could be assessed and some value for ultimate non-environmental fracture toughness could be measured. However, both of these parameters are difficult to predict. Also, the concept of an initial inherent crack or defect from which stress corrosion cracks will propagate is further undermined by the real nature of large industrial corrosion-resistant composite structures (which are not built with the same degree of control found for example, in the aerospace industry). In practice, the main structural part of a large industrial composite is seldom exposed directly to the aggressive chemical environment and some form of resin rich corrosion-barrier layer isolates the structural layer from the aggressive environment. Given these considerations, crack growth data generated using a fracture mechanics approach will be most useful in allowing direct comparisons between different composite systems without any complications arising from specimen shape, fibre orientation changes or construction variables. This ability to discriminate between materials should facilitate more reliable materials selections, particularly for higher temperature use in acidic environments.

The strain energy release rate, (G_I), for a given crack length was obtained by performing compliance calibration, and using the Irwin and Kies relationship [12].

$$G_I = \frac{P^2}{2\,t_g w}\,\frac{\mathrm{d}\,C}{d(a_a/w)} \tag{1}$$

where w = specimen width, d_n = web thickness and represents the thickness through the specimen at the crack front, P = applied load, a = crack length (see Fig. 2) and $\mathrm{d}\,C/\mathrm{d}\,(a/w)$ compliance derivative.

The above equation when combined with the Paris and Sih relationship for a plain strain conditions [13]:

$$K_I^2 = \frac{G_I E}{1-\nu^2} \tag{2}$$

where ν = Poisson's ratio, gives:

$$\frac{K_I}{P} = \left(\frac{E_{ef}}{2(1-\nu^2)}\,\frac{1}{t_g w}\,\frac{\mathrm{d}\,C}{\mathrm{d}\,(a/w)}\right)^{1/2} \tag{3}$$

where the Young's modulus in (Equation 2) is replaced by an effective modulus, E_{ef}, derived by Sih *et al.* [14]. From the above equation it can be seen that only the compliance derivative is required for the determination of the fracture toughness K_I for specimens of a given size.

3.2. Compliance calibration measurements

Compliance measurements of TDCB specimens were carried out using an Instron 1122 tensile machine. A clip-on COD (crack opening displacement) gauge was used, (UB-5A, by Techni Measure) since a compliance calculated from crosshead movement would be highly inaccurate due to the small displacements involved. Compliance calibration tests were performed at a crosshead speed of 0.2 mm per min. Fig. 4 shows a typical compliance curve for these specimens. The variation of the normalized stress intensity factor (K_I/P) with the normalized crack length (a/w) for all composite systems are shown in Fig. 5. It can be seen that for crack growth over the first 15 mm, ($0.4 < a/w < 0.53$), K_I is reasonably constant but thereafter, the crack tip K_I value increases with increasing crack length.

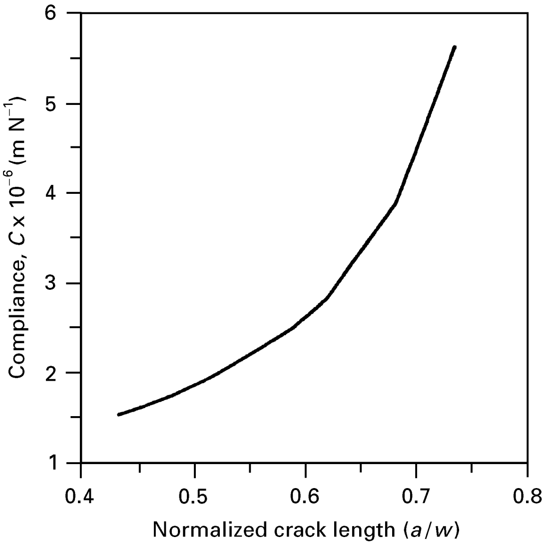


Figure 4 Crack opening compliance calibration curve for TDCB specimens.

