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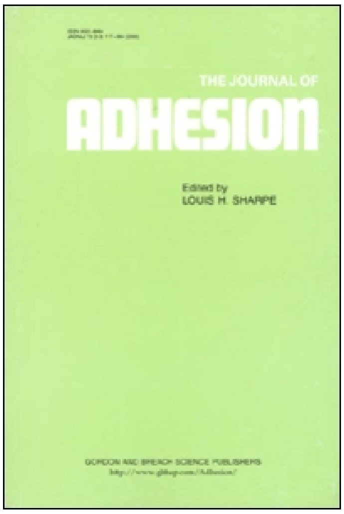

The Journal of Adhesion

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

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To cite this Article Chen, N. N. S. , Niem, P. I. F. and Lee, R. C.(1990) 'Experimental Investigation of Epoxy Bonded Polymethylmethacrylate Joints', The Journal of Adhesion, 31: 2, 161 — 176
To link to this Article: DOI: 10.1080/00218469008048222
URL: http://dx.doi.org/10.1080/00218469008048222

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J. Adhesion, 1990, Vol. 31, pp. 161–176
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Printed in the United Kingdom

Experimental Investigation of Epoxy Bonded Polymethylmethacrylate Joints

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(Received March 10, 1989; in final form November 28, 1989)

This paper presents an experimental investigation into various aspects of epoxy-bonded polymethylmethacrylate (PMMA) and PMMA-to-aluminium joints. The effects of adhesive thickness, overlap area, surface roughness, and environmental exposure on the joint strength were studied. Results indicated that the joint strength was not directly proportional to the overlap area, while sanding had a positive effect on the joint strength. A negative effect was observed when adhesive thickness was increased. The fatigue behaviour of adhesively-bonded joints under dynamic loading was found to be independent of frequency, for the range of values tested; however, it was dependent on the test temperature with greater reduction in fatigue life observed in PMMA-to-aluminium joints at higher temperature. Empirical equations from which the fatigue life of joints can be predicted were obtained by regression analysis. Intermittent fatigue testing of the joints was also performed. The epoxy adhesive tested proved to be a satisfactory choice for outdoor exposure. The rate of degradation of the adhesive was slow with the adherend itself degrading at a faster rate than the adhesive or the bondline.

KEY WORDS Fatigue test; environmental exposure; PMMA-to-aluminium joints; PMMA-to-PMMA joints; overlap area; adhesive thickness; surface roughness.

INTRODUCTION

In general, polymeric products can be formed by a variety of fabrication techniques. However, for large and intricate assemblies which cannot be moulded into one piece and for assemblies which involve integration of two incompatible materials, various joining techniques have to be employed. Adhesive bonding has often proved to be the most efficient, economical and desirable method for joining plastics and for joining plastics to other materials.

Improvement in strength, stability and ease of application have led to the increasing adoption of adhesives in a wide range of applications enabling them to displace traditional mechanical fastening methods. However, the lack of design data has often deterred engineers and designers from using adhesives because of the difficulty in predicting, with sufficient accuracy, the performance of the

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† Corresponding author.

adhesive joints. In view of this, experimental investigations into the various aspects of epoxy-bonded polymethylmethacrylate (PMMA) and PMMA-to-aluminium joints were carried out with a view to providing designers with additional information. These include the study of the effect of adhesive thickness, overlap area, surface roughness and environmental exposure on the strength of PMMA-to-PMMA and PMMA-to-aluminum joints.

EXPERIMENTAL PROCEDURES

Specimen Preparation

A standardized joint preparation procedure was used. This ensured reliable and reproducible test results.

The single lap joint was chosen as it is widely used and easy to make. According to ASTM D1002 and D3163, test specimens should be cut from bonded panels. However, individual specimens could also be prepared if cutting the bonded panels causes overheating, unwanted stress or physical damage to the specimen. The mechanical behaviour of plastics is extremely sensitive to machining, hence the method of individually-prepared specimens was used. Specimens were cut from cast sheets by circular saw and cut edges were then finished by milling to obtain smooth edges. The dimensions of the specimens used are given in Table I.

For optimum adhesion, the surfaces to be bonded must be cleaned or converted by chemical or physical treatments to a condition suitable for bonding. The bonding surfaces were first wiped with methanol and then sanded with No. 220 sandpaper. Adhesive tapes of thickness 0.12 mm were placed at the two longitudinal edges of one of the adherends so as to maintain a constant adhesive thickness. The surfaces were then wiped with methanol before bonding (ASTM D2093).

