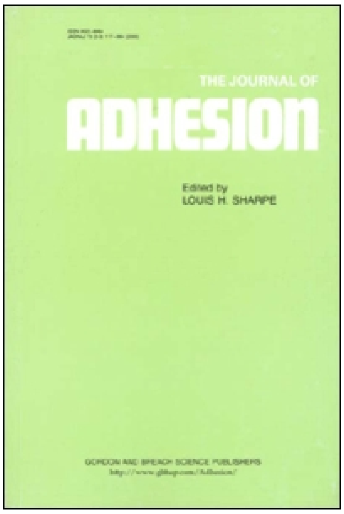

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Fatigue Performance of Adhesive Joints Immersed in Different Solutions

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Fatigue Performance of Adhesive Joints Immersed in Different Solutions

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Polymethyl methacrylate/epoxy/polymethyl methacrylate (PMMA/epoxy/PMMA) and aluminium/epoxy/aluminium joints were immersed in distilled water and in saline water at different temperatures and subjected to different sinusoidal tensile loads. The joints were also tested in air. The results suggested that the fatigue life of the adhesive joints decreased with increase in the temperature of the solution or load level. Also, PMMA joints showed better fatigue performance both in distilled water and sodium chloride solution than in air, while aluminium joints showed better fatigue performance in air.

KEY WORDS fatigue test; immersion test; saline water immersion; distilled water immersion; PMMA/epoxy joint; aluminium/epoxy joint; cyclic loading; durability.

INTRODUCTION

Adhesive bonding is an effective joining technique in producing useful structural assemblies.[1,2] The fatigue life of an adhesive joint is important in real life applications. There are many situations where a steady load application does not represent the real life service condition.

The authors[3] found that the joint strength of PMMA/epoxy joints increased when immersed in reagents and under static load, while that of aluminium/epoxy joints was reduced after immersion. It is, therefore, a natural follow-up to study the fatigue property of these adhesive joints immersed in different reagents.

In this study, the adhesive joints were tested under cyclic loading in different adverse conditions and with different load levels, in order to investigate the fatigue performance of the joints in such situations.

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EXPERIMENTAL

Specimens

Figure 1 shows the dimensions of the single lap adhesive joint which has been selected for its popularity in research of adhesively-bonded joints and for convenience in testing. The overlap area of the joints, 12.7 mm × 25.4 mm, was kept constant throughout the study.[4]

Similar to previous statically-loaded immersion tests,[3] aluminium alloy (99% Al, 0.5% Mg, 0.4% Cu, etc.) and pure cast PMMA were chosen as the adherends of the joints. The thickness of the aluminium adherend was 1.6 mm and that of PMMA was 4.5 mm. The thicknesses of the adherends were chosen by considering the thickness of the adherend material available in Hong Kong, and the strength of the adherend to withstand the applied load without failure. The adhesive chosen for the joints was a two-part epoxy (AW106/HV953U, CIBA-GEIGY), and the adhesive thickness was controlled to be between 0.13 to 0.16 mm by sticking two pieces of adhesive tape on the surface of one of the adherends in the adhesion zone. Two types of joints were used, namely PMMA/epoxy/PMMA, and aluminium/epoxy/aluminium.[3]

