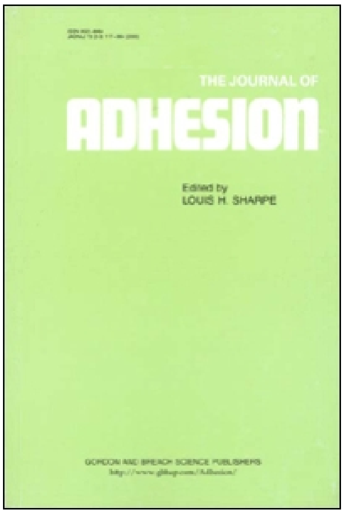

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Strength of Adhesive Joints Immersed in Different Solutions

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Polymethyl methacrylate/epoxy/polymethyl methacrylate (PMMA/epoxy/PMMA) and aluminium/epoxy/aluminium joints were immersed in different reagents including saline water, distilled water, sodium hydroxide solution, and sulphur dioxide solution. The joints were immersed in the reagents with and without a constant tensile load. The joints were also tested in air. Four loading periods were selected. At the end of each loading period, the joints were loaded in a tensile testing machine until failure. The results of the tests showed that the tensile strength of the aluminium joints was reduced, while that of polymethyl methacrylate was increased after immersion. The same results were obtained regardless of whether the submerged joint was loaded or not. Moreover, corrosion was found on the aluminium joints immersed in the sodium chloride solution and sulphur dioxide solution.

KEY WORDS Static immersion test; loaded immersion test; saline water immersion; distilled water immersion; sodium hydroxide solution immersion; sulphur dioxide solution immersion; PMMA/epoxy joint; aluminium/epoxy joint.

INTRODUCTION

Adhesive bonding is a very effective joining technique in producing useful structural assemblies.[1,2,3] However, the prediction of the performance of adhesive joints is still a problem due to a lack of data in different application areas and the separate and combined effects of many factors which affect the joint performance such as moisture, surface preparation, and properties of adhesive and adherend.

In this study, adhesive joints were tested in different adverse conditions that were simulations of actual environments in order to investigate the performance of the joints in such situations.

SPECIMENS

1. Type of Joint

The single lap joint was selected for its popularity in actual applications. The dimensions of the joints are shown in Figure 1. The overlap area, 12.7 mm × 25.4 mm, which was kept constant, was according to ASTM D2919.[4]

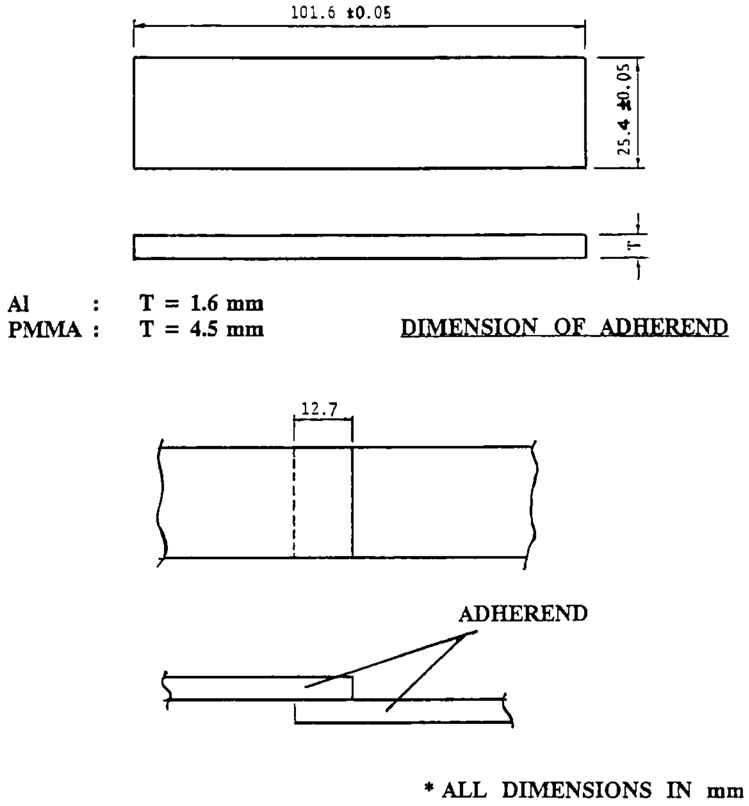

FIGURE 1 Dimensions of joints.

2. Adherends

Aluminium alloy (99% Al, 0.5% Mg, 0.4% Cu, etc.) and pure cast PMMA were chosen as the adherends of the joints because they are commonly used in industry. The thickness of the aluminium adherend was 1.6 mm and that of PMMA was 4.5 mm. Two types of joints were used, namely PMMA/epoxy/PMMA, and aluminium/epoxy/aluminium.

3. Adhesive

A 2-part epoxy-adhesive (AW106/HV953U, CIBA-GEIGY), commonly used in industry, which gives satisfactory joint strength and environmental resistance with aluminium and PMMA adherends, was selected. It is easy to handle due to its slow setting property and was, therefore, used in this study.

The Araldite® AW106 is a modified bisphenol A based epoxy resin and the hardener HV953U is a polyaminoamide modified with a tertiary amine. The mixing ratio of Araldite to hardener is 1 to 0.8 by weight. The adhesive joints were cured in ambient environment with 50% to 80% RH and 19°C to 24°C.

