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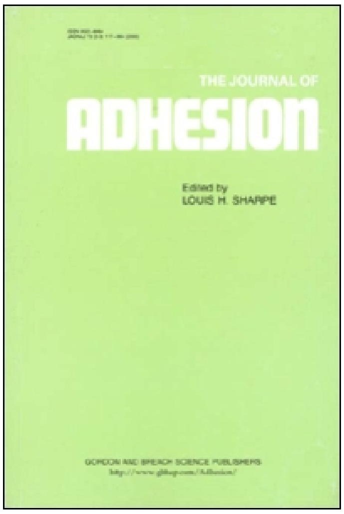

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Environmental Ageing of Adhesively-Bonded Joints. II. Mechanical Studies

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The effects of exposure to moisture on the mechanical properties of a series of adhesively-bonded structures are reported. Changes observed in the maximum load, shear modulus, strain at maximum load, fracture energy, fracture toughness and stress are discussed and correlated with variation of the dielectric parameters. An initial increase in fracture toughness observed in the joints correlates well with the uptake of moisture having led to a lowering of the glass-rubber transition temperature. Differences in the ultimate strength and energy to failure for different surface pretreatments are observed. Loss in mechanical properties observed over the period of the study are paralleled by changes in the dielectric properties of the joints.

Keywords: Mechanical studies; environmental ageing; fracture toughness; fracture energy; maximum load; shear modulus

INTRODUCTION

Adhesively-bonded structures are widely used in aircraft manufacture and a number of studies exist of the effects of moisture on their

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mechanical properties [1]. Cotter [2] has reported trials on adhesive joints exposed to hot/wet and hot/dry climatic conditions in Australia; Brewis and Comyn [3–5] have conducted parallel studies of hot/wet ageing using a humidity cabinet. In both studies, dramatic loss of bond strength was observed when the joints were aged in the presence of moisture. These and subsequent studies have stressed the importance of moisture in bond failure; however, the precise mechanism of action of ageing is still not completely understood. Moisture ingress will both change the mechanical properties of the adhesive and can lead to attack of the interfacial oxide. The interfacial oxide is formed to promote adhesion through a lock and key mechanism and is generated from a thoroughly clean surface which is subjected to vacuum blasting, etching and anodising. The anodising process is undertaken to improve the quality of the joint and is particularly important for epoxy-based adhesives used in aircraft design. The use of high frequency dielectric measurements to monitor water ingress into joint structures has been reported previously [6] and in this companion paper an attempt is made to correlate changes in the dielectric and mechanical properties on ageing of bonded joint structures.

Understanding of the stress distribution within a joint is important in designing its geometry and structural efficiency. However, the durability of a joint may be influenced by the environment in which it is used, the type of adhesive and nature of surface pre-treatment. A number of methods have been proposed to calculate the stress distribution. They include those by Volkersen [7] and Goland and Reissner [8] which assume the adhesive to behave as an elastic bond when the joint is in tension. Hart-Smith [9] has produced a modified approach in which the joint initially undergoes elastic followed by plastic deformation. Most elastic theories predict that stresses will decrease as the bond line thickness increases [10]. In practice, bond line thicknesses are usually carefully controlled to lie in the range 0.008 inch to 0.014 inch (0.20 mm to 0.36 mm). If the thickness is allowed to increase beyond the upper limit a significant reduction in strength is observed. Photoelastic stress analysis has shown that the position of maximum stress depends on the edge shape [11]. Classical mathematical solutions predict the highest stresses should be near the ends of the joint, but are unable to take into account the influence of the introduction of a defect on these stresses. It is in the regions of maximum stress associated with the defect that failure occurs and the assumed boundary conditions tend to break down. In this paper consideration will be given to evaluation of both G_{Ic}, the critical shear stress, and K_{Ic}, the critical shear energy, and their variation with ageing as these are less subject to problems described above.

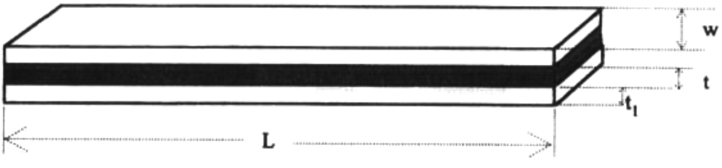
In a previous paper [6], the effects of exposure to moisture on the dielectric properties of aluminium adhesively-bonded joints which contained epoxy and phenolic adhesives and in which the metal was etched, etched and anodised, etched and anodised and primed as surface treatments, have been reported. This paper reports the corresponding investigation of the changes in the mechanical properties as a function of moisture exposure.