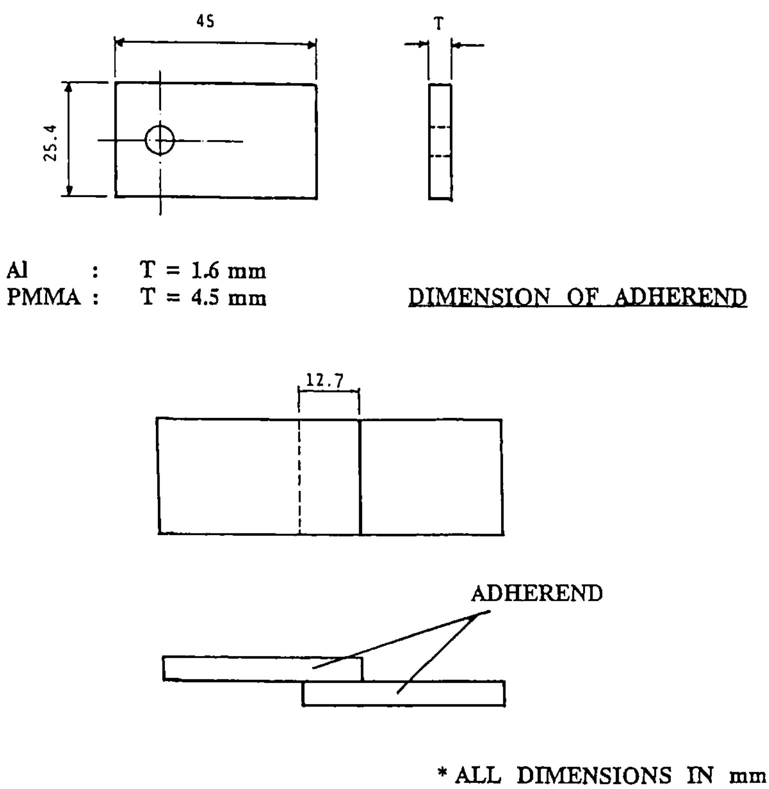

FIGURE 1 Dimensions of joints.

Selection of Test Conditions

Distilled water and sodium chloride solution were selected and used in subsequent experiments to simulate some common environments in which adhesive joints are likely to be used. The reagents were replaced after each test to minimize contamination.

Distilled Water Distilled water can affect the adhesive joint strength[3,5,6] and it is common to use adhesive joints in the presence of water. The impurities in tap water may affect the adhesive joint strength, therefore distilled water was used instead of tap water.

Sodium Chloride Solution The sodium chloride solution was prepared by dissolving high purity sodium chloride (the chemical was manufactured by May & Baker Ltd., England) in distilled water.[7] The percentage of sodium chloride in the reagent was 3% by weight so that the reagent was meant to be a simulation of sea water. The salinity of the reagent was monitored using a hydrometer.

Experiments

Preparation of Adhesive Joints The preparation procedure employed by the authors[3] for statically-loaded immersion tests were used for adhesive joints in the fatigue tests. Adherends were cut from cast PMMA sheets and aluminium sheets. The adherend surface was roughened with grade 'O' sanding cloth with the lays made by the sanding cloth perpendicular to the length of the adherend. The mechanically-prepared adherend was cleaned with trichloroethylene. The adherends were used immediately after surface treatment in order to reduce contamination. The adhesive was applied with a spatula.

The joints were prepared in batches, with 10 specimens per batch, at controlled laboratory environment with temperature ranging from 19°C to 24°C and relative humidity 50% to 80%. Adhesive tape was wrapped around the three edges of the adhesion region of the adherends to form a weak partition between the adherend and the epoxy overflow, so that the epoxy overflow would not bond with the edges of the adherends. Figure 2 shows the positions of the adhesive tapes. The adhesive tape was left in its position before and during the test in order to prevent the formation of micro-cracks arising from the tape removal process. A weight of 80 g, which was determined by experience, was placed on the joint to hold down the adherends and to ensure even spreading of the adhesive. The specimens were allowed to cure at controlled laboratory environment for 7 days to ensure complete setting of the adhesive.

Table I shows the range of the mean initial strength and the standard deviation (SD) of all the batches of adhesive joints tested in the study. The SD is within approximately 11% of the mean.

The preparation procedure employed previously[3] was used in this study because the method had proved itself to be satisfactory in producing well-prepared specimens for testing. However, in order to obtain useful and meaningful results, the authors were concerned that every step of the method was carefully examined and controlled, especially the use of adhesive tapes on the specimens.

