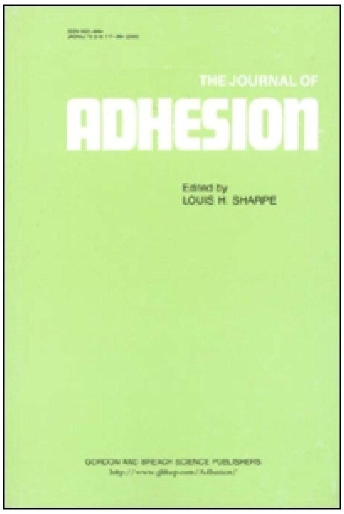

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The Effect of Ageing and Environment on the Static and Fatigue Strength of Adhesive Joints*

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This paper describes the results of a durability programme designed to test the effects of ageing and environment on the performance of adhesive joints. Specimens were kept under a variety of loading and environmental conditions and the paper reports results of static and fatigue tests after 8–9 years storage. Some adhesive joints showed excellent durability performance, while others were adversely affected by the environment, particularly high humidity and natural exposure. It was found that the effect of ageing on static and fatigue performance is not necessarily the same.

KEY WORDS durability; ageing under load; water absorption; high humidity; failure mode; effect of natural environment on adhesive joints; repair and maintenance of bridges; adhesive joints; fatigue.

INTRODUCTION

While structural adhesives are used in aerospace and automotive industries, apart from a number of repair and maintenance operations (*e.g.* bonding steel plates to concrete), they have yet to make any great impact in civil engineering. This is despite the fact that they offer a number of significant advantages over other joining techniques. In particular, the stress concentrations are much reduced when compared with mechanical fasteners, and the fatigue performance is superior to that of welded joints. One of the reasons for the reluctance to use structural adhesives in civil engineering is concern over the durability properties of adhesive joints. Many researchers (*e.g.* Hahn and Yi,[1] Fay and Maddison,[2] Cowling *et al.*[3]) have studied the effects of ageing and environment on the performance of adhesive joints, but most of the studies were of limited duration and restricted to static tests. Brockmann[4] carried out work on aluminium joints in which the joints were subject to cyclical load and natural and artificial environments; the study concentrated on the effects of surface pretreatment. Some researchers (*e.g.* Kinloch[5]) developed models, based on the mechanisms and kinetics of attack, to predict the service life of adhesive joints using results from short-term experiments. Civil engineering

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structures may be expected to serve for periods up to 100 years, and even repair operations can be required to have a life of the order of thirty years. Therefore, before adhesives can be fully accepted in civil engineering it is essential that more comprehensive durability data become available, and that the effects of ageing and environment are better understood. Unfortunately, there is a dearth of long-term durability data on the performance of adhesive joints, and the effect of ageing under various environmental conditions on the fatigue performance of adhesive joints is much in need of investigation. Herzberg and Manson[6] suggested that the general phenomenon of ageing must be taken into account in any consideration of deformation and fracture in polymers. The process of ageing of adhesives may be accelerated by some environmental conditions such as high temperature, moisture, aggressive media and mechanical stress. The behaviour of a material under static and fatigue loading may be quite different, and therefore for a full understanding of the long-term effects of ageing and environment on the performance of adhesive joints, both static and fatigue loading tests are necessary.

To help answer the above concerns, the Wolfson Bridge Research Unit (WBRU) set up a durability programme in 1980 designed to study the long-term effects of ageing under various environments on the performance of adhesive joints (the programme was designed to last for 20 years).[7] The adhesives were typical of those which might be suitable for civil engineering applications. The specimens were kept under a variety of environmental conditions (some were stored under load, others were stored unloaded) and were tested under both static and fatigue loading after 8 to 9 years. The results of tests after environmental exposure were compared with control results obtained at the beginning of the programme. Ageing, environmental and adhesive type were all found to affect the results.

ADHESIVES AND JOINT TYPE

The adhesives used were all two-part, cold-cure epoxies and identified by the numbers 1, 2, 4, 6, and 14. Details of their formulation and properties can be found in Lark,[8] but they are briefly described below. The manufacturer of each material and their own product number are given in parentheses:

Adhesive 1 (Ciba-Geigy XD800)—a thixotropic paste cured with aliphatic polyamine hardener; with high strength, high fracture toughness and Young's modulus.

Adhesive 2 (Ciba-Geigy AV100 + HV100)—a high viscosity liquid resin cured with polyamide hardener; with moderate strength, low Young's modulus and low fracture toughness.

Adhesive 4 (Ciba-Geigy XD548 + HY941)—a filled paste cured with hardener of the aromatic polyamine type; with low joint strength, slightly greater toughness and Young's modulus than adhesive 2.

Adhesive 6 (Sika Sikadur 31)—a thixotropic paste cured with aliphatic polyamine adduct hardener; with generally similar mechanical properties to adhesive 1.

TABLE I
Adhesive properties

Adhesive	Wt gain %	Young's modulus kN/mm^2		Shear strength N/mm^2		Joint strength N/mm^2	HDT °C	Fracture toughness MPa/m$^{1/2}$
		Before	After	Before	After			
1	5.0	8.8	3.2	35	17	17	41	2.3
2	8.1	2.1	0.6	19	7	13	40	0.5
4	1.2	3.6	3.2	35	28	11	48	0.8
6	0.9	7.8	5.5	28	16	17	43	1.6
14	3.1	2.1	0.3	18	8	13	34	NA

Before and After refer to before and after immersion in water of a 60 × 12 × 2 mm specimen for 2500 sec$^{1/2}$/mm.

HDT is heat distortion temperature.

Adhesive 14 (Permabond EPP 411)—a paste cured with polysulphide hardener; with low strength, low Young's modulus.