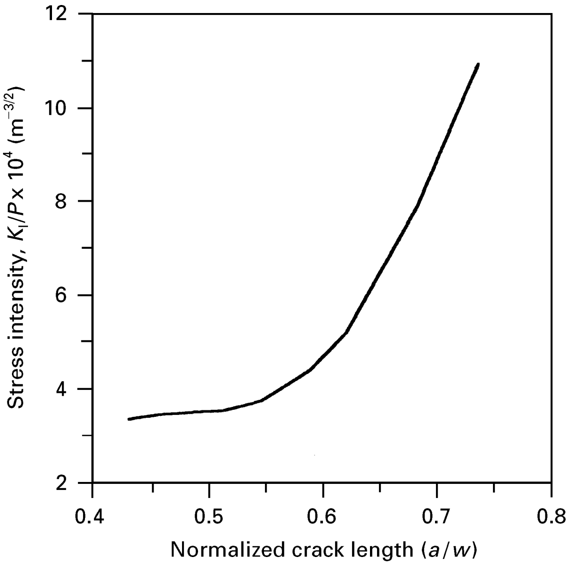


Figure 5 Variation of stress intensity with crack length in TDCB test.

4. Stress corrosion tests at elevated temperatures

When the specimens are loaded in the hostile acidic environment, a characteristic failure sequence occurs. After nucleation at the notch tip the stress corrosion crack propagates in a plane perpendicular to the fibre orientation. A series of typical curves showing the changes in crack length with time for all composites systems at 80 °C is shown in Figs 6 and 7. In all cases, there was an initial delay before cracking was observed and this was most pronounced for the DERAKANE 411-45 resin/$\overline{\mathrm{ECR}}$GLAS® based composite specimens. The measured crack growth rates are taken as the average of the rate over the first 10–15 mm of crack growth, where a relatively constant value of K_I can be assumed. It is clear that the $\overline{\mathrm{ECR}}$GLAS® fibre based composites show a superior performance under stress corrosion conditions as compared with E-glass fibre composites.

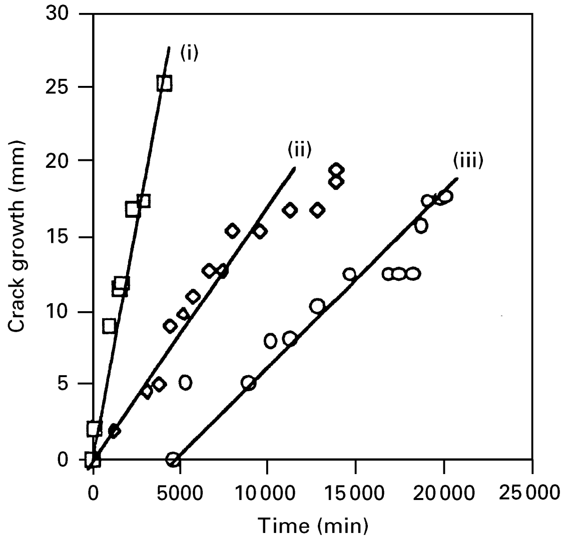


Figure 6 Variation of crack growth E-glass/DERAKANE 411-45 resin based composites tested at 80 °C with time for different values of K_I of; (□) Log $K_I = 0.77$ MPa m$^{1/2}$, (◇) Log $K_I = 0.69$ MPa m$^{1/2}$ and (○) Log $K_I = 0.65$ MPa m$^{1/2}$. The crack growth rate, (da/dt), calculated from the slope of the continuous line were for (i) 1.1×10^{-7} m s^{-1}, (ii) 3.3×10^{-8} m s^{-1} and (iii) 1.6×10^{-8} m s^{-1}.