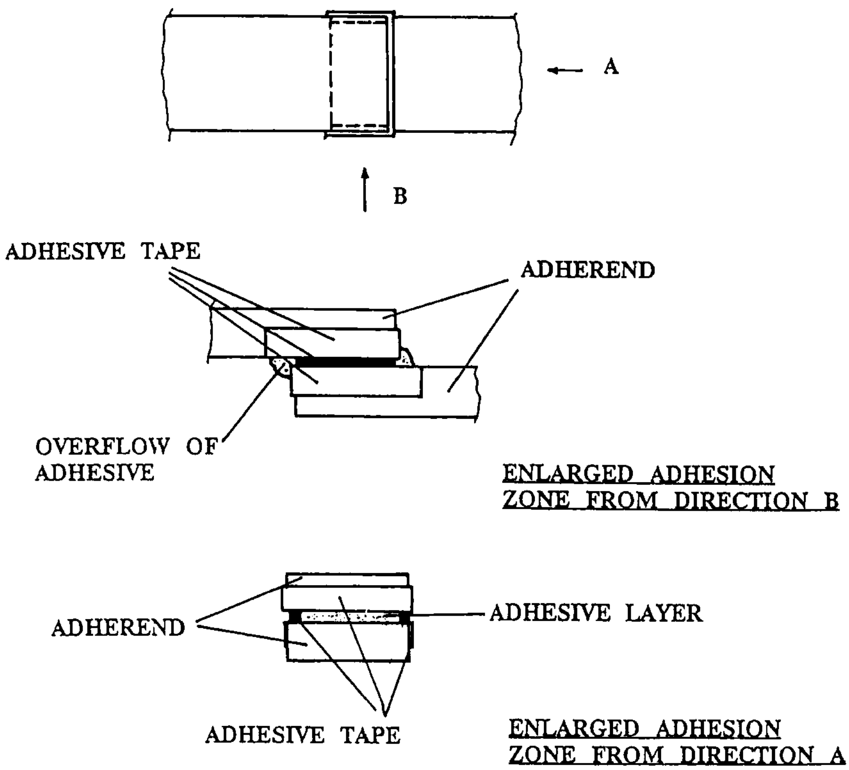

FIGURE 2 Positions of adhesive tapes.

The adhesive tape used to separate the filet and the adherend edge should cause no problems in the study. During the tests, it was found that the bond between the adhesive tape and the adherend failed while the filet was still adhered to the tape. Moreover, the crack formed between the adhesive tape and the adherend was perpendicular to the adhesive layer of the adhesive-bonded joint as well as to the direction of the force applied, the deformation of the adherend and the adhesive layer, as well as the shear direction. Therefore, this crack (*i.e.*, between the tape and the adherend) should have no effect on the formation and propagation of the crack in the adhesion zone.

The presence of adhesive tape in the adhesion zone was a possible source of error in measuring the strength of the adhesive-bonded joint because of the possibility of crack

TABLE I
The mean initial joint strength and standard deviation

	Strength (N)	Standard Deviation
Aluminium Joint	2900–3250	62–148
PMMA Joint	832–1050	24–106

initiation adjacent to the adhesive tapes in the adhesion zone. Therefore, as in the previous work,[3] the joints were observed under a LEITZ microscope with 100 and 200 times magnification before the test and no crack was found. Moreover, the cracks initiated during the test and in the previous work were at the ends of the adhesion zone with high stress concentration, but not at the edge of the adhesive tapes in the adhesive layer. The propagation of the crack was not along the adhesive tapes. These observations showed that the effect of the adhesive tapes in the adhesive layer is negligible.

Test Programme The prepared adhesive joints, which were selected randomly from the batches, were immersed in different reagents and in air in a KOTTERMANN temperature-controlled chamber. A dynamic load was applied by connecting the specimens to a specially-designed fatigue testing machine which generated a sinusoidal force by a cam-and-follower mechanism. Figure 3 shows the experimental set-up which includes the temperature-controlled chamber, the reagent container, and the fatigue testing machine. The displacement of the follower caused the spring to be compressed and a reaction force acted on the cam housing. The cam housing was connected to the test specimens inside the temperature-controlled chamber. Therefore, an axial load was applied to the specimens. The amplitude of the sinusoidal force could be changed by using different compression springs of different stiffnesses.

During the test, three specimens were connected in series. An aluminium dummy test piece was used to replace any failed specimen. If a specimen did not fail during the fatigue test, it was removed and washed with distilled water, dried with a soft wiper, and then loaded in a JJ Loyd T22K Tensile Tester to find the residual strength. The cross-head speed of the tensile tester was set to 4.5 mm/min.