Epoxy adhesive mix, Araldite AW 106/Hardner HV953U (Ciba-Geigy) {Diglycidyl Ether of Bisphenol A (DGEBA) with curing agent Triethylenetetramine (TETA)}, was prepared in the weight ratio 100:80 and then applied to one side of each adherend, the mating parts were assembled in a special fixture to

TABLE I
Specimen dimensions in mm

Dimensions (mm)	*Adherend*				
	Tensile test	Exposure test		Fatigue test	
		PMMA			
	PMMA	Unloaded	Loaded	PMMA	Aluminium
Width	25.4	25.4	25.4	25.4	25.4
Length	101.6	101.6	45.0	45.0	45.0
Thickness	4.3	4.3	4.3	4.3	1.6

ensure correct overlap and accurate alignment. A known weight of 0.9 N was placed over the joint to ensure even contact over the bonded area. Excess adhesive was removed before the adhesive had set. Specimens were allowed to cure at ambient condition (18–24°C, 50–80% Relative Humidity) for 3 days. Five specimens were tested for each set of conditions except for fatigue tests where three specimens were tested due to space limitation. The standard deviation of the joint strength was found to be 15%. The procedure was strictly followed throughout the testing programme, but preparation may vary with the testing requirements.

A series of experiments were performed. These include the study of the effect of adhesive thickness, overlap area, surface roughness, environmental exposure and fatigue tests.

Experiments

1 *Effect of Adhesive Thickness* When using adhesives, some variation in glueline thickness due to uneven surfaces is inevitable. The adherends were bonded together in the usual way with tapes of different thicknesses placed at the longitudinal edges of the adherends. Specimens were tested after curing and the results were tabulated in Table II.

2 *Effect of Overlap Area* The results of adhesive tests have often been expressed in terms of strength, *i.e.* breaking load per unit bonded area. This implies a relation between force and area. The aim of this experiment was to determine whether differences occurred in the joint strength with different overlap area by varying the length of the overlap and the width of the adherend.

Joints were prepared as shown in Figure 1 and their respective areas are given in Table III. Specimens were tested after curing and results are presented in Table IV.

3 *Effect of Surface Roughness* To investigate the effect of sanding on joint strength, different roughening processes, namely milling and sanding, were used. The surface roughness of the adherends were measure by a Taylor-Hobson Talysurf 10 and results are presented in Table V.

4 *Environmental Exposure* An open air area well away from the shielding effect of trees and buildings was selected for exposure tests. Specimens were loaded in series by means of a spring loaded fixture as depicted in Figure 2. A

TABLE II
Effect of glueline thickness on the joint strength

Adhesive thickness (mm)	Average failure load (N)
0.05	1150
0.15	1135
0.30	1046

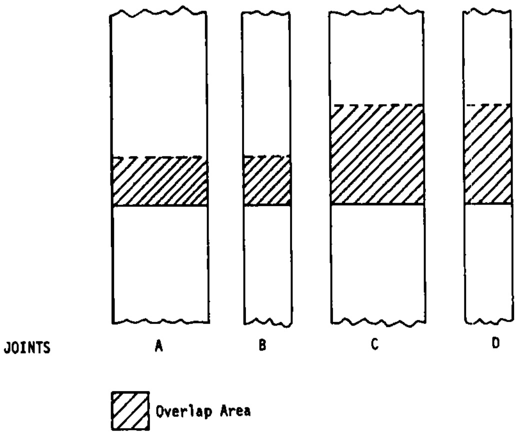

FIGURE 1 Joints with different overlap area.

TABLE III
Overlap dimensions of the joints

	Joints			
	A	B	C	D
Width, W (mm)	25	12.5	25	12.5
Overlap Length, l (mm)	12.5	12.5	25	25
Tape Area, At (mm^2)	20	20	40	40
Ovelap Area, Ao (mm^2)	313	156	625	313
Joint Area, $A = Ao - At$ (mm^2)	293	136	585	273

TABLE IV
Failure load of joints with different overlap area

Joints	Breaking load (N)
A	1146
B	592
C	1270
D	583

TABLE V
Effects of surface roughness on joint strength

	Average roughness (μm)	Breaking load (N)
Smooth	0.01	357
Milled	0.70	702
Sanded	1.89	1217