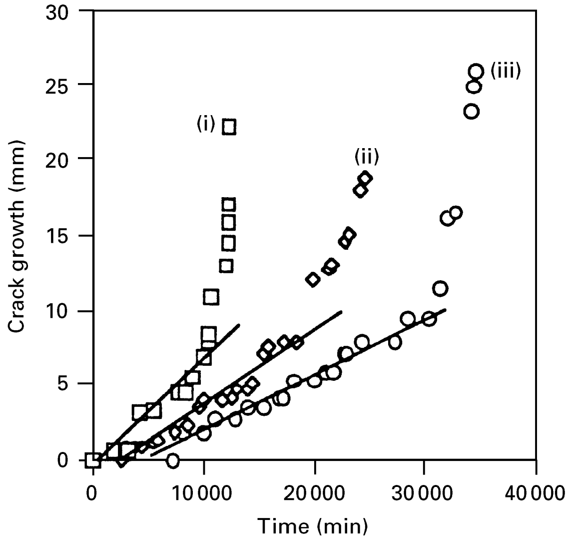


Figure 7 Variation of crack growth $\overline{\mathrm{ECR}}$GLAS®/DERAKANE 411-45 resin based composites tested at 80 °C with time for different values of K_I of; (□) Log $K_I = 1.32$ MPa m$^{1/2}$, (◇) Log $K_I = 1.22$ MPa m$^{1/2}$ and (○) Log $K_I = 1.08$ MPa m$^{1/2}$. The crack growth rate, (da/dt), calculated from the slope of the continuous line were for (i) 1.38 m s^{-1}, (ii) 1.2×10^{-9} m s^{-1} and (iii) 6.4×10^{-9} m s^{-1}.

The data generated by Price and Hull [16] are plotted in Fig. 8a, using similar TDCB specimens with 0.6 M hydrochloric acid at room temperature. All of their tests were made on unidirectional E-glass laminates with a range of polyester resins: a general purpose isophthalic resin, Crystic® 272 as the standard matrix; Crystic® 272 modified by the addition of 30 wt % Crystic® 586 and 20% Crestomer NV 1080 to produce two flexibilized versions; and Beetle® 870 a very brittle chemically resistant hexahydrophthalic anhydride (HET)-acid based polyester. Data for all these composite systems follow similar curves of increasing measured crack growth rate, da/dt, with increasing stress intensity K_I (as is shown in Fig. 8a). The relative positioning of the room temperature data

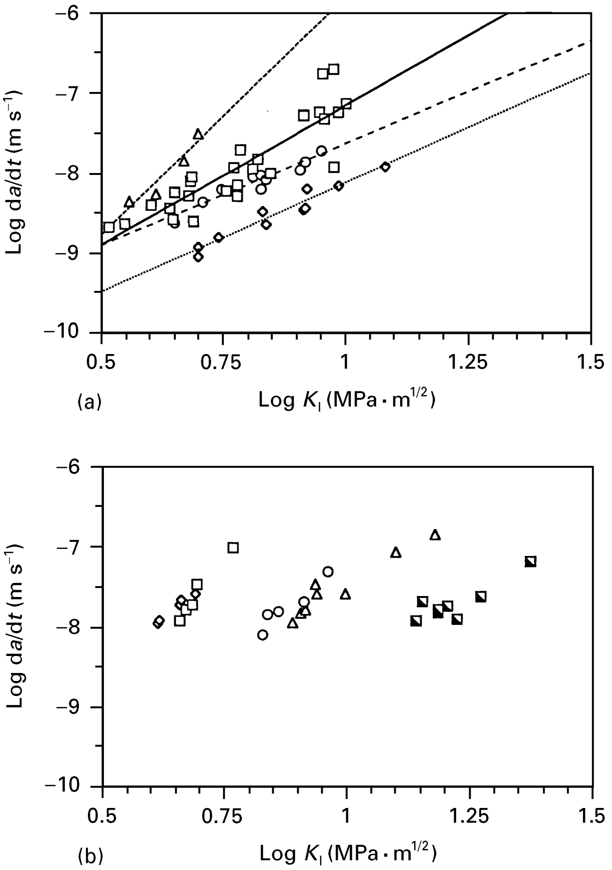


Figure 8a Variation of crack growth rate with applied stress intensity for four composite systems tested at room temperature in 0.6 M HCl. (Results from Price and Hull [16]). Data are presented for (□) E-glass/Crystic 272, (◇) E-glass/Crystic 272 + 30% Crystic 586, (○) E-glass/Crystic 272 + 20% NV1080 and (△) E-glass/Beetle 870.
Figure 8b Crack growth rate versus applied stress intensity for five unidirectional GRP systems. Tested in 1 M HCl at 80 °C were; (□) E-glass/DERAKANE 411-45 and (◇) E-glass/DERAKANE 470-30. Tested in 1 M HCl at room temperature were; (○) E-glass/DERAKANE 411-45 and (△) E-glass/DERAKANE 470-30 and (■) **ECR**GLAS/DERAKANE 411-45.

demonstrates the influence of the resin matrix on the stress corrosion cracking in these composite systems. Conversely, data for the brittle Beetle® 870 HET-acid polyester resin shows the poorest performance with rapid crack growth even at modest levels of K_I.