EXPERIMENTAL

Sample Preparation

Joints were prepared by British Aerospace at its Chester and Filton factories. The test pieces were constructed from two pieces of 4 mm thick (t_1) aircraft grade aluminium, to make joints 240 mm (L) × 25 mm (W) × ~8 mm (B), bonded using a 250 ± 50 μm (t) film adhesive which is optimum for development of bond strength. The details of the joint manufacture have been presented previously [6]. The joints conform to the ASTM-D-3433 specification for metal-to-metal laminate tests. Two film adhesive systems, phenolic and epoxy, and different surface treatments, as indicated in Table I, were used.

Details of the adhesives and the surface treatments are presented in the previous paper [6].



Picture of the joint.

Test Scheme

The test pieces were all initially tested dielectrically in their "as received" state and the data obtained are presented in the previous paper [6]. The mechanical properties of these pristine joints were also measured to determine that they were consistent with commercial specifications. Ageing of the samples was carried out in a large, temperature-controlled, water bath at 37°C ± 2°C filled with de-ionised water. Dry controls were stored in boxes at room temperature and samples examined as references for the dry ageing. Test pieces were removed after 21 days and at 70-day intervals and thereafter up to 600 days. A total of nine withdraws over a period of 600 days were carried out. Each test involved examination of five samples both dielectrically and mechanically and the data presented are the average of the data collected on these samples. Error bars are included in the graphs indicating the spread of the variation in the data observed for these samples. During the latter part of the investigations a small group of samples from each set were subjected to accelerated ageing at 70°C. In the original design of the experiment, based on publicised literature data on similar joints it had been anticipated that failure of the joints would occur within the 600-day period; however, it became clear after about 400 days that this would not occur and a small number of joints were subjected to accelerated ageing. It is now clear that these "production" joints were significantly more durable than similar joints previously reported.

Mechanical Properties

Two types of mechanical test were undertaken; shear tests in a lap joint configuration subject to tensile loading, and Mode I crack opening tests.

TABLE I Designation of the sets of samples generated for the study

SET	*ADHESIVE FILM*	*SURFACE PREPARATION*
1.	Phenolic	Etch
2.	Phenolic	Etch + Anodise
3.	Epoxy	Etch
4.	Epoxy	Etch + Anodise
5.	Epoxy	Etch + Anodise + Primer

1. Shear Tests on Lap Joints

The shear test measurements were made on bonded test pieces where a lap configuration had been manufactured by cutting slots 25 mm apart alternately in the upper and lower adherends and continuing through the adhesive bond line. The over lap generated is, thus, approximately 25 mm^2 in area.

This configuration corresponds to the requirements for the ASTM-D-3433 thick-adherend test specification. Tests were performed using a Zwick universal testing machine which provided autographic load/extension data, the extension being measured *via* an extensometer mounted on the specimen. This allowed determination of the ultimate tensile strength properties, the nominal shear modulus of the joint and the fracture energy (from the area under the load/extension curve). The shear stress levels within the joint were estimated from the measured loads, extensions and nominal shear moduli, *via* an elastic stress analysis theory.

2. Mode I Crack Opening Tests

The Mode I crack opening tests were carried out by propagating a crack incrementally in the adhesive layer, starting from a sharp notch produced by running a razor blade across the specimen on the bond line. The design of the loading rig permitted rotation of the load points, so that the adherend plates bent freely as cantilevers as the crack propagated. This test configuration complies with the ASTM-D-3433 test specification. For each test, the specimen was slowly loaded at a rate of 1.5 mm/min extension, to the point where the crack began to propagate. The loading screw mechanism was then stopped and the crack allowed to propagate naturally under "fixed grip" conditions, with the load dropping gradually to the point where crack arrest occurred. The crack lengths at initiation and arrest were measured, using a travelling microscope. The specimen was then reloaded in the same manner to produce further data for propagation and arrest loads, together with corresponding crack lengths. Five propagation and arrest measurements were normally achieved on each specimen. Loads and extensions were measured autographically and also with respect to a time base during the natural propagation phases.

From these data, relevant toughness parameters (G_{Ic}) for propagation and arrest were calculated, using the theory in the following section. This procedure was applied to phenolic and epoxy samples aged for 21, 350 and 600 days.