Numerical values for the adhesive properties by Lark[8] are given in Table I (the joint strengths were obtained from the control results), where HDT refers to heat distortion temperature. These values were obtained using four-point beam tests; the beam was subjected to a maximum fibre stress of 1.81 N/mm^2 (in accordance with BS 2782) and the HDT was taken as the temperature required to produce a further 0.25 mm deflection. While HDT is not the same as glass transition temperature, it does give an indication of the susceptibility to temperature. Further details can be found in Lark.[8]

Double lap joints of the configuration shown in Figure 1 were used for this study. The thickness of the central adherend was made the same as that of the side overlaps to facilitate construction in large numbers from standard metal sections. Bright mild steel flats were selected for their flatter profile and absence of rolled edges. Surfaces were prepared by grit blasting using the standardised WBRU procedure,[7] namely the adherends were degreased by washing with detergent and water, rinsed with

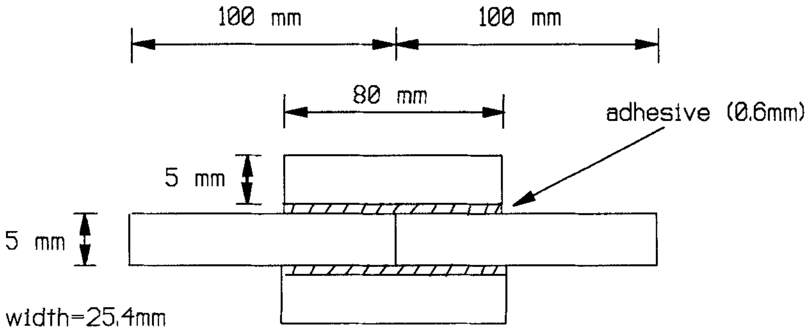

FIGURE 1 Geometry of double lap joint.

cold water, and then dried in a stream of warm air and with clean absorbent paper. Then they were grit blasted to grade Sa 2½ or 3 (Swedish Standard SIS 05 59 00—1967). A batch consisted of the manufacture of two 300 mm wide lap joints. Each "wide" lap was then cut (after at least 3 days curing) into a maximum of eleven individual 25.4 mm wide lap joints, neglecting edge strips. Piano wire was used to control the bondline thickness. All the specimens tested in the current study were cured at room temperature.

TEST PROCEDURE

Environmental Conditions

The specimens were stored under the following environmental conditions for about eight years:

A: Ambient laboratory temperature and humidity, laboratory clean air.
B: Hot room (45°C) at ambient humidity, laboratory clean air.
C: Cold room (−15°C) at ambient humidity, laboratory clean air.
D: Moderate humidity (60% RH) at ambient temperature, laboratory clean air.
E: High humidity (90% RH) at ambient temperature, laboratory clean air.
F: Natural exposure (roof of the structural laboratory), Dundee. Dundee is a coastal city. The relative humidity varies between 30 and 75%, annual rainfall is typically about 700 mm, and through the year the temperature varies between a few degrees below zero to a little over 20°C.

About half of the total specimens were sustained under load condition, a mean shear stress of 1 N/mm^2 being applied to the joints using constant load rigs designed by Hutchinson.[9] This value was chosen as it is a reasonable upper bound to the working stress level that the adhesive joint might be expected to experience in practice, *e.g.* in open sandwich bridge deck construction developed by the WBRU, Mays and Vardy.[10]

Static Tests

Some specimens were selected to test at the beginning as control results. All tests (static and fatigue) took place at room temperature. The static control tests were performed in a 10-ton capacity Avery testing machine.[11] The specimens were gripped in mechanical tensile wedge grips and loaded to failure at a rate of approximately 1 kN/minute. Two specimens were tested from each batch. The mean failure load was taken as the control result of that batch. The present static tests (*i.e.* those after 9-year environmental exposure) took place in an Instron 1196 testing machine at a displacement rate of 0.01 mm/minute.

Fatigue Tests

The fatigue control tests were performed using a hydraulic servo-control actuator of 160 kN dynamic load rating, mounted in a rigid loading machine.[11] The specimens were gripped in hydraulic tensile wedge grips mounted in pin joints and cycled under load control with a sinusoidal wave form. The load was measured using a strain gauge load cell of 160 kN rating and monitored by a digital monitor unit which held the peak value of the load (maximum and minimum value) for reading. The lap joints were tested in tension between a selected peak load and a minimum load equal to 10% of the peak. A dual trip unit was incorporated to ensure that the maximum and minimum load values did not deviate by more than 5% outside the selected range. If the load value was more than 5% outside the selected range, the unit would automatically stop the machine. 15 Hz was selected as the frequency of load cycling. The frequency was chosen so that it would not induce thermal failure, and to limit the test time. Due to the large number of specimens needed to be tested in the programme, the load range for each batch was selected in an attempt to produce failure within 1,500,000 cycles. The load range for each specimen was typically chosen to be 4–40% of the static failure load obtained for that batch. The purpose was to eliminate some of the effects of batch variability, but an adverse effect of this strategy is that different load ranges are applied to specimens made of the same adhesive, but coming from different batches. If a specimen survived 1.5 million cycles a new specimen from the batch was chosen and the load increased.

The present fatigue tests (*i.e.* those after 8-year environmental exposure) took place in two hydraulic servo-control actuators of 80 kN dynamic load rating with 25 KN and 50 KN load cell rating. Once the specimens were placed in the grips the load was slowly raised to the peak of the test range, it was then reduced to the mean load and the fatigue test was started. Further details can be found in Su.[12]

RESULTS AND DISCUSSION

Static and fatigue tests were carried out after the joints had been exposed for 8 to 9 years. The static strength and the fatigue life of the specimens were compared to those of the control specimens. Figure 2a shows the average of log (F/F_s) where F is the static strength in the most recent tests, and F_s is the static strength of the control specimens; Figure 2b shows log (F/F_c) where F is the fatigue life in the most recent tests, and F_c that of the corresponding control specimens. Figure 2 does not distinguish between the environment that the specimens were stored in, but does differentiate between those stored under load and those stored unloaded. The purpose of Figure 2 is to give a rough overall summary of the results. The results for each specimen tested are shown in Figures 3–7 where the effects of adhesive type, environment and loading are shown. Further details on the fatigue results can be found in Su *et al.*,[13] and on both the static and fatigue results in Su.[12]

While Figure 2 does mask the effects of environment it does immediately illustrate a number of features of the results; these are:

1. Adhesive 14 suffered a severe loss of static strength and fatigue resistance.
2. Adhesives 1 and 4 showed an overall improvement in both static strength and fatigue resistance.
3. Adhesives 1, 4 and 6 showed a significant improvement in fatigue resistance,