Data was obtained for the two DERAKANE resins with E-glass reinforcement, in 1 M hydrochloric acid at room temperature and it is shown in Fig. 8b. The data for the two DERAKANE resin systems overlaps with that of the Crystic® 272 resin, although the DERAKANE resin data lies generally at higher values of K_I. Fig. 8b also compares the crack growth rates for E-glass reinforced specimens based on the DERAKANE resins, tested at 80 °C and room temperature. It is evident that the E-glass reinforced specimens exhibited highly accelerated cracking at the higher temperature, showing similar behaviour to the Beetle® 870 resin/E-glass at room temperature. In contrast, the DERAKANE 411-45 resin based specimens tested with **ECR**GLAS® reinforcement, showed very high crack growth resistance even at 80 °C, considerably better than even the toughest of the experimentally toughened polyester resins tested at room temperature.

In the time scale of the programme, it was not possible to generate any failures with the DERAKANE resins/**ECR**GLAS® reinforcement at room temperature – the material was far too crack resistant.

Although the standard and flexibilized isophthalic polyester resins showed favourable ESCC resistance at room temperature in the form of virgin test specimens, the results do not reflect the changes that may take place over prolonged time in real industrial service. These polyester resins also have low values of deflection temperature under load, (DTUL) and would therefore be limited to low operating temperatures by the design standards. The addition of flexibilizing additives only further compounds this effect, making the polyester resin data interesting but of little practical significance for operating safely at elevated temperatures for long design lifetimes in demanding industrial applications. The DERAKANE resins in the current study are the de-facto standard for corrosion resistant industrial GRP and have been selected for their balance of practical toughness and high DTUL, and 30 year track record.

5. Fractography

Close examination of the stress corrosion fracture surfaces using scanning electron microscopy showed some of the features typically associated with the ESCC failure process. The striking nature of these fracture surfaces can be seen by comparing Figs 9 and 10. For the E-glass fibre based composites tested at 80 °C in 1 M hydrochloric acid, a planar fracture surface was produced with little or no fibre pull-out. Previous work on aligned GRP composites under similar stress corrosion conditions at room temperature showed the same characteristic features [16, 17]. The fracture surfaces were essentially planar, but did show some step formation on the fracture surfaces produced by transverse cracking along the fibre axis, this being evident in the crack growth data in Fig. 6. There was no evidence of individual fibre pull-out, which is characteristic of conventional mechanically induced fracture in non-corrosive environments. There was no discernible difference between the fracture surfaces of the DERAKANE 411-45 and DERAKANE 470-30 resin based E-glass composites, as under the acidic conditions it is the performance of the E-glass fibres which *dominates* the overall fracture behaviour of the composite.

Fracture surfaces of individual E-glass fibres that had failed at 80 °C were invariably smooth and mirror-like. This is a consequence of the low values of applied crack tip stress intensity imposed during the high temperature tests on the E-glass fibre composites. Similar fracture surfaces were observed by Hogg and Hull at crack initiation sites in stress corrosion tests using pipe shaped specimens, where the applied stress intensities were also low [18]. In regions of the test specimens where the values of K_I had increased (i.e., beyond the first 15 mm of crack growth) the fracture surfaces on individual fibres began to exhibit regions of mist and hackle in addition to the mirror zone, reflecting the increases in local fibre fracture stress