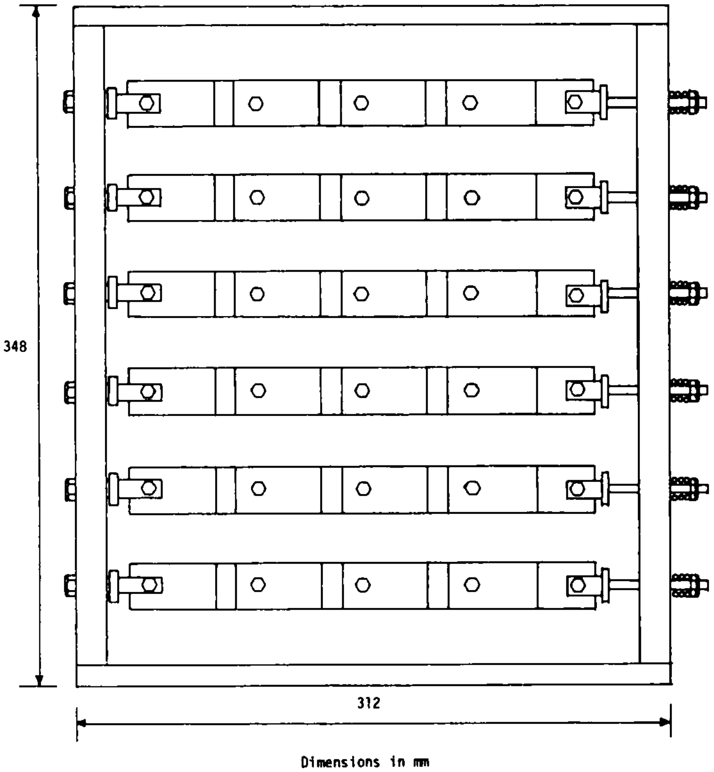

FIGURE 2 Environmental exposure specimen rig.

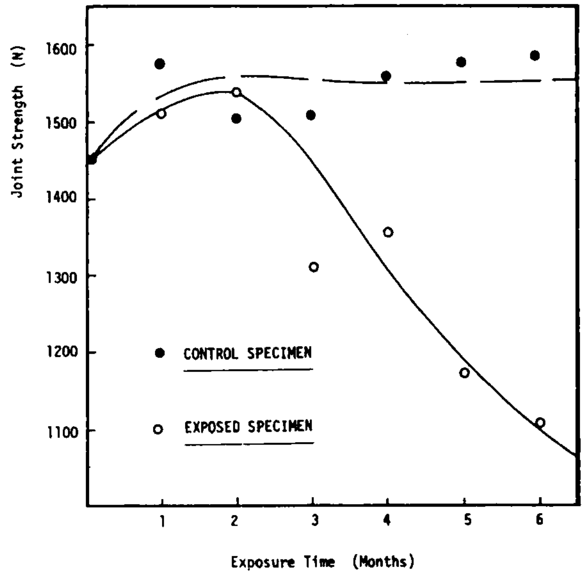

FIGURE 3 Variation in joint strength against exposure time for outdoor exposure of epoxy-bonded joints under sustained loading.

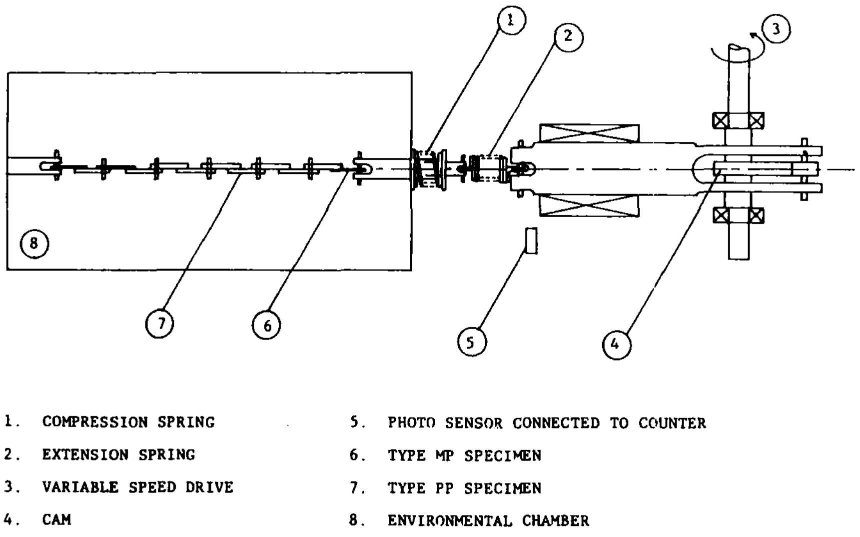

FIGURE 4 Schematic diagram of specimen loading system of the fatigue testing machine.

load of 200 N which was equivalent to approximately 15% of the initial strength of the specimen was applied. Specimens were tested after being exposed for the specified period. Cohesive failure was observed. Results are presented in Figure 3.