THEORY

Linear Elastic Solution for Shear Stress in Plate

Shear stress results in the lap joint tests are quoted in terms of the nominal or average stress given by

$$\tau = P/bl \tag{1}$$

where P is the applied load, b is the joint width and l is the joint length. The adhesive shear stress distribution $q(x)$, where x is measured from the mid-point of the joint, is given according to Ref. 12 by:

$$q(x) = \frac{P\eta}{b\lambda \sinh(\lambda l)}\left[\frac{E_1 t_1}{E_2 t_2}\cosh \lambda x + \cosh \lambda(1 - x)\right] \tag{2}$$

where $\eta = G_a/t_a t_1 E_1$ and $\lambda^2 = G_a/t_a((1/E_1 t_1 + 1/E_2 t_2))$; with E_1 and E_2 the Young's moduli of the respective adherends, t_1 and t_2 the respective thicknesses and G_a the shear modulus of the adhesive. The adhesive modulus is not known directly but can be determined inversely by equating the overall joint extension calculated through Equation (2) to the experimentally-measured joint extension.

Mode I Crack Opening Analysis

The compliance $C = \Delta/P$ of the adherend plates, treated as cantilevers, is given by [13, 14]:

$$C = \frac{2a}{3EI}\left[a^2 + \frac{4}{5}t^2\right] \tag{3}$$

where a is the crack length measured from the tensile load axis to the tip of the crack, E is the Young's modulus of the adherends, I is the second moment of area of the cantilever and t is the adherend thickness. The first term in this equation represents the bending deflection of the cantilever and the second term, arising from the shear deflection, is relatively small. The equation is very similar to a relationship derived by Ripling, Mostovoy and Patrick [15], *viz*:

$$C = \frac{2}{3EI}[(a + t_a)^3 + at^2] \tag{4}$$

In the present case, the thickness of the adhesive layer is relatively small and Equation (3) is therefore adequate.

The strain energy release rate, G_I, can be determined experimentally in a linear elastic case through the variation of compliance with crack length, in the form:

$$G_I = \frac{1}{2}P^2\frac{\partial C}{\partial a} \tag{5}$$

and making use of Equation (3), this becomes

$$G_I = \frac{4P^2}{Eb^2t^3}(3a^2 + t^2) \tag{6}$$

Hence, the initiation and arrest toughnesses, G_{Ic} and G_{Ia}, can be found from the maximum and minimum loads in the incremental tests and the corresponding crack lengths.

RESULTS AND DISCUSSION

Shear Test Results

It is evident from Equation (2) that the stress distribution in the joint is non-uniform and depends on the elastic modulus of the adhesive. This could, if ignored, result in anomalous interpretation of the effects of ageing on the global failure loads and strains. A preliminary examination was carried out to establish to possible effect of stress variation along the joint and effect of change in modulus. Figures 1a–e give typical variation of elastic stress for the phenolic resin, derived