4. Adhesive Thickness

It has been found, for a similar PMMA/epoxy/PMMA joint, that the average joint failure load for adhesive thickness between 0.05 mm and 0.15 mm remained approximately the same.[5] Hence, a thin adhesive tape of 0.12 mm thickness (with a trade name of "Zipaline" B-61-M, manufactured by Zipalone, Inc., USA) was used to control the adhesive layer thickness. The width of the tape was 0.79 mm (1/32 inch).

The application of the adhesive tape is discussed under "Bonding Procedure." On examining the prepared joint under a microscope, it was found that the thickness of the adhesive layer was between 0.13 to 0.16 mm.

SELECTION OF TEST CONDITIONS

Four types of reagents were selected and used in subsequent experiments: distilled water, sulphur dioxide solution, sodium hydroxide solution, and sodium chloride solution. These reagents were selected to simulate some common environments in which adhesive joints are likly to be used.

1. Distilled Water

It is common to use adhesive joints in the presence of water and researchers[6,7] have found that water can affect the strength of the adhesive joints. Distilled water was used instead of tap water in order to eliminate any effect on joint strength due to impurities in the water. For the same reason, it was also used for the preparation of other solutions as required by ASTM D896.[8]

2. Sulphur Dioxide Solution

The sulphur dioxide solution is a major component of acid deposition in the environment, therefore it was selected in order to study the performance of the adhesive joints in an industrial area. The amount of sulphur dioxide in water varies greatly with the geographic location. Park[9] stated that the lowest pH value of rainfall in the world was 3.5, therefore a pH value of 3.5 was selected. The percentage of sulphur dioxide in the solution was 0.007%.

The pH value of the reagent was checked every day using a portable digital pH meter (JENCO 602).

3. Sodium Hydroxide Solution

Sodium hydroxide is widely used in paper making, in the textile industry, in soap making, and also in the pretreatment for aluminium components which are to be anodized.[10,11] The concentration of the reagent was similar to the alkaline etchants used for the etching of aluminium components before anodizing.[11] The percentage of sodium hydroxide to water was 5% by weight.

4. Sodium Chloride Solution

This reagent is meant to be a simulation of sea water. The percentage of sodium chloride to distilled water was 3% by weight. The salinity of the sodium chloride solution was monitored using a hydrometer every week-end.

EXPERIMENTS

1. Joint Preparation

The adherend surface was roughened with grade "0" sanding cloth and cleaned with trichloroethylene according to the procedures described in ASTM D2093.[12]

The adherend to be treated was held on a specially designed metal plate and the sanding cloth was held on a metal block as shown in Figure 2. The sanding direction was guided so that the lays made by the sanding cloth were perpendicular to the length of the adherend as shown in Figure 3. The number of to and fro sanding movements was 15 times, and the sanding cloth was discarded after it was used for sanding 3 pieces of adherend.

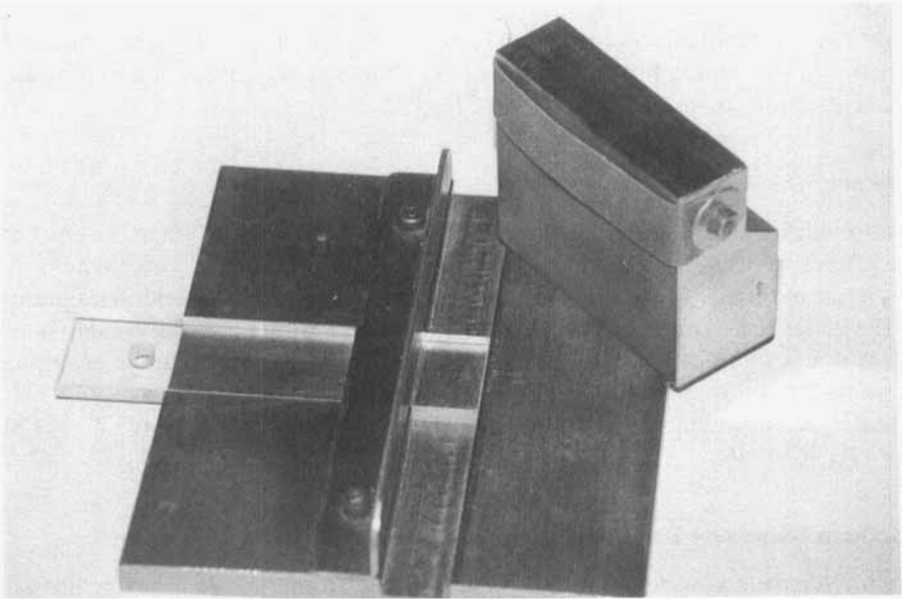

FIGURE 2 Sanding fixture.

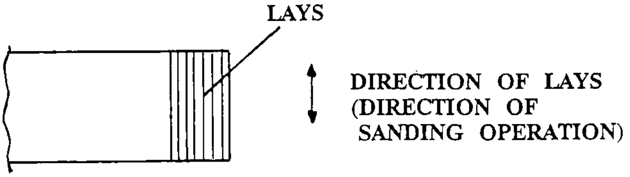

FIGURE 3 Direction of lays.