5 *Fatigue Tests* A fatigue testing machine was designed and constructed for the fatigue tests. It consisted of a cam and spring mechanism which generated a sinusoidal load at a frequency range of 54 to 135 cmp, as shown in Figure 4.

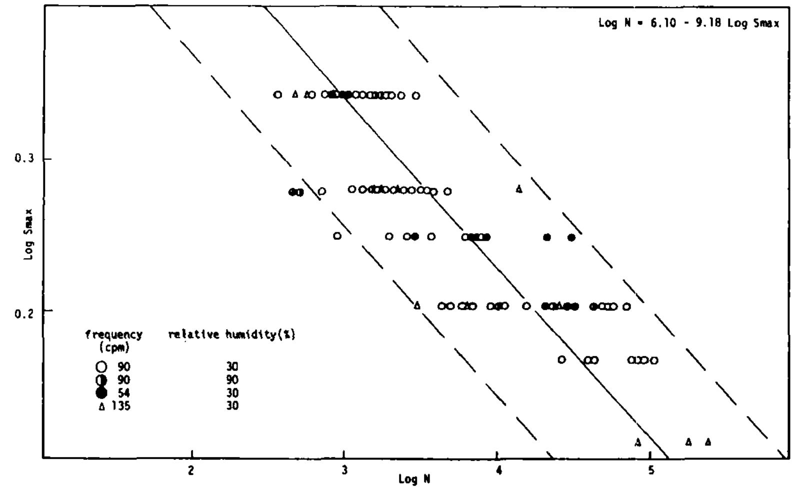

FIGURE 5 S–N diagram of PMMA-to-PMMA joints.

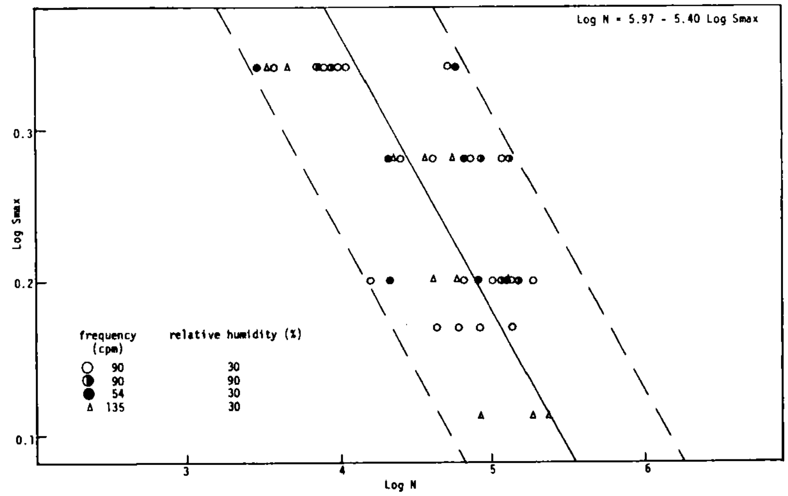

FIGURE 6 S–N diagram of PMMA-to-aluminium joints.

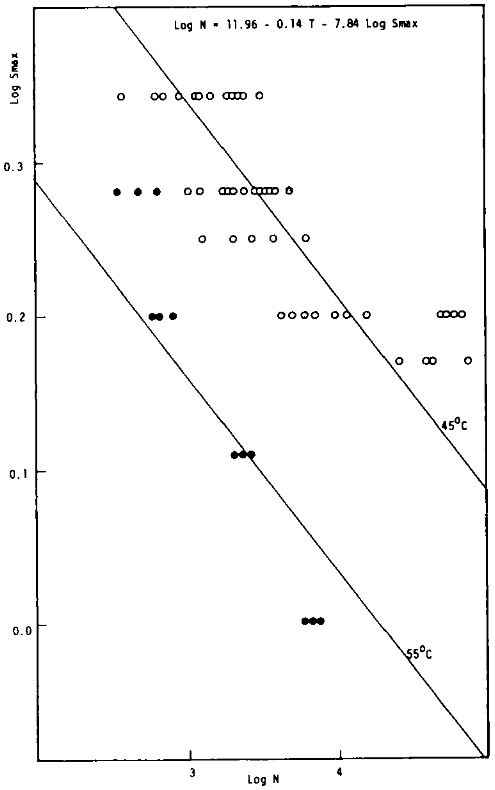

FIGURE 7 S–N diagram of PMMA-to-PMMA joints at different temperatures.