Four cyclic loads with a frequency of 60 cycles per minute were employed after taking into account the strength of the prepared joints, the ASTM Standard D3166[8] and the springs available in Hong Kong. They were: 220N, 300N, 450N and 570N. A pre-stress was placed on the specimens to ensure constant contact between the cam and the follower of the fatigue testing machine. The pre-stress was arbitrarily set at 5% of the

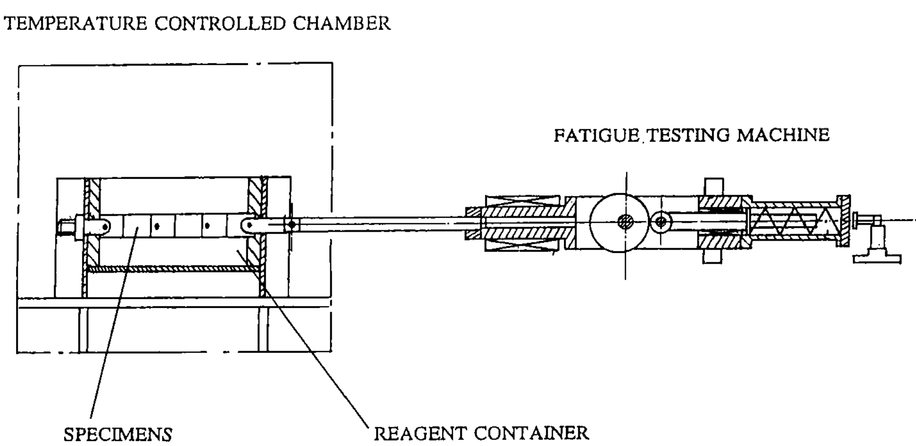

FIGURE 3 The experimental set-up.

corresponding load. The stress ratio of the sinusoidal load pattern was +0.05. The maximum number of cycles employed was 200,000. Such a number should have a noticeable effect on the adhesive joints.[9,10]

The specimens were tested under four different temperatures: 5°C, 23°C, 35°C, and 45°C. The temperature range was selected by referring to the normal possible working temperature of adhesive joints in real life and the specification of the adhesive.[10] A temperature of 45°C was a reasonably high temperature under a living environment.

The lower limit of the temperature range was set by considering the freezing point of water, and 5°C was chosen.

Also, 23°C was selected because it is the normal room temperature, and 35°C was arbitrarily selected since it is between 23°C and 45°C.

Earlier results of static tests of PMMA adhesive joints[3] showed no significant difference between the effect of water and sodium chloride (NaCl) solution on the strength of the joints within the test period. In this study, NaCl solution was used to make a comparison with water immersion and so only room temperature, *i.e.*, 23°C, was employed.

An endurance limit may exist at each temperature within the load range. The joint was tested at the highest load level and from the highest temperature to the lowest. The load level was then reduced and the joint was again tested at different temperatures. The process continued until the endurance limit was reached. At this point, it could be concluded that the joint would not fail below the combination of temperature and load level. The load level was then further reduced and the joint was again tested at different temperatures (from the highest to the lowest) to examine whether there was another endurance limit with another combination of load level and temperature. The process continued until all possible endurance limits were determined within the range of temperature and load employed.

RESULTS AND DISCUSSION

PMMA Adhesive Joints

Figures 4 and 5 show the S-N diagrams of PMMA adhesive joints by relating the number of cycles to failure to load level at different temperatures in air and in water immersion. It can be seen that the fatigue life was reduced as the temperature or load level was increased. Also, the joints showed higher fatigue performance in distilled water than in air.

Visible crack propagation was observed in the PMMA adhesive joints because of their transparency. However, researchers[11-16] suggested that one mode of fatigue damage of aluminium adhesive joints was crack growth at the interface of the adhesive layer and the adherend. The concepts of linear elastic fracture mechanics can be applied to characterize quantitatively the fatigue cracking. The lines on Figures 4 and 5 were obtained by simple linear regression analysis:

$$\mathrm{Log}\, n = \mathrm{Log}\, a - b\, \mathrm{Log}\, s$$

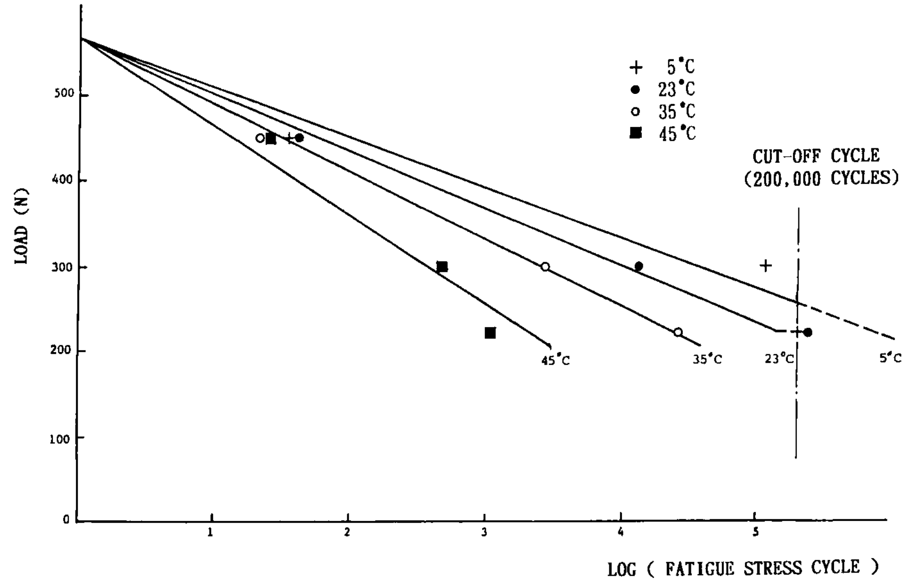

FIGURE 4 S-N diagram of PMMA joints in distilled water.

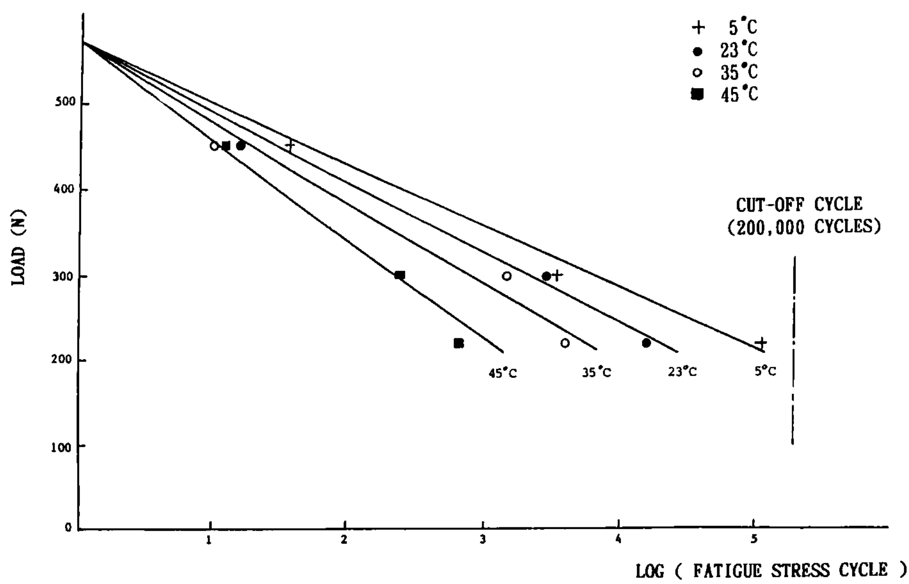

FIGURE 5 S-N diagram of PMMA joints in ambient condition.

where

n = the number of cycles to failure
s = maximum stress level
a, b = constants

The results show that the fatigue performance was better in water immersion than in air. This could be due to the softening of the adhesive layer which resulted in more even distribution of stress in the adhesion region.[3] Sauer *et al.*[17] found similar results and stated that the fatigue lifetime of polystyrene could be enhanced if tests were carried out in the presence of water. They also suggested that the fluid in which the joints were immersed inhibited or delayed crack development by filling in surface voids and flaws, and exerted surface tension forces across the interfaces, so that an increase in the liquid surface tension would further increase the degree of fatigue performance.

In a static immersion test performed by the authors,[3] PMMA joints showed satisfactory performance with a 300 N load. However, under a cyclic load of the same magnitude, the joint failed within 20,000 cycles at room temperature. This suggested that PMMA joints are not suitable for applications under cyclic load.

The performance of PMMA adhesive joints was low even at the 220N load level (28% of the mean initial strength of all PMMA adhesive joints), when the joints were used at a temperature higher than room temperature.