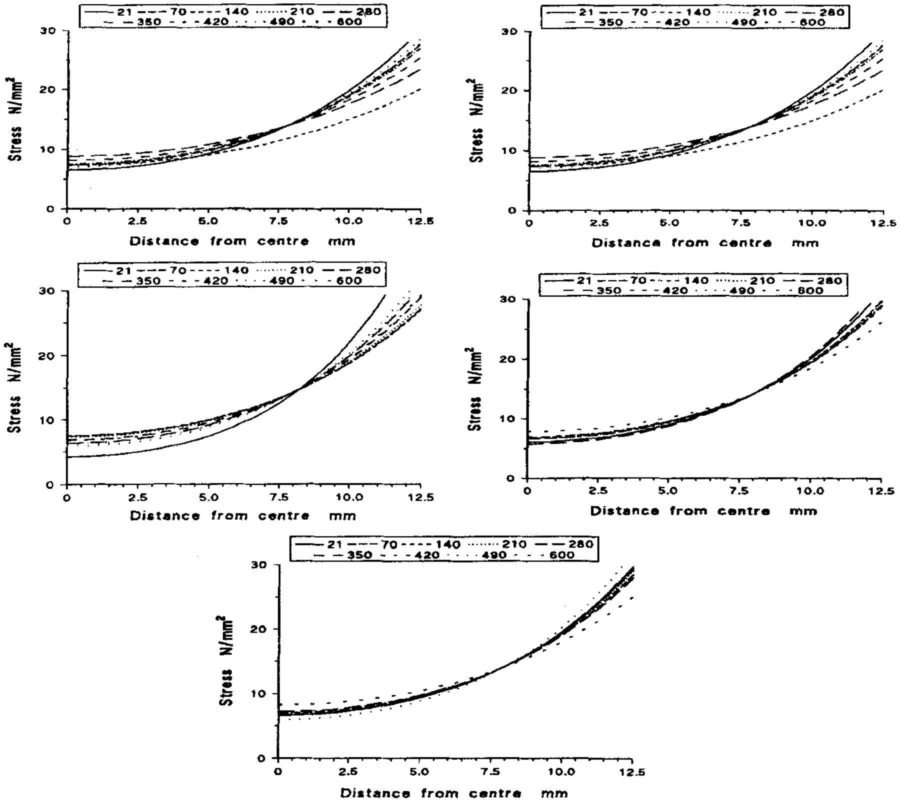


FIGURE 1 Variation of the elastic stress within the adhesive joint as a function of ageing period in days. a) phenolic - etched, b) phenolic anodised and etched, c) epoxy - etched, d) etched and anodised, e) etched, anodised and primed. Key:- ■ - dry, Δ - 40°C, * - 70°C.

through the experimental global modulus data and the inverse calculation procedure discussed earlier. This shows that although the stress does indeed rise towards the ends of the joint, the variation is not extreme and may in practice become more uniform if the adhesive yields before failure. In addition, the variation in modulus, due to ageing, does not have a marked effect for the configuration tested here. The most extreme example of the ageing effect, (Figs. 1a–e) occurs early in the ageing scenario. Similar curves were obtained for all the samples and only data for the phenolic resin are presented here.

No significant reduction in the maximum load to failure was observed for all the samples over the entire ageing period, Figures 2a–e. The exceptions were the phenolic etched-and-anodised samples where the dielectric data had indicated a high level of water uptake was occurring. The uptake of water has apparently little effect, in general, on the maximum load to failure and a close correspondence is observed between the wet and dry samples. However, all samples when subjected to accelerated ageing at 70°C exhibited a significant loss of strength. Use of the higher temperature would reduce the difference between the ageing temperature and the effective glass transition temperature of the plasticised adhesive, accelerating stress relaxation as well as attack of the interfacial oxide. The dielectric data indicate that at the higher temperature changes in the nature of the oxide interface could be occurring and this may, in part, be responsible for the loss of strength observed. An initial increase in the value of the shear modulus, Figure 3, obtained from the linear part of the plot of extension *versus* load, was observed especially in the case of the epoxy etch and etch/anodised joints. This effect can be attributed to plasticisation within the adhesive layer as a consequence of water uptake. The failure strain follows a similar but less marked trend, Figure 4. The clearest example, despite the large errors, is the phenolic etched/anodised joints. Once more, the data do not show significant deviations as would be reflected in the fact that only small changes in the interfacial component have occurred.

Fracture Test Results

Opening-Mode fracture provides a severe form of test for adhesive bonds. However, the results should be more independent of joint