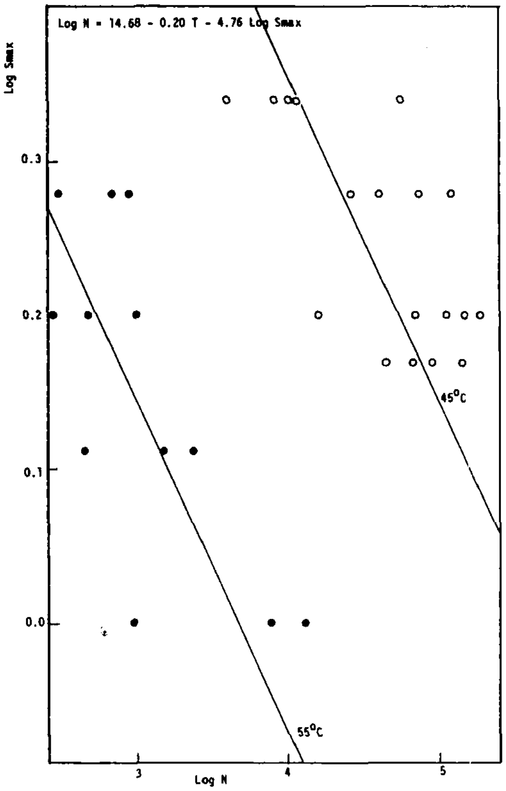

FIGURE 8 S–N diagram of PMMA-to-aluminium joints at different temperature.

Specimens were initially pre-stressed to the desired stress level and then subjected to tensile fatigue loading at a predetermined temperature, relative humidity (RH) and frequency. Failed specimens were immediately replaced by dummy specimens. The tests were continued until all specimens had failed. For specimens that did not fail at a runout cycle of 2×10^5 cycles, residual shear strengths were determined. In order for test conditions to resemble normal service conditions, intermittent testing with overnight stops were also carried out. Results are shown in Figures 5 through 8.

DISCUSSIONS OF RESULTS

Effect of Adhesive Thickness

It has long been established that the thinner the adhesive layer (except at very thin adhesive thickness), the stronger is the joint.[1] This prediction was supported by the results shown in Table II. While the average joint failure load for adhesive thickness between 0.05 mm and 0.15 mm remained approximately the same, further increase in thickness up to 0.30 mm resulted in 8% reduction in strength.

Several factors were thought to contribute to the strengthening of joints with decreasing adhesive thickness.

Curing and thermal shrinkage which result in residual stresses in the adhesive could lower the joint strength. Stress could result from contraction of the adhesive on setting or by differential contraction between adhesive and adherend after curing at an elevated temperature. These stresses were believed to be a function of adhesive thickness.[2] The development of full joint strength was dependent upon the complete cure of the entire resin layer between the adherends. Bowditch, *et al.*,[3] studied the effect of glueline thickness on joint strength. They found that the termination reactions in an acrylic adhesive were determined by a free-radical mechanism. The adhesive was unable to cure through large thickness, giving rise to weaker bondlines. No adverse effects were noted for a glueline thickness of up to 0.1 mm. However, beyond this point, the reduction in joint strength was rapid, with very low strength developed at a glueline thickness of 2.0 mm. Bikerman[1] argued that as the thickness of the glueline increases, the probability of an internal imperfection also increases. Although a thin glueline gives higher joint strength, it should be noted that a thin adhesive layer can result in voids due to insufficient adhesive. This formation of a starved joint at very thin adhesive thickness can lead to the lowering of the adhesive joint strength.

Effect of Overlap Area

Based on the results in Tables III and IV, it is apparent that the breaking force is not a direct function of the overlap area. Joint C with an overlap area four times that of Joint B only had its breaking force doubled. Joint D had a joint strength of 40% lower than Joint A, even though both joints had similar overlap area. It can be seen that Joint A and Joint C had a breaking force that was approximately twice that of Joint B and Joint D, respectively. With the overlap width remaining constant and the length doubled as between Joint A and Joint C, Joint B and Joint D, the breaking force was increased by only 10 to 15%.