2. Bonding Procedure

The adherends cut from cast PMMA sheets and aluminium plates were prepared with the procedures stated above. Then the adhesive tape was stuck on the surface of one of the adherends to control the adhesive layer thickness as shown in Figure 4. The small width and the low adhesive strength of the tape produced minimal effect on the strength of the joint. The edges of the adherends around the adhesion zone were wrapped with adhesive tape, so that the epoxy overflow fillet would not

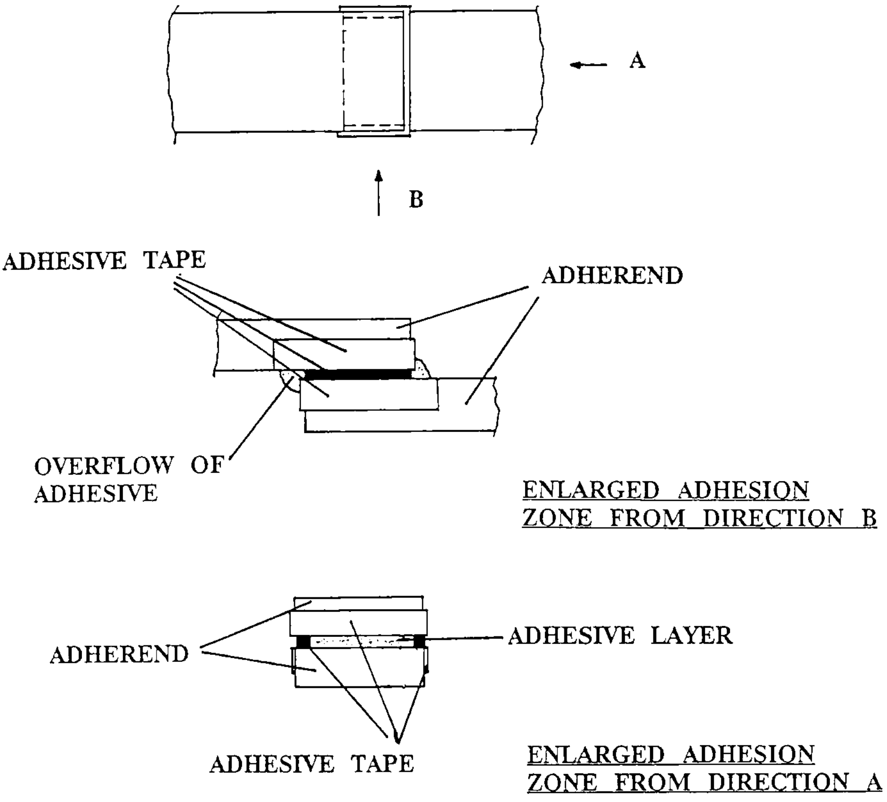

FIGURE 4 Positions of adhesive tapes.

TABLE I
Mean initial joint strength and its standard deviation

	Strength (N)	Standard deviation
Aluminium joint	2623–3595	56–303
PMMA joint	703–952	23–101

bond with the edges of the adherends. Because of the weak adhesion between the adhesive tape and the adherend, the test result should be not affected. The adherends were used immediately after surface treatment to reduce contamination.

The joints were prepared in batches of 10 specimens per batch, under a controlled laboratory environment with temperature ranging from 19°C to 24°C and relative humidity 50% to 80%. The adhesive, mixed according to the ratio suggested by the manufacturer, was applied to the adherends immediately with a spatula. A weight of 80 g was placed on the joint to hold down the adherends to ensure even spreading of the adhesive. The appropriate weight had been determined by experience.

The prepared joints were used 7 days after preparation to eliminate strength variation due to storage and to ensure complete setting of the adhesive.

The adhesive tape applied around the edges of the lap region was not removed before the joint was tested in order to prevent the formation of microcracks during the tape removal process. Table I shows the range of the mean initial strength and the standard deviation of all the batches of adhesive joints tested in the study which is within approximately 10% of the mean.

3. Tests

Immersion Test with Static Load The prepared adhesive joints were mounted on racks and subjected to static tensile load, and immersed in different reagents, as shown in Figure 5. The containers were located in a controlled laboratory environment. The load applied was 300 N which was 38% of the mean of the initial strength of the PMMA joints. It was also the load that, when applied to the joints in air, produced no crack within 7 days. It was observed that if 50% of the strength was used, cracks would appear on the joints within 2 days.

The time periods chosen for the immersion tests were 7, 20, 30, and 60 days. According to ASTM D896,[13] when a specimen is tested in a chemical reagent, the recommended test period is 7 days.

From the manufacturer's specification of the adhesive,[14] the adhesive strength is reduced to about 70% of the maximum after 30 days in a tropical weathering test, and reduced to 64% after 60 days. Also, the strength is reduced by 30% with water immersion at 20°C for 60 days. Therefore, an immersion test with 60 days as the upper time limit was adopted, and 30 days was chosen as one of the time intervals in the tests.

However, it was found that greater reduction in strength took place within 30 days than between 30 to 60 days. Hence, one more interval of 20 days was added between 7 and 30 day period.