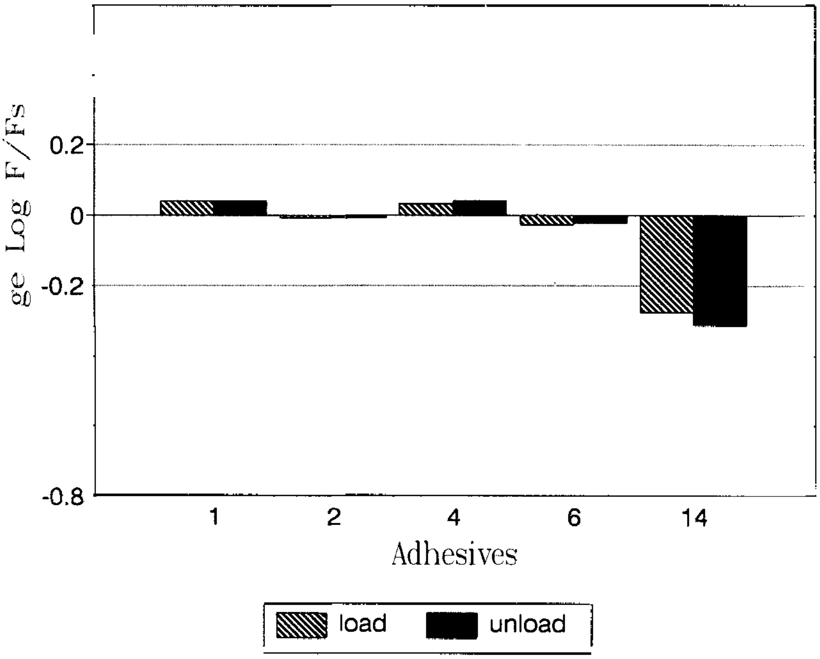

FIGURE 2a Average change in static strength.

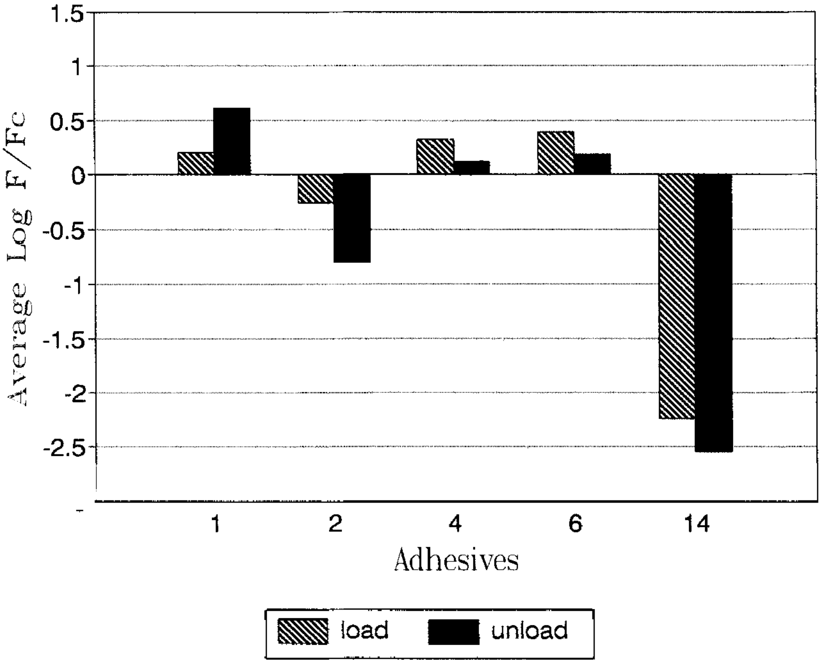

FIGURE 2b Average change in fatigue life.

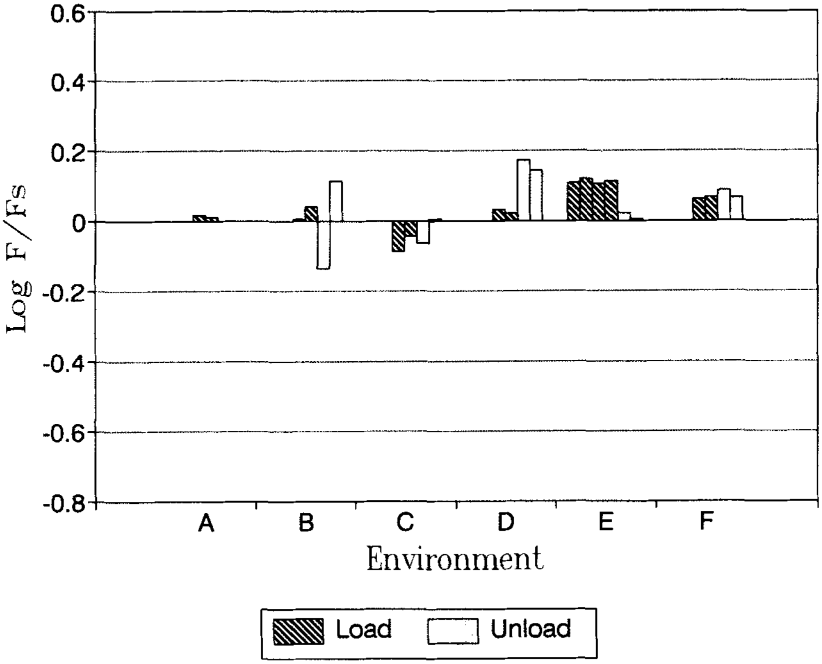

FIGURE 3a Static test results for adhesive 1.