An endurance limit was found in the fatigue test with water immersion at 23°C. A number of researchers[18–22] had claimed that the endurance limit was related to the ultimate tensile strength and the formation and propagation of cracks within the adhesive layer. They suggested different ratios of maximum loading of the adhesive joint to ultimate tensile strength, ranging from 0.2 to 0.5. The results of the fatigue tests of that study were found to be at the lower end of the range at 23°C in air. However, the current fatigue tests showed that the ratio would be changed with the environment within which the joints were tested. High temperature can lower the fatigue performance while, as discussed above, immersion in reagent can improve the performance of PMMA joints.

Table II shows the comparison of mean values of fatigue test results of PMMA adhesive joints at 23°C in distilled water and in NaCl solution. It was found that the

TABLE II
The fatigue test results of PMMA joints at 23°C in water and in NaCl solution

Load amplitude (N)	Fatigue life in reagents (Cycles)	
	Water	NaCl solution
220	*(1)	*(2)
300	12863	10401
450	36	28

* Endurance limit was found at the corresponding combination of load and temperature, the mean residual strengths of the joints were (1) 1435N, (2) 1507N.

endurance limit for joints immersed in sodium chloride (NaCl) solution was about the same as that of water, *i.e.*, at 23°C and 220 N load level.

The residual strength at the endurance limit showed that the strength of the joints increased after immersion in different reagents, as in the static test.[3] Compared with the strength of the joints before immersion, the percentage increase at the endurance limit of 220 N ranged from 46% to 69%, which was about the same as the increase in the joint strength after a 20-day static immersion with a 300 N load. This showed a higher diffusion rate of reagent into the adhesive layer with cyclic load than under static load. Thus, cyclic loading should attract more attention than static loading for adhesive joints in practical applications. Although the increment in the residual strength of the joints immersed in NaCl solution was similar to that of joints immersed in water, the mean number of fatigue cycles at joint failure in NaCl solution and under a 300 N load was 19% lower than that in water. This showed that the joints were less strong in NaCl solution than in water under a high number of cyclic loadings.

Moreover, the joint performance dropped sharply above the 220 N load level, *i.e.*, the endurance limit. This showed that the effect of the load level on joint performance was great when the level was higher than that of the endurance limit, and so it is important in actual applications to keep the load level below the endurance limit. The short fatigue life of the joints at 450 N (57% of tensile strength) showed that such a load level was too severe for PMMA joints and should not be used in practice.

Aluminium Joints

Figure 6 shows the lines connecting the endurance limits for different combinations of load and temperature in air and in water. The fatigue life showed that the fatigue

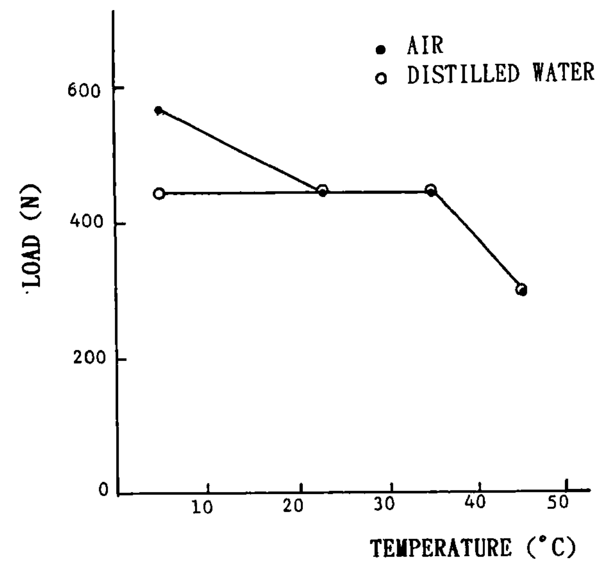

FIGURE 6 Endurance limits of aluminium adhesive joints at different combinations of load and temperature in ambient environment and in distilled water.

performance of the aluminium adhesive joints was influenced by the applied load level and temperature. However, the fatigue performance of the adhesive joints has been found to be higher in air than in water.[3] This may be due to the influence of water on the adhesive/adherend interface as shown in the static immersion test of aluminium joints. The high load level and temperature employed for the fatigue tests could enhance the diffusion rate of water in the adhesive layer and in the interface and, hence, could influence the adhesive joint strength.[23-26] Although the endurance limits were the same in air and immersion in water at and above room temperature, the residual strength of the adhesive joints tested in air (3027 N at 35°C and 2780 N at 45°C) was higher than that in water (2620 N at 35°C and 2393 N at 45°C). This showed that the performance of the adhesive joints under cyclic load should be higher in air than in water.