At the end of each time period, the specimens were removed from the reagents,

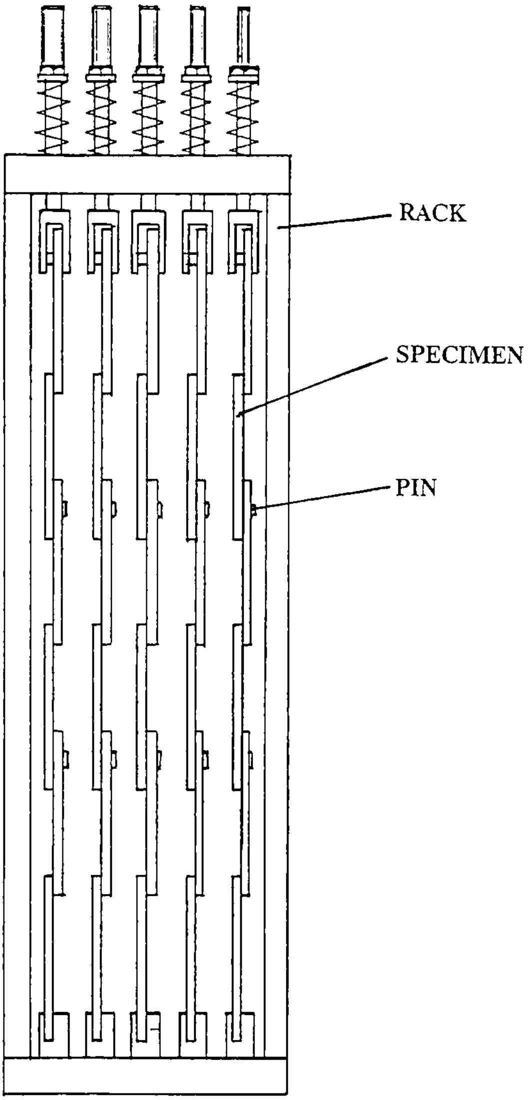

FIGURE 5 Setup for immersion test with static load.

washed in distilled water, dried with a soft wiper, and then tested by using a tensile tester with a cross-head speed of 4.5 mm per minute.

Since the sodium hydroxide solution can attack aluminium quickly, the aluminium joints were not tested in this reagent.

Free Immersion Test The prepared adhesive joints were freely immersed in different reagents in the controlled laboratory environment without any load applied. The test was conducted for two time periods of 30 days and 60 days. For each time period, three PMMA joints and three aluminium joints were used. As in static load tests, the aluminium joint was not used in the sodium hydroxide solution.

Test on Epoxy Properties Three blocks each of epoxy and PMMA were prepared and immersed in the reagents to examine the variation, if any, in the properties of these materials after different periods of immersion in different reagents. The same number of test blocks were hung in air in the laboratory environment. The size of the epoxy cast blocks was 40 mm × 25 mm × 10 mm, and that of PMMA was 45 mm × 25.4 mm × 4.5 mm. The PMMA specimen used was the same as the adherend used for preparing the joints.

The hardness of the blocks was tested before immersion by using a Type D Durometer. The weight of each block was measured.

Similar to the loaded and free immersion tests, the test period of immersion was 60 days. Then the period was divided into 6 equal periods, each of 10 days. At the end of each period, the surface of the specimens were dried with a soft wiper, and then the hardness and the weight of the blocks of epoxy and PMMA were measured.

In order to ascertain that the test blocks were thoroughly dry before the test, they were baked in a temperature-controlled chamber at 40°C for 15 hours; the relative humidity in the chamber was around 40%.

Three pieces of unbaked blocks were also immersed in water and subjected to the same test procedure as the baked blocks in order to find if there was any difference between the baked and unbaked blocks. Table II shows the tests carried out on these test blocks.

In addition, tensile specimens of cast epoxy were prepared according to the type I specimen stated in ASTM D638,[15] and immersed in the reagents in order to find the change in tensile strength of the epoxy with respect to the time of immersion. Two time periods, 30 and 60 days, were employed.

RESULTS AND DISCUSSION

The results of the study are discussed in the following sections. All corresponding curves were drawn through the mean value of the variable such as joint strength and reagent absorption for each time period, and were smoothed. In order to ensure that the results were reliable, checks on out-of-trend results were carried out by

TABLE II
The test program of epoxy blocks

Block	Condition
Baked	Air
	Water
	Sodium Hydroxide Solution
	Sodium Chloride Solution
	Sulphur Dioxide Solution
Unbaked	Water

repeating experiments. Although deviations were found between the batches of joints, nonetheless, the results indicated a definite trend.

Reagent Absorption and Change in Hardness of Epoxy Blocks

Figure 6 shows the reagent absorption of baked epoxy resin blocks. The curves show that the epoxy blocks did absorb the reagents and hence increased in weight. The shape of the curves for different reagents are quite similar to each other. This indicates that the amount absorbed was approximately the same for different reagents.

A swelling phenomenon was observed and the colour of the blocks was darkened.