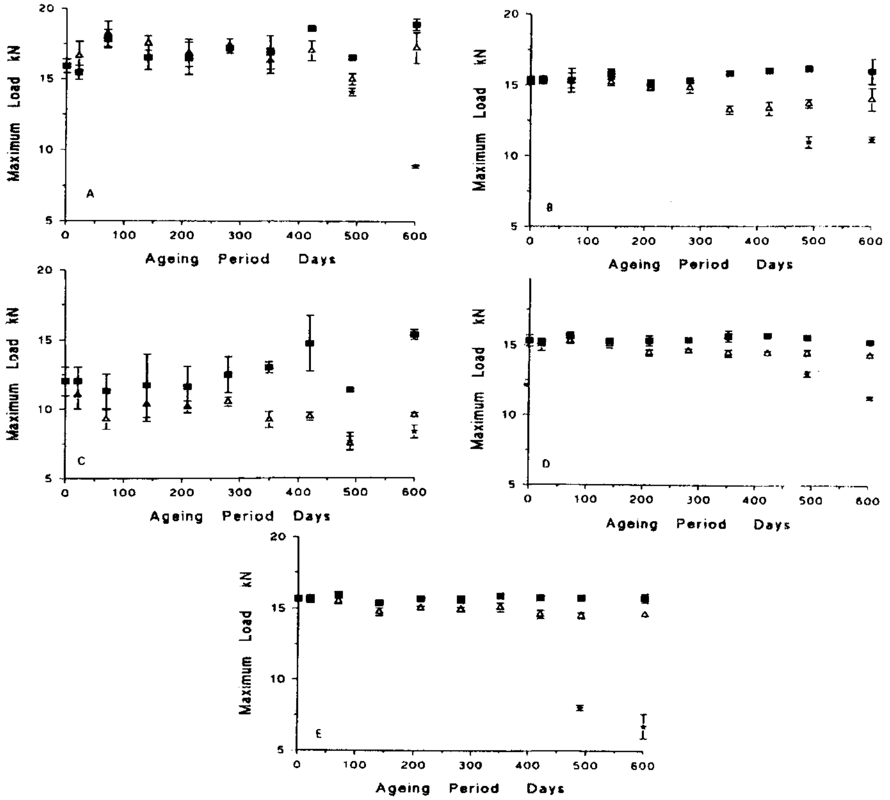


FIGURE 2 Variation of the maximum load as a function of ageing during shear testing. A) phenolic - etched, B) phenolic anodised and etched, C) epoxy - etched, D) etched and anodised, E) etched, anodised and primed. Key:- ■ - dry, Δ - 40°C, * - 70°C.

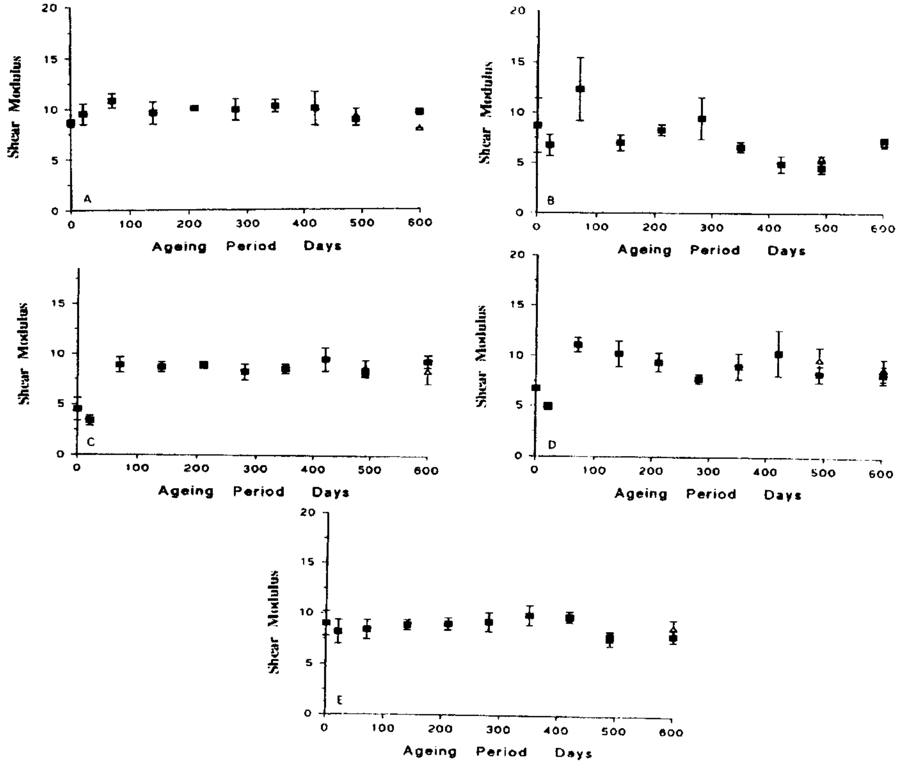


FIGURE 3 Variation of the shear modulus as a function of ageing. A) phenolic - etched, B) phenolic anodised and etched, C) epoxy - etched, D) etched and anodised, E) etched, anodised and primed. Key:- ■ - dry, Δ - 40°C, * - 70°C.