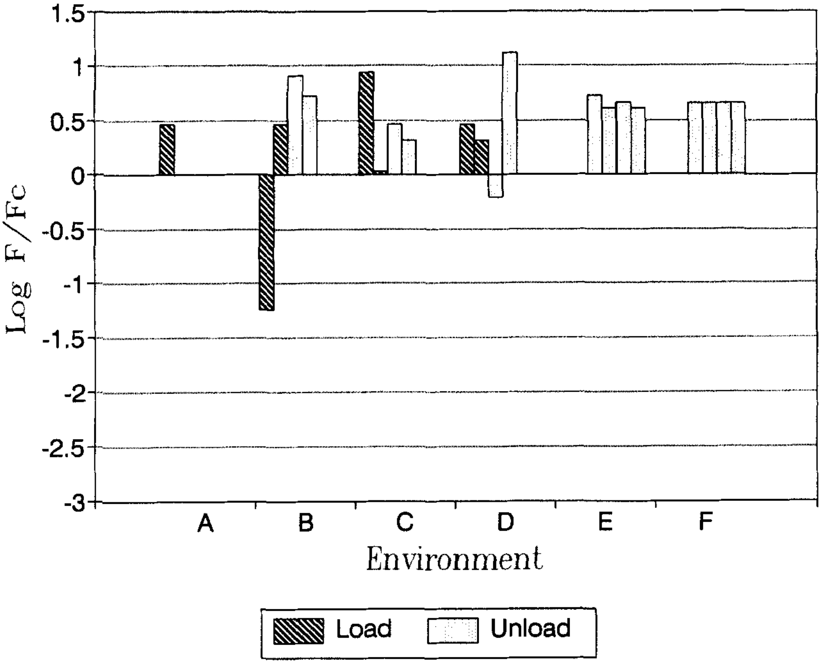

FIGURE 3b Fatigue test results for adhesive 1.

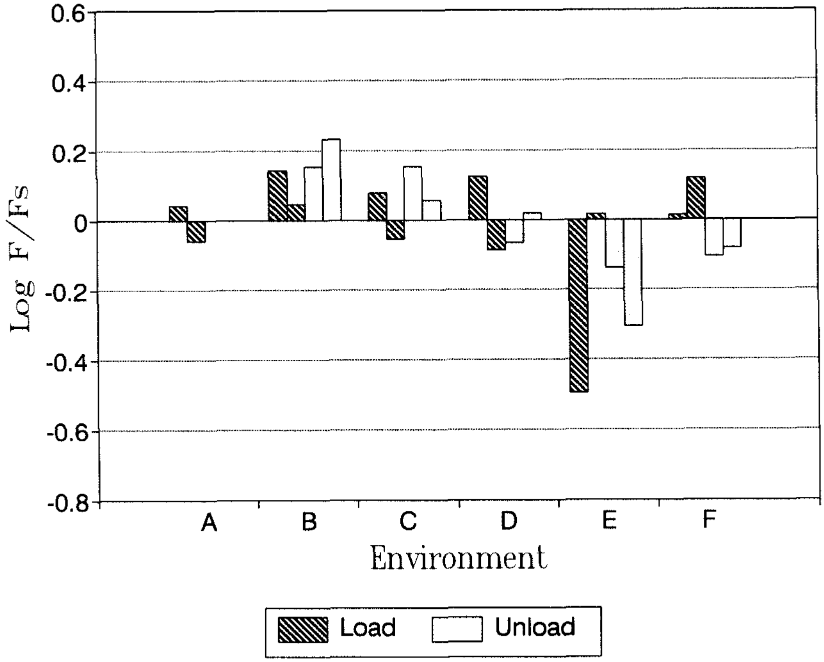

FIGURE 4a Static test results for adhesive 2.

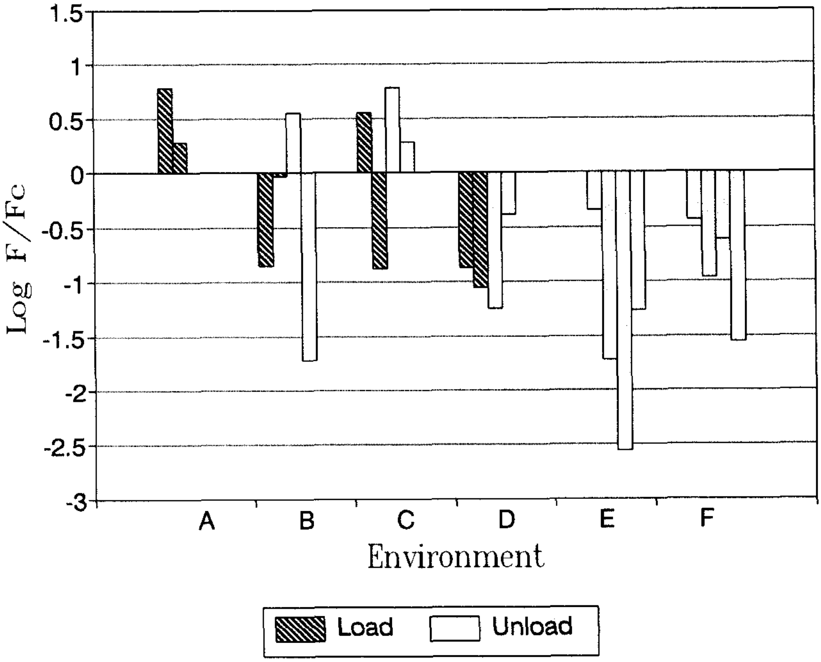

FIGURE 4b Fatigue test results for adhesive 2.

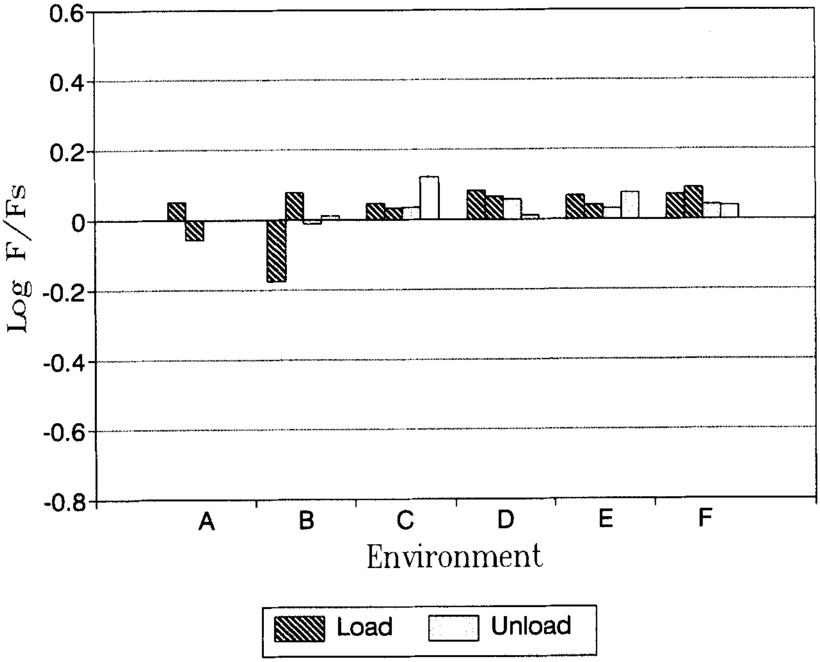

FIGURE 5a Static test results for adhesive 4.