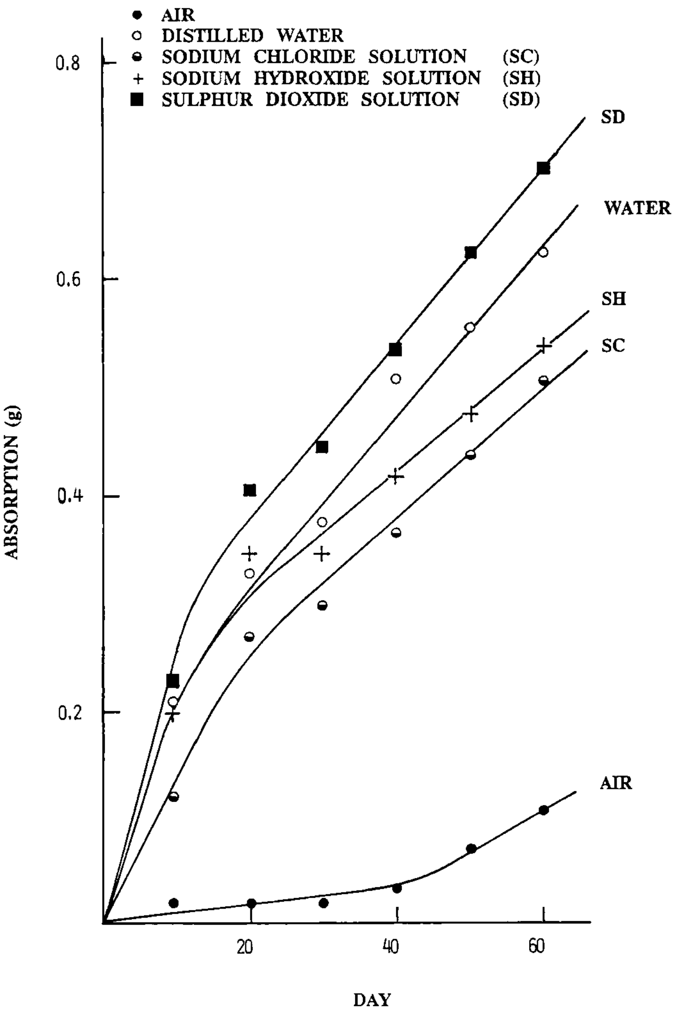

FIGURE 6 Reagent absorption of baked epoxy blocks.

This means that the blocks were probably chemically attacked by the absorbed reagents. The attack on epoxy was also reported by Althof.[16] He found that the amount of moisture absorbed decreased from the outer layer towards the centre of the specimen. This suggested that moisture was diffused from the surrounding environment towards the centre of the blocks.

Figure 7 shows the water absorption of the baked and unbaked epoxy resin blocks. The curves show that less water was absorbed by the unbaked epoxy block.

Figure 8 shows that as a result of the diffusion and attack of the reagents, the hardness of the specimens decreased. The change in hardness was slowed down after about 50-days immersion because the attack on the epoxy became steady, although more reagent was absorbed into the centre of the blocks. The hardness of

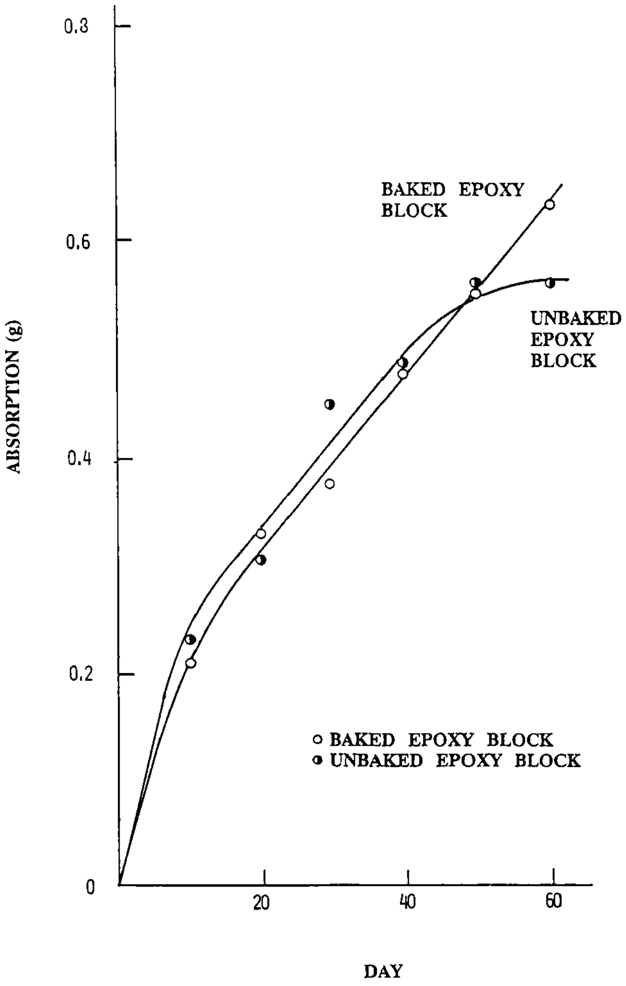

FIGURE 7 Water absorption of baked and unbaked epoxy blocks.