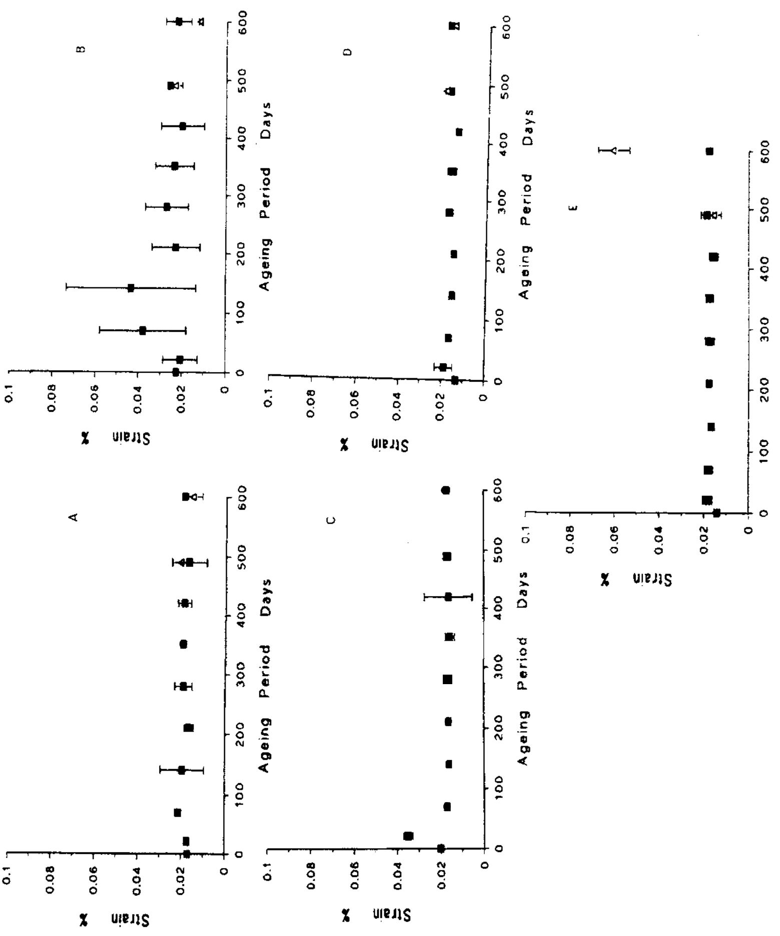


FIGURE 4 Variation of percentage strain at maximum load. A) phenolic - etched, B) phenolic anodised and etched, C) epoxy - etched, D) etched and anodised, E) etched, anodised and primed. Key:- ■ - dry, Δ - 40°C, * - 70°C.

configuration than the shear tests and, therefore, more transferable to other geometries. Specimen geometry-independence and good correlation between bulk adhesive toughness and measured toughness in a joint have certainly been shown for brittle adhesives [16–18]. Likewise, it has been shown that the adhesive thickness does not greatly affect the result. However, this is not true for tougher adhesives [19–20] where interactions between yielding, thickness and the triaxial constraint offered by adherends will come into play. An investigation of the interaction of joint width and adhesive thickness [20] has also shown that marked effects only occur in toughened epoxies.

In most of the present tests, three samples of a given joint, subject to the same environmental treatment, were tested, and up to five discrete initiation and arrest toughness values were measured on each sample. This gave some insight into the consistency, both of the testing method and of the samples themselves. The method used to calculate the toughness values from the concurrent loads and crack lengths (as in Equation (4)) was designed to achieve geometry-independence (in particular, with respect to the crack length). However, some variation in measured toughness for different crack lengths may be expected. This is due to interactions between specimen and testing machine compliances, and instabilities between the mechanical energy delivered by the test system and the fracture energy absorbed by the specimen.

Some general features of the test results are worth noting. The results showed consistency between the three samples, with a slight tendency to a rising toughness characteristic along the length of the sample. This was the most common trend where the toughness was not constant along the length, although in a few cases (mostly in brittle examples) a gradually falling trend could also be observed.

In the case of the samples aged for 600 days, distinct differences between the three samples were observed, although the consistency along the length suggests that a real difference in toughness between these samples had been revealed. This may, of course, be due to the path which the crack has followed in each case, relative to the position of the adhesive/adherend interface, although no evidence of this could be established from visual examination. In all cases except one (epoxy: etched, anodised and primed) failure occurred within the adhesive.

In order to simplify the comparison and interpretation of data, toughness levels at a standard crack length of 30 mm have been ex-

TABLE II Summary of the average values of G_{Ic} at upper load initiation and G_{Ia} at arrest of load and the maximum crack length before failure