The non-linearity of the load/overlap characteristic was caused by changes in the stress concentration factor which arose from varying strain conditions in the adherend and adhesive. When the specimen was under stress, the shear load in the adhesive was concentrated at the ends of the overlap. A simplified stress concentration factor (n) derived by Volkersen[4] relates the stress concentration to Young's modulus and the shear modulus of the adherend and adhesive, respectively, and to the joint dimension by the expression:

$$n = \frac{LG}{Etq}$$

where n = stress concentration factor
L = overlap length
G = shear modulus of adhesive
E = Young's modulus of adherend
t = adherend thickness
q = glueline thickness

Stress concentration is a function of the overlap length. Hence, an increase in overlap length which also increases the bonded area will result in an increase in stress concentration factor which in effect cancels out any gain in joint strength.

This disproportionate effect between the force and area should be considered when comparing performance between joints of different overlap area. Similar findings have been reported by Lees.[5] A limiting value of the joint strength was shown to exist and that, beyond a certain point, it was not possible to increase the strength of the joint simply by increasing the overlap area.

Effect of Surface Roughness

Roughening an adherend can produce a positive effect on joint strength by removing contaminants from the surface, increasing surface bonding area, providing a scarf-like surface geometry and increasing the tendency of the adhesive to spread on the adherend surface. However, negative effects can also arise if abrasive particles are left on the surface or if wetting is incomplete and an entrapment of bubbles or voids is produced at the interface. These flaws or discontinuities serve as sources of stress concentration and weakness within the substrate and adhesive interface.[6] In the case of the present work, surface roughening had a positive effect on the joint strength of the specimens. The increase in strength is a function of the roughness of the adherend with an increase in joint strength with increasing rugosity of the adherend.

Environmental Exposure

There was a slight increase in joint strength in both the control and exposed specimens within the first two months. Thereafter, the joint strength of the exposed specimens began to decrease while that of the control specimen remained at approximatly the same level. This deviation in joint strength became more significant as the exposure time was increased.

Visual inspection of specimens showed that control specimens remained the same in appearance and mode of failure. However, for exposed specimens, the adherends as well as the adhesive layer had turned yellowish and dull, indicating a change in properties due to degradation of the PMMA, thus lowering the joint strength significantly.

Jellinek[7] offered some explanations for the degradation of plastics exposed to the environment. Yellowing and a loss of gloss of a material are usually the first indication of material degradation. Photooxidation is often the main cause of degradation in the ambient atmosphere where oxygen and sunlight are ubiquitous. Oxidation of polymers causes deterioration in physical properties. As oxidation proceeds, decrease in molecular weight and discolouration of polymers are often observed. Oxidized polymers have lower mechanical strength. This accounts for the yellowing and lowering of joint strength in the specimens. In the presence of stress, mechanical degradation sets in. Application of stress results in physical changes which involve orientation, crystallization, flow, etc; and chemical changes such as scission along the main chain, scission at crosslinking, exchange reaction, crosslinking, etc. These changes of the polymer structure, in turn, affect the mechanical properties of the polymer. Another possible cause of discolouration could be the adhesive itself, in particular, the curing agent diffusing into the PMMA.

In short, environmental exposure under stress had accelerated the breakdown and deterioration of the adhesive joints. Epoxy adhesive had proved to be a satisfactory choice for joints exposed to the outdoor environment. According to Lee,[8] an epoxy cured with stochiometric amounts of TETA showed virtually no change in tensile strength after exposure to weathering for one year. This suggests that the rate of degradation of the adhesive was slow and the PMMA itself was degrading at a faster rate than that of the adhesive or the bondline.

Fatigue Tests

The maximum (S_{max}) and minimum (S_{min}) stress level and the number of cycles to failure (N) were recorded. Plotting these data with linear scales for both N and S_{max} revealed a need for transformation of the data. By a simple linear regression analysis, fatigue results can be expressed by the relationship as derived from the linear elastic fracture mechanics concept:

$$\text{Log}\, N = \text{Log}\, C' - m\, \text{Log}\, S_{max}$$

where C' and m are constants.

Due to the long and tedious computation involved, a packaged computer program was used. Raw data were input into the computer. Backward regression was used to eliminate variables which had little or no effect on the fatigue life. The fatigue performance of epoxy-bonded joints was found to be independent of the test frequency (within the range of 54 to 135 cmp) and humidity, but dependent on the test temperature and stress level.