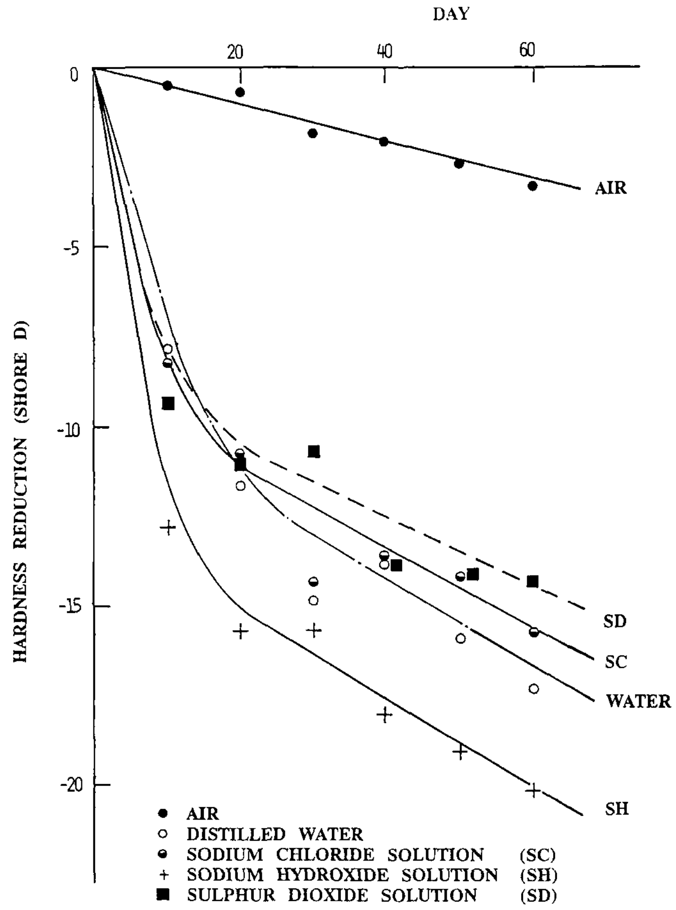

FIGURE 8 Hardness reduction of epoxy blocks after immersion.

the epoxy blocks in air also decreased but to a less extent, as only water vapour in the air was absorbed.

Table III shows the percentage of reduction in strength of the epoxy specimens after immersion in different reagents.

A change in hardness and strength of the epoxy specimens suggests changes in the rigidity, modulus of elasticity and shear modulus of the epoxy. The softening of the adhesive layer was also observed when the cleaned joint was loaded in the tensile tester until failure. It was found that the adhesive layer was soft just after the failure of the specimen and became harder after about 30 minutes. The change may be due to evaporation of the absorbed reagent in the thin adhesive layer.

The softened epoxy layer can distribute the stress more evenly to reduce stress concentration. More stress can be absorbed by the adhesive layer, which results in an increase in the strength of the joint. Althof[16,17] stated that the moisture content

TABLE III
The percentage of reduction in strength of the epoxy specimens

Solution	After 30 days (%)	After 60 days (%)
Sodium Hydroxide	−29	−21
Sodium Chloride	−44	−47
Sulphur Dioxide	−45	−47
Water	−44	−47

in the adhesive layer reduced the stress at the ends and increased the stress in the middle of the adhesive zone. In addition, Wang[18] stated that the stress distribution in the adhesive layer of the joint was influenced by the modulus of elasticity and shear modulus of the adhesive.

The reduced rigidity in the structure of the adhesive layer decreases the restriction to the movement of the molecules of the adhesive. When the joints were loaded in the tensile tester, the movement and deformation of the molecules could absorb part of the energy applied on the joint and help to spread the applied load more evenly over the adhesive zone.

The PMMA blocks were very stable in the reagents and no significant variations in hardness and weight of the test blocks were found.

Immersion Tests with Static Load

1. Aluminium Joint Figure 9 shows the percentage change in tensile shear strength of aluminium joints after different periods of immersion in different reagents as compared with the pre-test tensile shear strength of the joint. It can be seen that the strength of the aluminium joints decreased in the tests, particularly when the joints were immersed in salty water. However, the curve of the strength is fluctuating. This may be explained by the random strength variation of different prepared joints. Since most of the results are located on the negative side of the graph, that is, below the zero line, it can be concluded that the joint strength deteriorated as a result of immersion. These results agree with the observations of A. J. Kinloch.[20,22]

Comyn et al.[7] and Deryaguin et al.[19] treated the adhesive joint as a capacitor of infinite length. Absorption and aggregation of the reagent in the interface and in the adhesive layer will weaken the effect of ion-pair interactions. This is due to the increase in dielectric constant and could explain some of the observed property degradation. Another factor could be the hydrolysis of the anodic oxide. This could change the oxide into the mechanically weak aluminium hydroxide which adheres less firmly to the aluminium substrate as reported by Kinloch and Minford.[20,21,22]

The strength is mostly affected by the sodium chloride solution due to the corrosion of aluminium.[20,21,22,23] This is supported by Figure 10 which shows that the adherends were corroded around and underneath the adhesive film. The sulphur dioxide solution can also exert the same effect on aluminium but to a lesser extent.

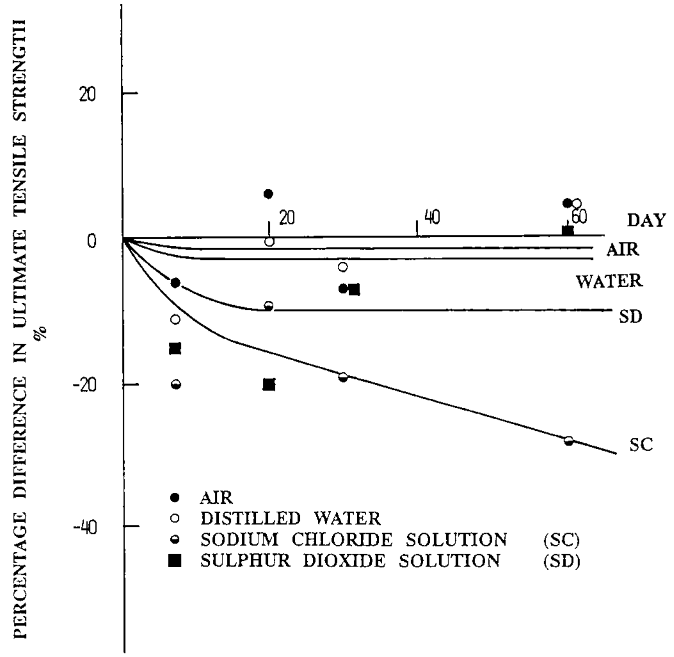

FIGURE 9 Percentage change in tensile strength of aluminium joints after immersion.