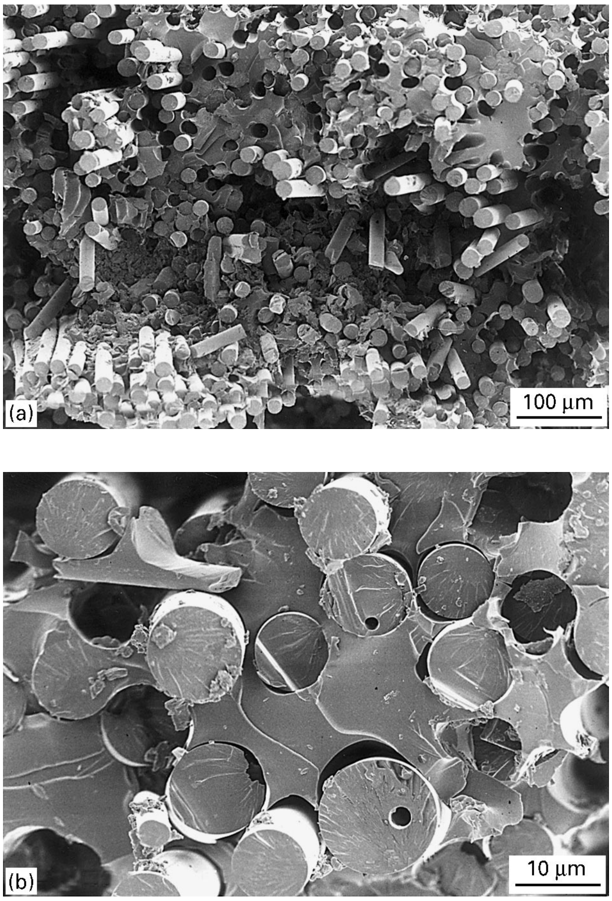


Figure 9 Scanning electron micrographs of DERAKANE 411-45 resin/ $\overline{\textbf{ECR}}$GLAS® based composite sample tested at 80 °C in 1 M hydrochloric acid: (a) region at lower magnification showing the 'roughness' of the fracture surface, (b) area at higher magnification showing directions of crack propagation and matrix river lines.