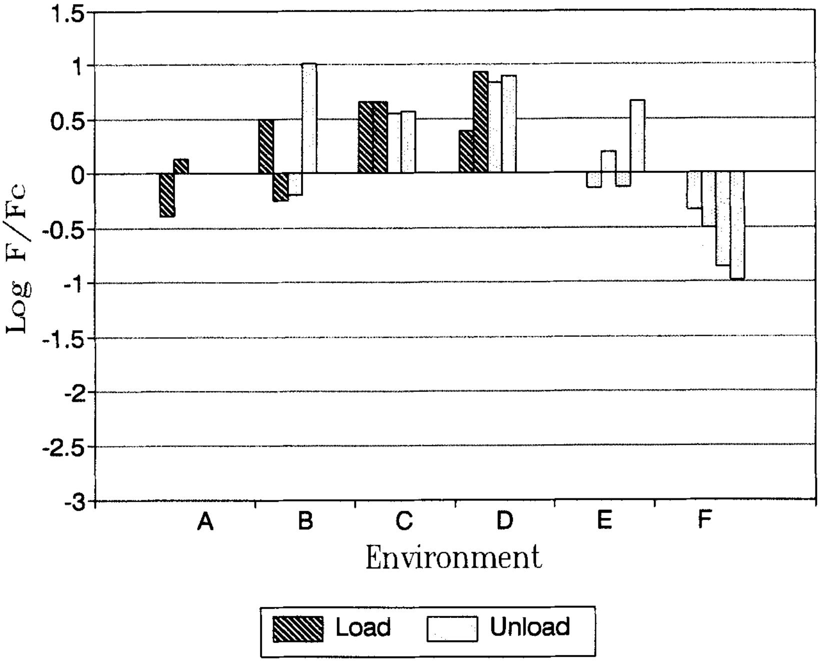

FIGURE 5b Fatigue test results for adhesive 4.

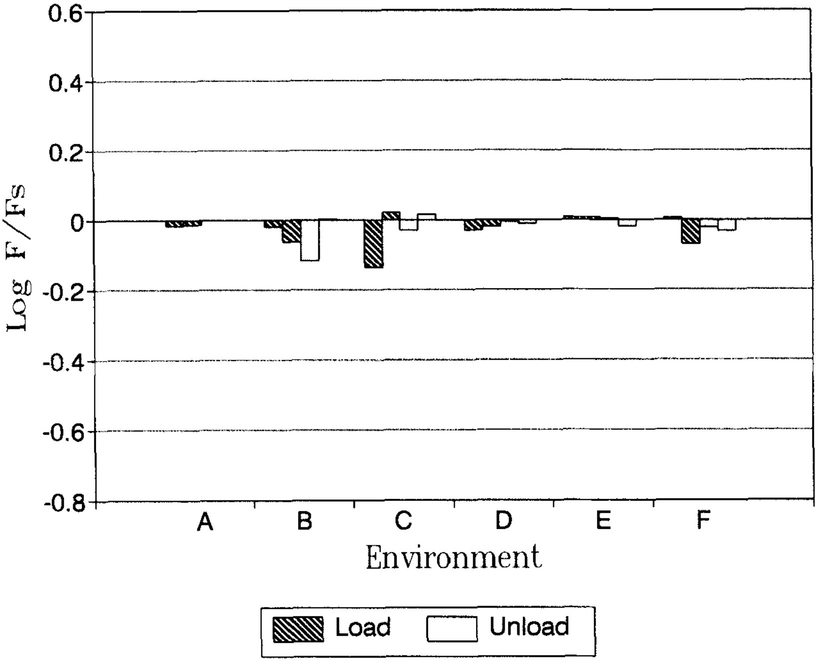

FIGURE 6a Static test results for adhesive 6.

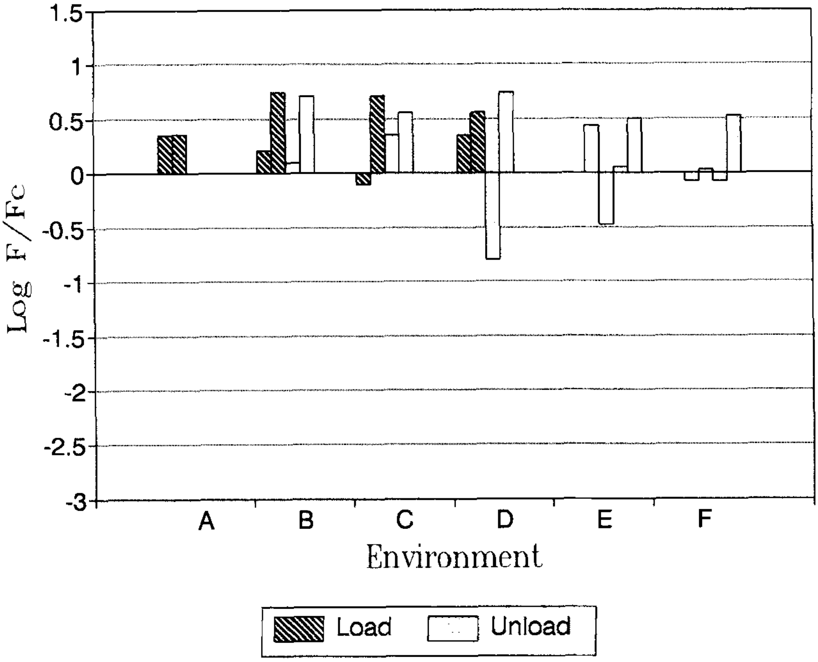

FIGURE 6b Fatigue test results for adhesive 6.