FIGURE 10 Corroded aluminium joints (after 60-days immersion in sodium chloride solution). See Color Plate I.

The aluminium joints were mounted on a steel rack which was cathodic to aluminium. Electrochemical corrosion occurred and the surface of the aluminium adherend was roughened. This roughening process, in turn, enhanced the localized corrosion by pitting and forming of an electrochemical cell between that part of the aluminium covered by epoxy and the part which was not. It was observed that localized corrosion of the aluminium adherend occurred at the edge of the adhesion zone. Cracks could easily develop in the corroded area and reduce the joint strength.

The situation would be worse if the adherend surface cavities were not fully filled by the epoxy or when a crack was formed since they would provide an opportunity for corrosion to take place.

2. *PMMA Joints* Figure 11 shows the percentage change in the tensile strength of the PMMA joints after different periods of immersion in different reagents as

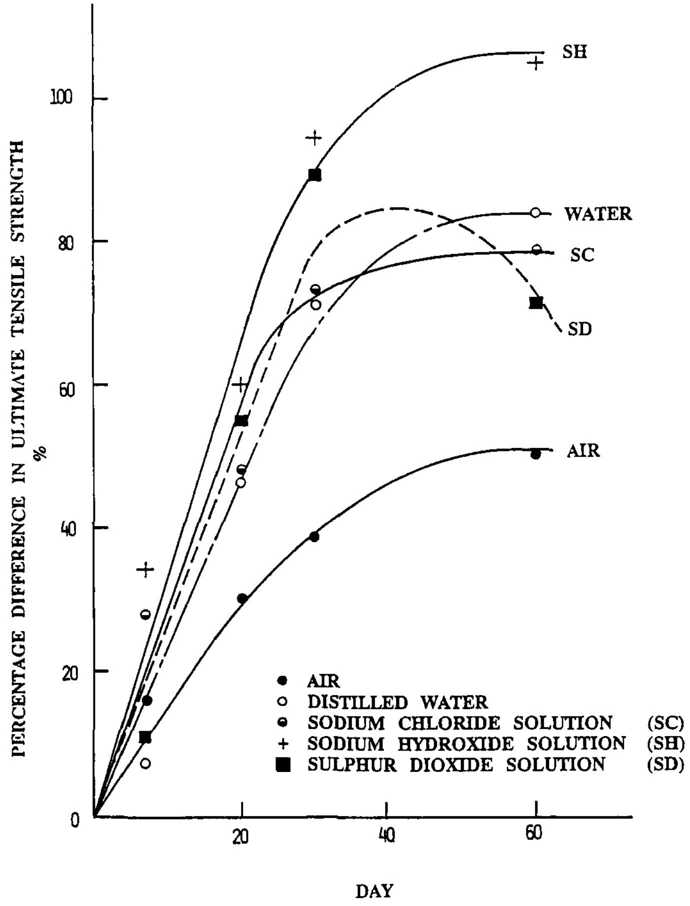

FIGURE 11 Percentage change in tensile strength of PMMA joints after immersion.

compared with the pre-test tensile strength of the joint. It can be seen that the results are totally different from that of the aluminium joints. The joint strength increased instead of decreased. Initially the joint strength increased at a fast rate, followed by a further increase but at a slower rate, except for the case of immersion in the sulphur dioxide solution which showed a decrease after an initial gain in joint strength. This suggests that the strength of the PMMA joint with the epoxy adhesive will be reduced after prolonged immersion in the sulphur dioxide solution.

Besides the effect on joint strength, a change in the rate of crack propagation was also observed. Before immersion, the joint was firstly deformed under a tensile load and then broken by the load with identifiable crack propagation. The crack was formed either at one end or at both ends of the adhesive zone. When the crack was formed at one end, it propagated along the adhesive zone towards the other end of the zone. When the cracks were formed at both ends of the joint, they propagated towards the opposite ends, and finally met at the centre of the adhesive zone.

After immersion, the manner in which the joint failed was altered. Under a tensile load, the joint firstly deformed. When the load reached a certain value, a crack was formed at one end of the adhesive zone and when the load increased, the crack immediately extended to reach the other end of the adhesive zone. That is, the joint broke at once without identifiable crack propagation.

Although the results showed that the strength of the PMMA joint increased after immersion, there was no "warning signal" before failure occurred. This is not a good characteristic for real life applications since a corrective action cannot be taken before failure occurs.