Specimen	*Ageing period*	*Temperture of ageing*	G_{Ic} *at upper load initiation**	G_{Ic} *at arrest of load**	*Crack length*
	days	°*C*			*mm.*
phenolic-etched	21	40	160 ± 30	60 ± 10	32
	280	40	150 ± 50	100 ± 50	28
	600	40	175 ± 35	100 ± 25	34
	420/180	40/70	175 ± 10	75 ± 10	33
phenolic-etched + anodised	21	40	300 ± 90	100 ± 25	45
	280	40	300 ± 120	100 ± 40	43
	600	40	440 ± 150	150 ± 80	60
	350/250	40/70	250 ± 50	125 ± 25	35
epoxy-etched	21	40	1750 ± 500	1200 ± 400	37
	280	40	1200 ± 300	800 ± 200	38
	600	40	600 ± 100	400 ± 100	40
	420/180	40/70	850 ± 150	550 ± 100	40
epoxy-etched + anodised	21	40	1500 ± 300	1000 ± 180	37
	280	40	550 ± 80	350 ± 75	35
	600	40	850 ± 200	550 ± 150	36
	350/250	40	600 ± 100	350 ± 70	37
epoxy-etched + anodised + primed	21	40	1050 ± 350	700 ± 160	36
	280	40	900 ± 300	750 ± 150	36
	600	40	750 ± 250	650 ± 150	35
	350/250	40/70	220 ± 50	150 ± 50	40

*range of values; in most cases a smooth change was observed with increasing crack length. The ± values are indicative of the range of the variation.

tracted and are listed in Table II. The mean toughness value at that crack length and the range of values between samples are shown. The final crack length before complete failure also gives some impression of toughness, as it is difficult to sustain incremental growth in a brittle joint up to a large crack size.

In summary, the toughness of the phenolic joints tends to increase with ageing (from a low starting point) or, at worst, stays constant. However, in the epoxy joints, the toughness tends to decrease with ageing (from a high starting point). Extra surface preparation treatments also had contradictory results for the two materials. Anodising apparently improved the performance of phenolic adhesives aged at 40°C, whereas extra surface treatments tended to reduce the resistance of epoxies to crack propagation.

There appears to be no effect of plasticisation in phenolic adhesives in the "etch only" condition (the toughess remains constant with ageing). However, in the anodised samples, the toughness tends to increase with exposure but falls off under high temperature ageing – all suggesting that the greater affinity for moisture may have effects on the interface.

Epoxies show a larger scatter in data during the initial stages of ageing, but this scatter tends to decrease as the surface treatment becomes more effective, albeit at the expense of reduced toughness. The epoxy bonds were much more consistently manufactured in terms of bondline thickness, which makes the initial scatter difficult to attribute to a specific cause. The low values found in epoxies do, however, correlate with the shear test results where interfacial failure was found. The fracture toughness of epoxies exposed to moisture does, therefore, depend on the nature of the surface treatment, whereas phenolics are less sensitive.

The dielectric data show that joints manufactured from anodised adherends for the vinyl phenolic adhesive had the higher water content relative to these joints manufactured from adherends with the etch-only pretreatments. The higher water uptake does appear to contribute to the higher fracture toughness value. The possibility of water diffusing through the adherend–adhesive interface must be considered and it would appear, therefore, that this, in part, explains the observed effects. However, the differences in behaviour of the phenolic and epoxy are not explicable on this basis. In the case of the epoxy, a polyester or nylon yarn carrier is used and edge diffusion down the fibres provides an alternative mechanism for moisture ingress. The yarn can also influence the crack propagation measurements acting as "crack stoppers". The influence of the yarn may change as the joint is subject to ageing causing the bond between the adhesive and itself to deteriorate, resulting in a line of weakness in the adhesive layer. In part, the data imply that the long term fracture data may be influenced by the presence of the woven carrier which forms part of the epoxy system but is not present in the case of the phenolic.

Fracture energy can be calculated from the area under the curve for the plot of load *versus* extension. In the case of the phenolics the fracture energy for the aged sample increased, Figure 5 and this can be related to an increase in plasticisation on ageing as a result of