The relationship between the fatigue life and maximum stress level at 45°C under different humidity and frequency conditions is expressed as follows:

1) For PMMA-to-PMMA joints

$$\text{Log}\, N = 6.10 - 9.18\, \text{Log}\, S_{max} \tag{1}$$
$$r = -0.84$$
$$s = 0.38$$

where N = number of cycles to failure
S_{max} = maximum stress level in MPa
r = correlation coefficient
s = standard diviation

2) For PMMA-to-aluminium joints

$$\text{Log}\, N = 5.97 - 5.40\, \text{Log}\, S_{max} \tag{2}$$
$$r = -0.74$$
$$s = 0.36$$

Lines were drawn through the mean value of the groups of replicated test specimens and the regression curves with the upper and lower confidence bound at 95.5% confidence are shown in Figures 5 and 6.

Earlier attempts[9] had been made to express the results in the form:

$$1/\text{Log}\,N = mS + C$$

where N = number of cycles to failure

S = mean stress level in MPa

This expression gave a satisfactory presentation of the relation between the number of cycles to failure and the mean stress level. However, the present expression derived from the linear elastic fracture mechanics concept gave a better correlation between the fatigue life and the maximum stress.

If temperature was introduced as an additional variable, the following equations were obtained.

1) For PMMA-to-PMMA joints

$$\text{Log}\,N = 11.96 - 0.14T - 7.84\,\text{Log}\,S_{\max} \tag{3}$$

$$r = -0.83$$

$$s = 0.40$$

where T = temperature in °C

2) For PMMA-to-aluminium joints

$$\text{Log}\,N = 14.68 - 0.20T - 4.76\,\text{Log}\,S_{\max} \tag{4}$$

$$r = -0.88$$

$$s = 0.39$$

Regression lines at 45°C and 55°C for different joints are shown in Figures 7 and 8.

Effect of Temperature

Service temperature has long been recognized as an important factor which limits the use of adhesives. Test results showed that there was a general decrease in fatigue life of the specimen as the test temperature was raised, and this effect was more pronounced in PMMA-to-Aluminium joints. This observation coincided with those made by previous research workers.[10–14]

Matting and Draugelates[10] claimed that as the ambient temperature was raised towards the glass transition temperature of the adhesive, the S–N curve was transposed downwards on the stress scale. For an epoxy adhesive, the change was significant even within the range 20°C to 40°C while for a poly(vinylformal phenolic) the test temperature had to be raised to 60°C before there was appreciable change in fatigue life of joints when compared to those tested at 20°C. In Mostovoy's experiment,[11] the crack growth rates were higher and the

TABLE VI
Coefficient of thermal expansion of selected materials

Material	Thermal expansion (10^6 per °C)
Aluminium	25
PMMA	81
EPOXY	30.9–90

fatigue resistance was lower as the test temperature was increased. Marceau, *et al.*[12] and Wegman, *et al.*[13] also reported that elevated temperature shortened the fatigue life of specimens. This was true for both lap shear and double cantilever beam specimens. Mays and Tilly[14] tested the specimens over a temperature range from −25°C to 55°C. Though the fatigue data can be grouped in the three bands, that is −25° to 0°, 20°C to 45°C, and 55°C, significant differences in performance were only observed for specimens tested at 55°C. The joints at 55°C were considerably weakened with over 50% reduction in the value of the endurance limit.

Differences in thermal expansion between the adherend and adhesive could well have contributed to the decrease in joint strength. When the test temperature was increased, thermal stresses were increased as a result of thermal expansion. This effect, being more severe as the difference between the coefficient of thermal expansion between the adherend and the adhesive increases, is the main reason for the marked decrease in fatigue life of the PMMA-to-Aluminium specimens. As seen in Table VI,[15] the coefficient of thermal expansion of polymeric materials is much greater than that of the aluminium. Similar findings were observed in the work of King and Bell.[16] A temperature gradient across the epoxy coating inhibited the lower layer from contracting, resulting in tensile stresses. A coefficient of expansion mismatch also resulted in a stress difference between the metal and epoxy. These two stresses, being greatest in the epoxy, led to failure in the epoxy layer adjacent to the metal.

A difference in tensile modulus of the adherends could have contributed to the early failure of the joint. As seen in Table VII,[15] aluminium has a much higher tensile modulus than PMMA, thus a greater strain occurs in the epoxy when a given stress is applied. The effect is believed to be more pronounced at higher temperature.

TABLE VII
Modulus of elasticity in tension of selected materials

Material	Modulus of elasticity in tension (lb/in^2)
Aluminium	$10 \times 10^6 - 10.3 \times 10^6$
PMMA	$3.5 \times 10^5 - 5 \times 10^5$

Effect of Frequency

No frequency effect was observed. The low and narrow range of frequencies selected could be a possible explanation. As suggested by Muhkerjee and Burns,[17] thermal failure can be avoided at a test frequency below 240 cpm. Thus, at these low frequencies (54 to 135 cpm), hysteresis heating is considered negligible and thermal failure is unlikely to happen.