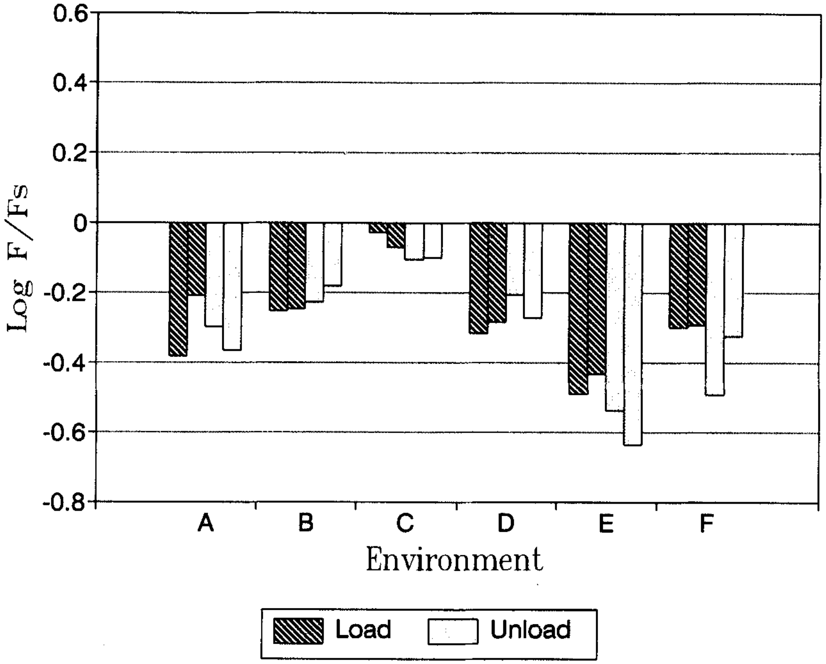

FIGURE 7a Static test results for adhesive 14.

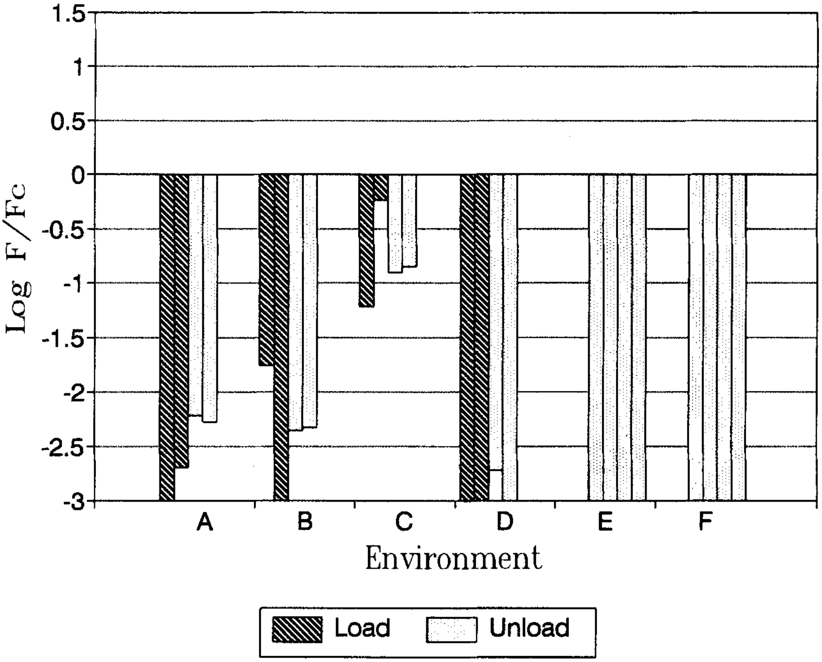

FIGURE 7b Fatigue test results for adhesive 14.

while adhesive 2 showed a serious loss. It is especially interesting to note that although adhesive 6 showed a small loss in static strength, its fatigue resistance improved.

4. Storing the specimens under load does not seem to have had any serious effect on joint performance. However, it should be noted that the load was chosen to be typical of the load that a joint might be designed to carry, not so that the joint would be severely stressed. If a higher load had been used then it might be expected that loading would have had a significant effect.

Adhesive 1

Most of the specimens showed an increase in static strength, the main exception was those stored in environment C (sub-zero). Most of the fatigue specimens showed significant increases in fatigue life, including those stored in environment C.

Adhesive 2

Most of the specimens exhibited little change in static strength. The exceptions to this were those stored in environment E (high humidity) and the unloaded specimens in environment F (natural exposure). The fatigue performance was much worse, with significant decreases being observed in all environments except A and C.

Adhesive 4

As with adhesive 1 both the static strength and fatigue life improved in most cases. There was one very notable exception to this, namely the fatigue performance of those specimens exposed to the natural environment. It is important to note that the other tests gave no indication of this poor performance, *i.e.* the static tests on the specimens stored in environment F gave good results, and the fatigue tests in environment E, which one might have expected to be a useful guide, were similar to those obtained in environments A and B. This should serve as a warning against extrapolating results obtained in one environment, or under one type of test, to another.

Adhesive 6

The vast majority of specimens showed a decrease in static strength, albeit a small one in most cases. Contrary to this, most specimens exhibited a significant improvement in fatigue life. Taken with the results for adhesive 4 in environment F, this gives a clear warning that the effect of exposure on the static strength does not necessarily predict the effect on fatigue performance.

Adhesive 14

Adhesive 14 showed a serious loss of static strength, and an almost total loss of fatigue resistance in all cases. It should be noted that the loss of fatigue resistance can be largely accounted for by the reduction in static strength, in some cases this having dropped below the maximum stress applied in the fatigue tests.

Effects of Environment

Since adhesive 14 performed uniformly badly the results will be ignored for the discussion on the effects of environment. Environment A had the smallest effect on adhesive joint performance. This is not too surprising since A was ambient conditions. Likewise, B and C had a limited effect in most cases, with the exception of the decrease in static strength of adhesive 1 in environment C. It should be noted that before testing the specimens were taken out of their storage environment and allowed to equilibrate to ambient conditions. If the tests themselves had been performed in the environments in which the specimens were stored one would expect the results to be different. The tests that were carried out suggest that the raised or sub-zero temperatures had little, if any, permanent effect on the adhesives or the joints. On the other hand, the effects of environments D, E and F would be expected to have a permanent effect since moisture absorption would be a key factor. For adhesives 1 and 6 the effect of these three environments was broadly similar. For adhesive 2 environment E clearly had the severest effect on static strength, with all three being more or less equally detrimental to the fatigue life. This could be taken to indicate that adhesive 2 was susceptible to the effects of water absorption. The effect of environment on adhesive 4 has already been discussed.