Such a phenomenon was observed in the PMMA joint because of its transparency. The stress-strain curve of the PMMA joint also showed a clear point of crack initiation. Figure 12 shows the simplified stress-strain curves of the PMMA joints

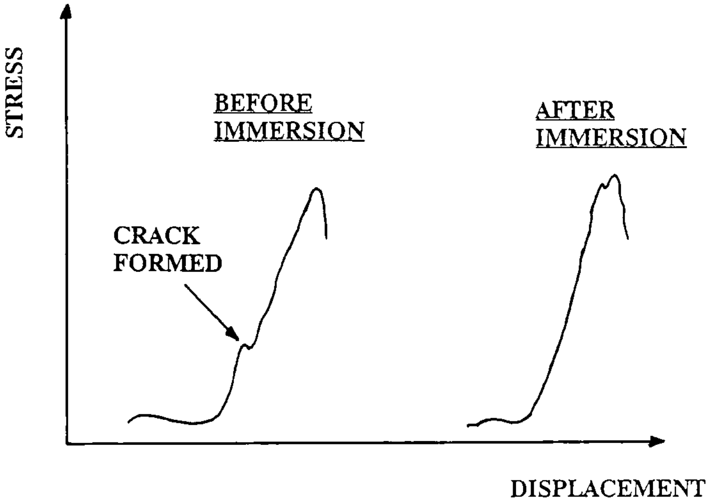

FIGURE 12 Simplified stress-strain curves of PMMA joints before and after immersion.

with and without immersion. The point that indicates the formation of a crack of the immersed joint cannot be clearly identified and is very close to the failure point. On the other hand, no clear crack initiation point was found on the stress-strain curves of the aluminium joints before and after immersion, therefore such a phenomenon may not exist in the aluminium joint under a tensile load within the range of test conditions used in this study. However, Watts, Castle and Hall[6] carried out a 180° test of an aluminium joint and found that, after water immersion, the gradual crack growth observed with the dry joint was changed to a sudden failure for the wet joint. They concluded that the effect of water on the adhesive led to the embrittlement of the interface.

Free Immersion Test

Table IV shows the results of the free immersion test of the aluminium joints in terms of the mean value of the percentage deviation from the strength of the joints before immersion. The values are also compared with those of the joints subjected to the immersion test under load. It shows that the strength of both the freely immersed specimens and the loaded was reduced. As the applied load was too low (300 N only), the percentage deviation was small except when the joints were immersed in the sodium chloride solution. The combined effect of load and corrosive environment with the chloride ion present[24,25] accelerated the corrosion rate of the aluminium adherend.

Table V shows the free immersion test of the PMMA joints. The strength of the joints was increased but to a lesser extent than that obtained from the loaded test. The percentages show that the applied load can lead to a much higher increment of strength after immersion.

PMMA and Aluminium Joints in Adverse Environment

The difference between the results of the PMMA and aluminium joints under the same adverse environment shows that the attack on the aluminium joints is greater than that on the PMMA joints. The attack on the adhesive layer should be the same for both joints. Water can diffuse into, and aggregate in, the adhesive region even with the PMMA adherend, especially when there are imperfections at the interface

TABLE IV
The deviation of aluminium joint strength after immersion

	% Deviation in joint strength		
Solution	After 30 days (%)	After 60 days (%)	Loading
Sulphur Dioxide	−2	−7	Free
	−6	1	Loaded
Sodium Chloride	0	−3	Free
	−19	−28	Loaded
Water	−7	−7	Free
	−4	4	Loaded

TABLE V
The deviation of PMMA joint strength after immersion

	% Deviation in joint strength		
Solution	After 30 days (%)	After 60 days (%)	Loading
Sulphur Dioxide	5	2	Free
	89	71	Loaded
Sodium Hydroxide	1	0	Free
	94	105	Loaded
Sodium Chloride	2	9	Free
	74	79	Loaded
Water	−1	18	Free
	71	84	Loaded

such as voids and cracks. However, in the aluminium joint water diffusing into the region can react with, and weaken, the metal oxide. Therefore, the difference between PMMA and aluminium joints could be attributed to the environmental resistance of the adherends. That is, since the aluminium surface is more reactive to its environment than PMMA, the bonding at the interface between the adhesive layer and the adherend could be affected by other factors including the hydrolysis of the anodic oxide, and also electrochemical corrosion of the aluminium adherend.

CONCLUSION

1. The results suggest that the strength of the aluminium joints was reduced after immersion in the different reagents. The static load applied was too low to have any significant influence on the aluminium joint strength, except when immersed in the sodium chloride solution.
2. In the sodium chloride solution, the aluminium adherend was corroded around and underneath the epoxy film. The corrosion enhanced the deterioration of these joints. This suggests that corrosion prevention, which is recognised as one of the merits of the adhesive joint, is not applicable in some applications involving prolonged immersion, especially in a marine atmosphere.
3. Under the same test conditions used for the aluminium joint, the PMMA joints behaved differently. The joint strength increased instead of decreased as in the case of the aluminium joint. In addition, the loaded specimens showed much greater increase in joint strength. Since the PMMA joint which was immersed in the sulphur dioxide solution showed a decrease after the initial gain in joint strength, the PMMA joint should not be subjected to prolonged exposure in an industrial area.
4. The improved strength of the PMMA joints may be due to the change in the epoxy adhesive properties. It was found that the adhesive absorbed the reagent and was attacked by it, resulting in the reduction of hardness. The increase in the flexibility of the adhesive, on the other hand, reduced the stress concentration and so increased the strength of the joint.

5. Although the strength of the PMMA joint increased after immersion, this was offset by the loss of any warning signal of impending joint failure. In practice, this means the joint may not be mended in a timely way before the failure occurs.
6. The effects of the reagents on joint strength can either be positive such as the increase of epoxy flexibility, or negative such as the corrosion of adherend and the deterioration of interface adhesion.

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