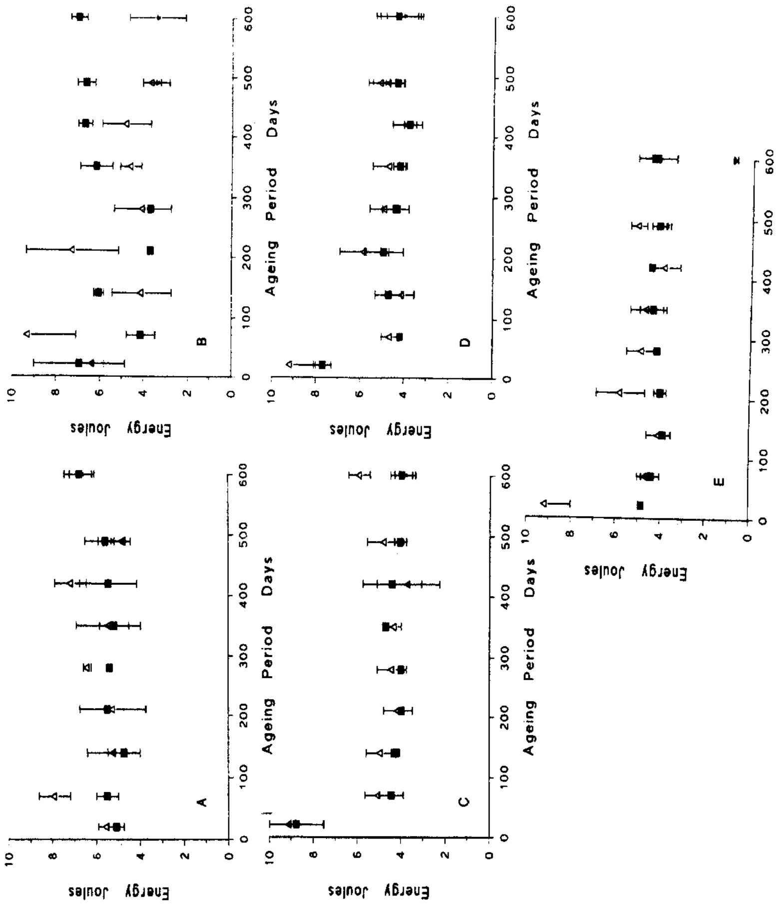


FIGURE 5 Energy required for fracture as function of aging. A) phenolic - etched, B) phenolic anodised and etched, C) epoxy - etched, D) etched and anodised, E) etched, anodised and primed. Key:- ■ - dry, Δ - 40°C, * - 70°C.

water absorption. Figure 5 (Phenolic-Etch + Anodise) shows that there is a decrease in fracture energy half-way through (200–300 days) which could be due to the way the force is transmitted through the phenolic resin to the substrate which is anodised. Epoxy bonds are stronger and need a larger energy to fracture in the absence of ageing, Figure 5. The exception to this trend is the set where a primer has been used which showed a fairly uniform fracture energy, Figure 5. After the intial drop in fracture energy all epoxy joints tend to stabilise around 5 Joules for the samples aged at 40°C. Optical examination of the failure surfaces indicated that for short exposures no interfacial fracture occurred during shear testing of bonds; however, interfacial failure did occur on extended exposure and use of elevated temperatures and can be attributed to dewetting of the interfacial layer.

CONCLUSIONS

Loss of bond strength was not as significant in these tests as compared with data obtained from the literature [1–3]. A certain amount of water within a bond actually increases the amount of load it can withstand prior to fracture. Phenolic bonds do not show any change in the energy required for fracture except in the extreme case of high temperature. In the case of epoxy bonds a significant decrease in fracture energy results on completion of 70 days of ageing. The fracture-mechanics-based measurements reveal several influences on ageing and surface preparation. In the case of phenolics which are already brittle, ageing has little effect, although there is a tendency for the speciemens with anodised surface preparation to show an increased toughness as the crack penetrates along the length. The superior initial toughness of epoxies suffers as a result of ageing; surface preparation reduces the scatter in performance but, again, does not increase the toughness. The dielectric data showed no evidence from these experiments of significant changes in the nature of the surface oxide layer and this implies that the loss of strength observed over this 600-day period may not be attributed to destabilization of the oxide interface but rather to changes occurring within the resin.

In conclusion, the combination of dielectric and mechanical data indicates that the toughening of the adhesive joints on exposure to water can, in part, be attributed to water leading to plasticisation of the adhesive. The joints maintained their strength up to 600 days and there was little evidence in these systems of significant conversion of the surface oxide to hydroxide. The observed changes in the adhesive properties must, therefore, be associated with changes in the mechanical properties of the resin and possibly stress relaxation rather than chemical changes in the oxide layer. Combination of the dielectric and mechanical measurements can provide a new insight into the mechanisms of ageing in adhesively-bonded structures. The dielectric method allows *in situ* assessment of the water content as well as providing information on the nature of the interfacial oxide layer. Previously, this information could only be inferred or obtained by analysis of fractured joints. Further research into the long term ageing of adhesively-bonded structures is continuing using a combination of dielectric and mechanical analysis. This paper indicates the new insight which can be obtained on ageing in adhesively-bonded structures by moni-toring dielectric and mechanical data.

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