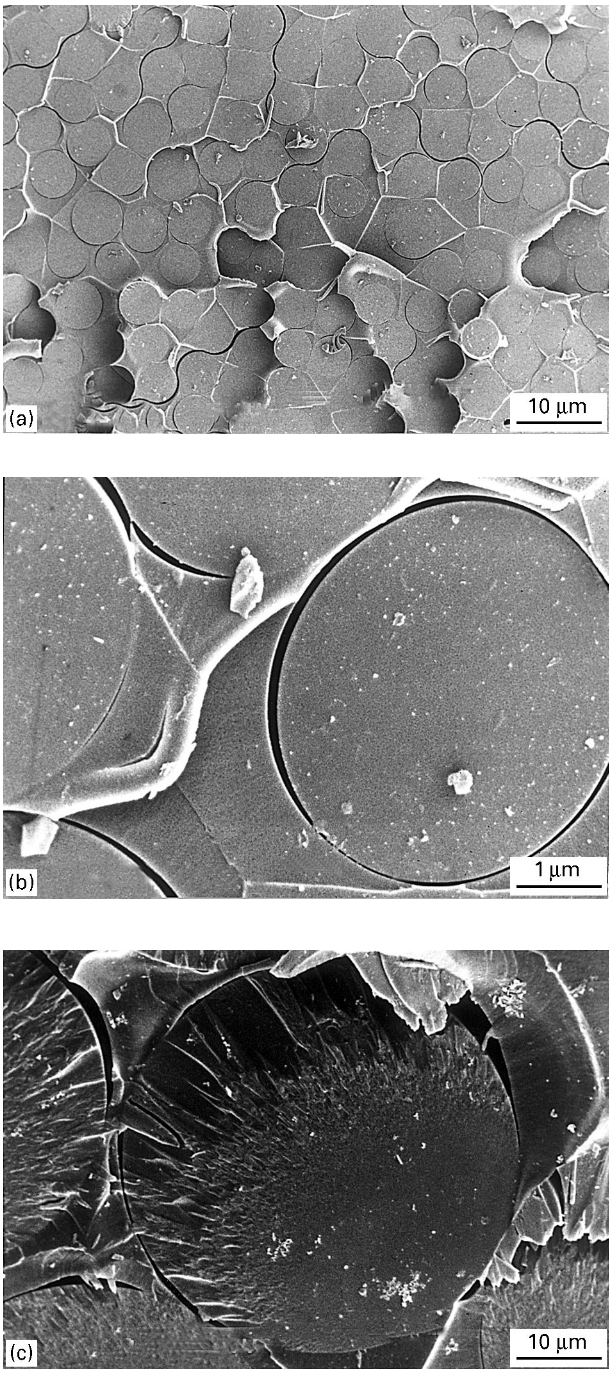


Figure 10 Scanning electron micrographs of DERAKANE 411-45 resin/ E-glass based composite sample tested at 80 °C in 1 M hydrochloric acid: (a) smooth fracture surface in the nucleation region; (b) same region at higher magnification showing the featureless fracture surface indicating low stress fracture, polygonal lines and interface, (c) details of a fibre fracture at higher values of K_{I}.

with increased crack length in the test specimen. Polygonal lines were observed on the resin matrix areas of the fracture surfaces of the DERAKANE 411-45 and DERAKANE 470-30 resin based composites, which formed a network around the fibres as is shown in Fig. 10c. Matrix cracks extended only part-way across the web of resin between fibres, so that fibres ahead of the crack were to some extent protected from the corrosive environment. Stress-induced microcavitation or cracking has been postulated to explain the fact that the attacking species achieve access to the glass fibres [19].

A totally different fracture surface was observed for the DERAKANE 411-45 resin/$\overline{\textbf{ECR}}$GLAS® fibre based composite system. Extensive pull-out and bush-like surfaces were produced, even under elevated temperature ESCC conditions. A noticeable feature of stress corrosion cracking at 80 °C was the lack of helical or longitudinal microcracks on the external surface of the $\overline{\textbf{ECR}}$GLAS® fibres, which is a well documented phenomenon for E-glass fibres subjected to acid corrosion [20]. Debonding was evident around the fibres.

6. Discussion

The results obtained for the E-glass fibre reinforced composites were just as would be expected based on prior experience of testing at room temperature. The increase in testing temperature from 20 to 80 °C resulted in a significant increase in the crack growth rates. This indicated that the influence of temperature on the mechanism of E-glass fibre fracture under acidic conditions out weighed other contributions from the toughness of the resin matrix. Previous work [16] (albeit at 20 °C) has shown that matrix toughness (and in particular the ability of the resin to yield at the crack tip, so reducing stress on the fibre) can be a major factor in controlling crack growth rates in E-glass fibre composites.

The DERAKANE resin/$\overline{\textbf{ECR}}$GLAS® reinforced composite performed significantly better than the

E-glass fibre composites. No tests could be completed with **ECR**GLAS® specimens at room temperature. This was partly because the fibres were so much more resistance to acid attack that the specimens required much higher applied loads. This resulted in premature failure in the loading arms of the specimen via a different failure mechanism (effectively transverse creep rupture). It is possible that if sufficient time had been available, tests at room temperature at low applied stress (i.e. of the same order as used for the E-glass fibre specimens) may have resulted in valid ESCC cracking, but this is only speculation.

Even at the elevated temperature of 80 °C, high applied loads (and hence, imposed crack tip K_{I} values) were still required to generate crack growth rates that could be readily measured. However, the loads required to cause measurable crack growth rates are likely to be way in excess of typical design loadings, further emphasizing the resistance to ESCC of these DERAKANE 470-30 resin/**ECR**GLAS® fibre composites and its suitability for long term use at high temperatures.

The nature of the fracture surfaces in these specimens (rough and highly crack resistant), was significantly different to those observed in similar E-glass composites, (flat and brittle). This reflects the significant change in composite failure process due to the enhanced acid resistance of the fibre reinforcement and perhaps also partly due to the higher levels of applied K_{I} required to cause fracture in the **ECR**GLAS® composite. The greater the applied K_{I} on the specimens, the greater the likelihood of interfacial cracking occurring simultaneously with fibre fracture, leading to fibre pull-out and a 'rougher' appearance of the fracture surface. The increased fracture surface roughness could also reflect a reduced level of interfacial bonding in the composite system, such as a time dependent attack on the interfacial bond. No direct experiments have been conducted to compare the initial bond strengths in E-glass and **ECR**GLAS® reinforced laminates.