In the current investigations, the temperature of the specimen was constantly measured by placing a thermocouple probe in an indented hole on the surface of the joint area and readings were obtained from a thermocouple meter. Temperature measurement of the specimen during testing showed little or no temperature rise. The frequency range may be too narrow for any effect to be detected. Should a wider range of frequency have been tested, results might have been different. However, testing at high frequency will present an unrealistic picture of the fatigue behaviour of the adhesive joints.

In fact, a higher rate could produce an apparent improvement in fatigue properties as pointed out by Mostovoy and Matting.[10–11] This suggests the need to evaluate these adhesives at a cyclic rate that can be associated with service frequency, as in many structural applications such as neon signs, etc. Thus the selected frequencies, although within a narrow range, should give a true representation of the fatigue behaviour of the adhesive joints.

Effect of Rest Periods

A series of tests was carried out to investigate the effect of intermittent testing on the fatigue life of the joints. Intermittent testing with prestress applied during the rest period did not improve the fatigue life of the joint.

Stinkas and Ratner[18] applied cyclic stressing to plastic specimens with and without rest periods. It was found that the fatigue life of specimens tested with rest periods was 50% higher than those tested continuously. A similar effect was revealed in the work of Koo, *et al.*[19–20] on fluoropolymers in which intermittent fatigue strength was found to be infinite, as long as the specimens were kept below a steady state temperature. Borduas, *et al.*[21] noted that when short stops and overnight interruptions were introduced during fatigue testing of PMMA, changes in crack growth rate occurred in some cases, but these changes were random in nature and did not affect the general picture of the curve. Cessna, *et al.*[22] showed that significant permanent damage did not occur unless the terminal fatigue behaviour region was entered.

In this experiment, no temperature rise was observed in the specimens, the little heat that was generated could be readily transferred to the surroundings. In view of the fatigue variable chosen for the tests, it was unlikely that thermal failure would result, for the following reasons:

i) Low or intermediate loss complicance of adhesive and adherend.

ii) A low frequency of 90 cpm was selected as compared to the 1800 cpm commonly used.

iii) The applied stress was relatively low as compared to the tensile strength of the adherend.

Based on the above points, it is evident that the joint failed primarily by crack propagation. With thermal heating practically absent in these specimens, interruptions which essentially allowed self-cooling resulting in partial recovery and delayed failure will not affect the general picture of the S–N curve for epoxy-bonded PMMA joints. This is true when there is intermission in loading. However, because prestress was present during the rest periods in this case, a reduction of fatigue life by 40% was observed. When at rest, the variable load was removed but the prestress load remained applied. In other words, the specimens were stressed all the time regardless of whether they were subjected to alternating dynamic loading. The stress element in Eqs. (1) and (2) comprises two components—constant prestress and variable stress. Therefore, the fatigue life obtained using Eqs. (1) and (2) will indicate fatigue strength of a joint subjected to a permanent prestress and a variable load. However, if it is applied to predict the strength of joints which are not subjected to prestressing, it will tend to predict a lower fatigue life. In other words, using these equations for prediction will incorporate a safety factor.

CONCLUSIONS

On the basis of the experimental results obtained, the following conclusions regarding the performance and behaviour of adhesive joints can be drawn:

1) The joint strength was not directly proportional to the overlap area.

2) Joint strength was found to decrease as the adhesive thickness was increased.

3) Sanding (abrasion) improved the strength of the joints.

4) The epoxy adhesive tested here is a satisfactory choice for making joints which were subjected to outdoor exposure. The degradation of the adherend (PMMA), rather than the adhesive, was the dominating factor influencing the joint strength.

5) The fatigue life of adhesively bonded PMMA-to-PMMA and PMMA-to-Aluminium joints was independent of frequency for the range of values tested.

6) The fatigue life of adhesively-bonded PMMA-to-PMMA and PMMA-to-Aluminium joints was dependent on the applied stress and test temperature. PMMA-to-Aluminium joints were more sensitive to temperature than PMMA-to-PMMA joints, and showed greater reduction in fatigue life when the test temperature was raised.

7) Given that specimens were subjected to prestressing, dynamic loading of joints with rest periods produced shorter fatigue life.

8) Empirical equations proposed will indicate a safe value for fatigue life joints.

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