Figure 7 shows the reduction of the joint strength with changing temperature at a constant (570 N) load level. The curves show that above room temperature the

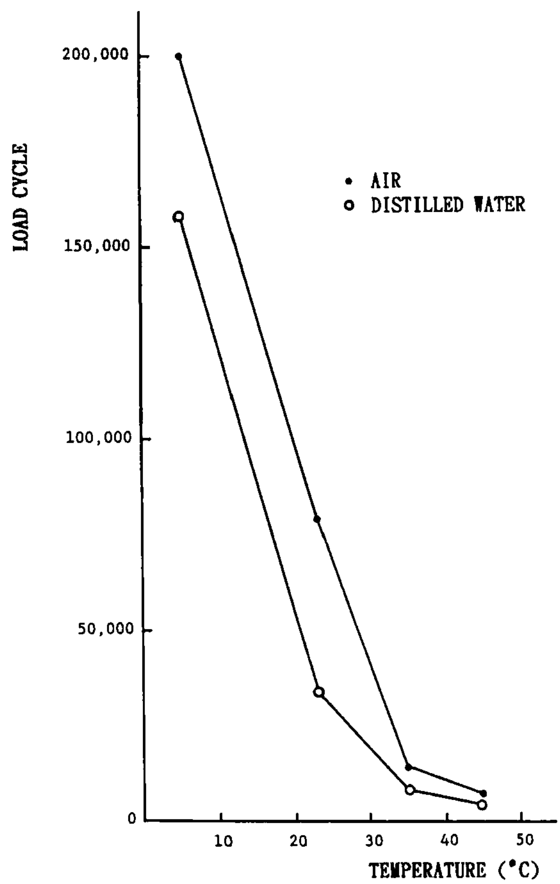

FIGURE 7 Rate of reduction of strength of aluminium adhesive joint with different temperatures at 570 N load level.

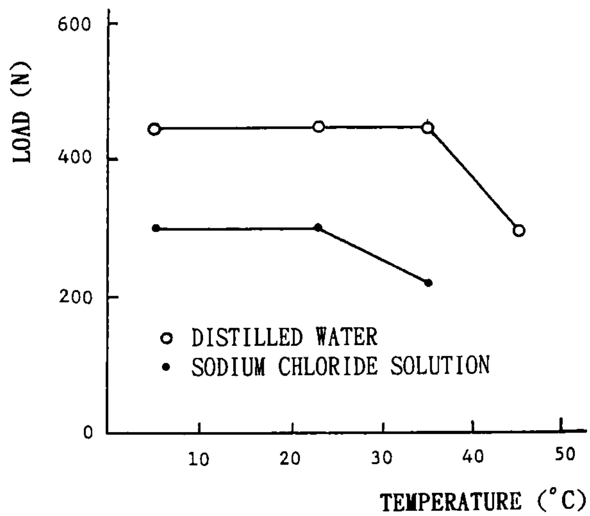

FIGURE 8 Endurance limits of aluminium adhesive joints at different combinations of load and temperature in sodium chloride solution and in distilled water.

performance of the aluminium adhesive joints immersed in water was close to that in air. That may be because when the test temperature was increased, the fatigue life of the adhesive joints was reduced and the influence of water on the joint interface was diminished. The diminishing effect was due to the shortened immersion time, thus causing the difference between air and water immersion to be reduced.

The fatigue test results for aluminium adhesive joints in NaCl solution showed that the fatigue performance was greatly reduced by replacing distilled water with NaCl solution, as shown in Figure 8. This indicates that NaCl solution was a more aggressive reagent to aluminium adhesive joints than water under cyclic load, as was found earlier in statically-loaded immersion tests.[3]

CONCLUSIONS

From the results obtained in this study, it is possible to draw the following conclusions.

1. The fatigue performance of adhesive joints deteriorated under high temperature and applied load.
2. The fatigue performance of adhesively-bonded aluminium joints deteriorated when they were immersed in water or sodium chloride solution, while that of PMMA joints was improved.
3. The rate of change of joint performance (improvement or reduction in joint strength) was faster under cyclic load than under static load.
4. Sodium chloride solution is a more aggressive reagent to PMMA and aluminium adhesive joints than water under cyclic load.

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