The performance of the DERAKANE resin/**ECR**GLAS® fibre composites at 80 °C was similar to, if not superior to the best results obtained by Price and Hull for E-glass composites at 20 °C, using experimental toughened polyester resin systems [16]. The results clearly indicate the suitability of the DERAKANE resin/**ECR**GLAS® fibres for use in composite structures operating at elevated temperatures in acidic environments as found in modern flue gas desulphurization and waste incineration gas cleaning plants. Design procedures used with E-glass reinforcement for use at 20 °C should be directly applicable to the DERAKANE resin/**ECR**GLAS® fibres for use under these more arduous service conditions.

7. Conclusions

(1) Raising the temperature from 20 °C to 80 °C under acidic conditions, increases the rate of stress corrosion cracking in E-glass composites. While resin toughness does play a role in modifying crack growth rates, the greatly accelerated fibre attack at the higher temperature outweighed any positive effect from toughening of the matrix resin.

(2) DERAKANE resin/**ECR**GLAS® fibre composites provide dramatically superior resistance to stress corrosion cracking and at elevated temperatures (80 °C) are superior to conventional E-glass fibre composites tested at 20 °C based on the same resin.

(3) It should be noted that while the role of epoxy vinyl ester resin has not been explicitly examined in this paper, these matrices are a pre-requisite for high temperature stress corrosion resistance.

Acknowledgements

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References

1. S. W. TSAI, in "Proceedings of the fifth symposium on Naval Structural mechanics, Mechanics of composite materials", 1967, Eds F. W. Wendt, H. Liebowitz and N. Perrone (Pergamon Press, Oxford, 1970) 749.
2. P. J. HOGG and D. HULL, in "Developments in GRP Technology", Vol. 1, ed. B. Harris (Elsevier Applied Science, London, 1983) pp. 37–90.
3. F. R. JONES, J. W. ROCK and J. E. BAILEY, *J. Mater. Sci.* **17** (1983) 1059.
4. W. S. CARSWELL and R. C. ROBERTS, *Composites* **11** (1980) 95.
5. C. LHYMN and J. M. SCHULTZ, *J. Mater. Sci.* **18** (1983) 2923.
6. K. FRIEDRICH, *ibid.* **16** (1981) 3292.
7. M. O'CONNOR, in "GRP Vessels and Pipework for the Chemical and Process Industries" (Mechanical Engineering Publication, London, 1983) pp. 56–79.
8. L. S. NORWOOD and P. J. HOGG, *Composite Structures* **2** (1984) 1.
9. J. N. PRICE, Ph.D. thesis, University of Liverpool (1985).
10. M. F. KANNINEN, E. F. RYBICKI and H. F. BRINSON, *Composites* **8** (1977) 17.
11. J. M. WHITNEY and R. J. NUISMER, *J. Composite Mater.* **8** (1974) 253.
12. G. R. IRWIN and J. A. KIES, *Welding Journal Research Supplement* **33** (1954) 193s.
13. P. C. PARIS and G. G. SIH, "Fracture toughness testing and its applications" ASTM STP 381 (American Society for Testing and Materials, Philadelphia, PA, 1964) pp. 30–83.
14. G. G. SIH, P. C. PARIS and G. R. IRWIN, *Int. J. Fract. Mech.* **1** (1965) 189.
15. P. J. HOGG, J. N. PRICE and D. HULL, in Proceedings of 39th Annual Conference, Reinforced Plastics/Composite Institute, The Society of the Plastics Industry, Inc., session 5-C (New York, 1984).
16. J. N. PRICE and D. HULL, *Composite Sci. Tech.* **28** (1987) 193.
17. P. J. HOGG, *Progress in Rubber and Plastic Technology* **5** (1989) 112.
18. P. J. HOGG and D. HULL, *Metal Science* **14** (1980) 441.
19. F. R. JONES, *J. Strain Analysis* **24** (1989) 223.
20. A. K. BLEDZKI, G. W. EHRENSTEIN and A. SCHIEMANN, *Kunststoffe* **79** (1989) 416.

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