Failure Mode

In the control tests, adhesives 1, 4 and 6 all failed adhesively (*i.e.*, at, or near, the adhesive/adherend interface), adhesive 2 failed in a mixed mode (though largely adhesive) and adhesive 14 failed cohesively. In most circumstances the failure modes remained the same over the 8–9 year period for both static and fatigue tests. The exceptions were that adhesive 1 changed to cohesive or mixed mode for static tests in environment E and for the fatigue tests for environments E and F. The adhesive 2 specimens that underwent static tests failed totally adhesively in all environments, as did those tested under fatigue for environments E and F. Some degree of adhesive failure appeared for adhesive 14 in environments E and F. This indicates that moisture uptake was responsible for the changes in failure mode. It is interesting to note that the change for adhesive 1 was from adhesive to mixed mode. One often finds an instinctive distruct of adhesive joints that fail adhesively, the reasoning being that any effects of environment will have a detrimental effect on the interface. These results demonstrate that this is not a very useful guide. The joints that performed best (1, 4 and 6) were also the ones that failed adhesively. Moreover, the effect of water uptake for adhesive 1 seems to have been primarily on the adhesive itself, rather than the interface. This is not to say that it is a good sign for a joint to fail adhesively, but if a joint performs well adhesive failure should not necessarily be taken as a negative feature.

The differences observed in some cases between static and fatigue performance may be due to different failure mechanisms. For instance, in fatigue failure a process of damage accumulation usually exists before final disastrous failure occurs, see

Romanko and Knauss.[14] Bascom and Mostovoy[15] observed that in fatigue-failed specimens a larger scale radial micro-cracking was superimposed on the crack tip micro-yielding observed in normal fracture failure.

Water Absorption Tests

It is widely acknowledged that water absorption can have a serious effect on adhesives, and the present tests show that it was those environments that would subject the joints to humidity that had the greatest effect. Moisture uptake tests were carried out on the adhesives at the commencement of the durability programme by Lark.[8] Bulk specimens of the adhesives were completely immersed in water. Table I shows the percentage water uptake when water absorption had reached equilibrium, and the effect of this uptake on adhesive shear strength. Figure 8a shows the shear strength before and after water absorption. Figure 8b compares the adhesive strength after immersion with that of the initial joint strength (*i.e.* before ageing); the graph shows the bulk adhesive strength as a percentage of the initial joint strength. Clearly the amount of water absorbed from high humidity conditions or exposure to the natural environment will not be the same as that occurring when fully immersed in water, nor will the effect be identical, but it is not unreasonable to expect the results to have some indication of the long term performance of the joint. Accordingly, one would expect adhesives 1, 4 and 6 to perform best, and adhesives 2 and 14 to perform poorly, especially as the adhesive failure load has

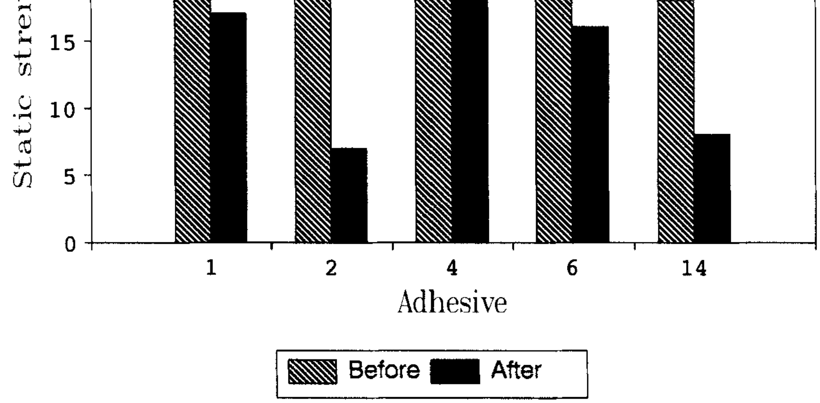

FIGURE 8a Effect of water absorption on bulk adhesive strength.

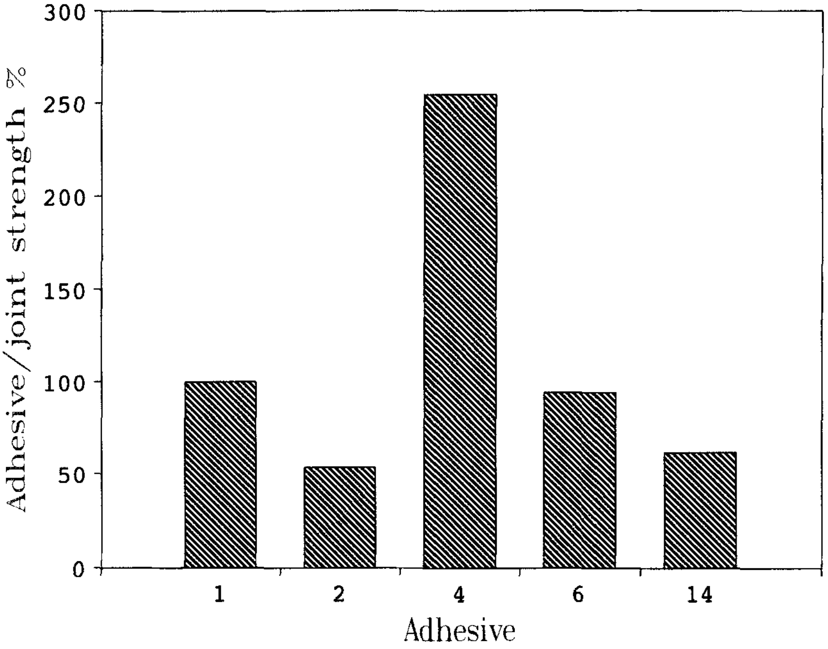

FIGURE 8b Effect of water absorption on bulk adhesive strength compared with initial joint strength.

fallen significantly below the joint strength. In the durability programme adhesives 1, 4 and 6 did indeed perform best, and 2 and 14 performed badly. Hence water absorption tests are useful, but it should also be noted that the water absorption test gave no indication of adhesives 4's poor fatigue performance in environment F. It is interesting to note that while adhesive 1 had the second highest water uptake in terms of weight, joints made with adhesive 1 performed best in the durability programme. This shows that it is not sufficient to consider water uptake on its own, rather its effect on adhesive strength must be considered as well.

Improved Performance with Age

A feature of the test results was the recurrent improvement in performance, especially in fatigue performance. It should be noted that fatigue results are inherently subject to experimental error, and the control tests were carried out on a different fatigue machine. However, there was no reason to doubt the reliability of either fatigue machine, and the improvement in fatigue life was repeatedly observed. Improvements in performance have been observed by other researchers. Hockney[16,17] investigated the effect of environmental conditions on joint strength. A number of adhesives were used and the joints were exposed to the weather in temperate, hot-dry, hot-wet, and tropical locations. Some joints were loaded and others unloaded. Tests were carried out after 6 months, 1, 2 and 4 years and the strength of most joints was found to have decreased. However, improvement of strength was found in a hot-dry environment when the epoxy-ether was part of a phenol-formaldehyde resin and not merely cross-linked with it. Brewis *et al.*[18] and Comyn[19] have also observed that the strength of single lap joints increased by about 35% upon moisture uptake by DGEBA adhesives. They proposed a relief of internal stresses by water plasticization as the reason for this strengthening. Water plasticization has been cited by Brockmann[20] as leading to greater peel resistance, and by Bascom[21] as playing a significant role in stress corrosion resistance. The reason for this effect is that moisture plasticizes the adhesive near crack tips, see Ripling *et al.*[22] Redistribution of stresses can also occur due to the viscous behaviour of the adhesives, Su *et al.*,[23] leading to lower peak stresses; this may also be a contributory factor. However, they also noted that the stress relief was associated with increase in shear strain, and while the stress relief may have a beneficial effect for a time the increase in shear strain may eventually lead to failure. Likewise, while water plasticization may mean that moisture uptake has a beneficial effect for a time in some cases, it is possible that eventually other effects of moisture may lead to deterioration in joint performance.

CONCLUSIONS

1. It is possible for adhesives to maintain both static and fatigue performance over long periods, even when exposed to very damp and natural environments. In this study two adhesives showed consistently good durability properties.
2. Exposure to the natural environment has the greatest effect on performance, and testing under very humid conditions does not necessarily give an indication of an adhesive joint's performance under natural exposure.
3. Water absorption properties and the effect of this on adhesive strength yield valuable information on the likely ability of the adhesive joint to resist the effects of exposure. However, the test is not an infallible guide.
4. In this programme the adhesives that showed the best performance were cured with polyamine hardener, and had high initial strength and Young's modulus.
5. It is possible for the static and fatigue performance of some adhesive joints to improve with age.
6. The long term effects of ageing and environment on the static strength of a joint may differ from the effect on its fatigue resistance.

Acknowledgement

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References

1. O. Hahn and X. S. Yi, *Welding in the World,* **25,** 96, (1987).
2. P. A. Fay and A. Maddison, *Int. J. Adhesion & Adhesives,* **10,** 179, (1990).
3. M. J. Cowling, I. E. Winkle, S. A. Hashim and E. M. Smith, "Adhesive bonding for marine environment," Symposium on Adhesion in Hostile Environments, London (1991).
4. W. Brockmann, *Adhesives Age,* **19,** 33 (1976).

5. A. J. Kinloch, *Adhesion and Adhesives Science and Technology* (Chapman and Hall, London, 1987).
6. R. W. Herzberg and J. A. Manson, *Fatigue of Engineering Plastics* (Academic Press, London, 1980).
7. G. C. Mays, *Interim Report WBRU/IR35* (University of Dundee, UK, 1982).
8. R. J. Lark, *The classification and control of epoxy adhesives in civil engineering,* (PhD Thesis, University of Dundee, 1983).
9. A. R. Hutchinson, *Durability of structural adhesive joints,* (PhD Thesis, University of Dundee, 1985).
10. G. C. Mays and A. E. Vardy, *Int. J. Adhesion & Adhesives,* **2,** 103, (1982).
11. G. C. Mays, *Fatigue Performance and durability of structural adhesive joints* (PhD Thesis, University of Dundee, 1985).
12. N. Su, *Durability and fatigue performance of structural adhesives and adhesive joints,* (PhD Thesis, University of Dundee, 1992).
13. N. Su, R. I. Mackie and W. J. Harvey, *Int. J. Adhesion & Adhesives,* **12,** 85, (1992).
14. J. Romanko and W. G. Knauss, "Fatigue Performance of Bonded Joints," in *Developments in Adhesives-2,* A. J. Kinloch, Ed. (Applied Science Publishers, London, 1981), p. 173.
15. W. D. Bascom and S. Mostovoy, ACS—Div. Org. Coat. Plast. Chem. Prepr. **38,** Nat. Meet. of ACS, 175th, Anaheim, California, Mar 12–17 (1978), p. 152.
16. M. G. D. Hockney, *RAE Technical Report 73016* (Royal Aircraft Establishment, Farnborough, UK, 1973).
17. M. G. D. Hockney, *RAE Technical Memo MAT 175* (Royal Aircraft Establishment, Farnborough, UK, 1973).
18. D. M. Brewis, J. Comyn and J. L. Tegg, *Int. J. Adhesion & Adhesives,* **1,** 35 (1980).
19. J. Comyn, in *Developments in Adhesives-2,* A. J. Kinloch, Ed. (Applied Science Publishers, London, 1981), p. 279.
20. W. Brockmann, in *Durability of Structural Adhesives,* A. J. Kinloch, Ed. (Appl. Sci. Publishers, London, 1983).
21. W. D. Bascom, *Adhesives Age,* **22,** 28, (1979).
22. E. J. Ripling, S. Mostovoy and C. F. Bersch, *J. Adhesion,* **3,** 145 (1971).
23. N. Su, R. I. Mackie and A. E. Vardy, *Creep analysis of adhesive joints,* Conf. on Struct. Adhesives in Engineering III, 30 June–2 July 1992 (Plastics and Rubber Institute, London, 1992), pp. 10.1–10.6.