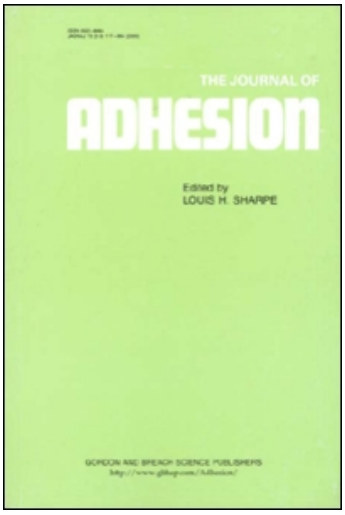

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Fatigue Crack Growth in Epoxy/Aluminum and Epoxy/Steel Joints*

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The fatigue crack growth rate within epoxy/aluminum and epoxy/steel joints was evaluated as a function of a) type of surface pretreatment, b) water soak, c) fatigue cycle rate (Hz), d) adhesive thickness and e) type of epoxy adhesive.

For both adherends, aluminum and steel, a significant improvement in the fatigue behavior was obtained by use of a mercaptoester coupling agent. After an 8-day, 57°C water soak, the metal surfaces which were pretreated with coupling agent (CA) or by phosphoric acid anodization (PAA) still resulted in cohesive failure, while the controls had higher crack growth rate and showed greater scatter. The room-temperature cure matrix with CA-treated aluminum showed a less dramatic improvement, probably because of a known difference in the application procedure. For the steel joints and room-temperature adhesive the improvement in the fatigue behavior of CA-treated samples was maintained after the 8-day hot water soak. No significant change was found in the fatigue crack growth rate over a frequency range of 1 to 5 Hz, but a significant change was found as a function of the bondline thickness. The room temperature curing adhesive evaluated herein exhibited a much lower fatigue resistance than a heat-cured commercial structural adhesive FM-73.

KEY WORDS mercaptoester coupling agent; phosphoric acid anodization (PAA); surface pretreatment; water soak; film adhesive; room temperature curing adhesive; cyclic loading; durability; double cantilever beam specimen; fracture mechanics; effect of bondline thickness.

INTRODUCTION

The potential number of applications for adhesives in bonding together metal components as well as composite materials is rising rapidly. Interest is particularly high in the aerospace, automotive and marine communities where many new applications will require bond integrity over a long period of time. It is known that both moisture and cyclic loading can accelerate failure of bonds, and both of these elements are present in many application areas of interest. Most of the information relating to the durability of adhesive joints has come from studies of strength after long-term aging in natural outdoor environments, or after laboratory testing of varying duration. The materials most frequently investigated have been those of

*One of a Collection of papers honoring A. J. Kinloch, the recipient in February 1992 of *The Adhesion Society Award for Excellence in Adhesion Science, Sponsored by 3M.*

interest in the aircraft industry, *viz.* aluminum adherends and heat-cured adhesives. Much less attention has been given to other metals and room-temperature cured adhesives. However, there is no general agreement regarding appropriate failure rules or test procedures for joints subjected to cyclic loading. Currently, the state of the art in testing adhesives is primarily based upon static strength or fracture toughness properties. The American Society for Testing and Materials recommendations contain only one standard for measuring fatigue strength of bonds (using the somewhat questionable single lap shear specimen).[1,2]

Very often an adhesive joint fails by separation of the adhesive from the adherends. Furthermore, failure is initiated at flaws in the adhesive bond. This suggests that the methods of fracture mechanics would be useful in the study of adhesive joint failure and, indeed, in recent years, attention has been focused on fracture mechanics description for failure rather than the conventional failure criterion based on stress/number of cycles.[3–7]

Numerous fatigue crack growth "laws" have been stated in the past, many of which have been reviewed by Luckyram and Vardy.[8] The empirical equation of Paris and Erdogan is widely used. The equation can be expressed in terms of strain energy release rate G as

$$da/dN = A(\Delta G)^n$$

where a is the crack length, N the number of cycles and A and n are material constants. The measured debond growth rate, da/dN, data are correlated with the corresponding strain energy release rate range, $\Delta G = G_{max} - G_{min}$. A least-squares fit can be applied to a plot of log da/dN as a function of logΔG to obtain the two constants.

Mostovoy and Ripling[9,10] pioneered the use of fracture mechanics in analyzing adhesives and mostly used TDCB (tapered double cantilever beam) specimens. These are easily pre-cracked to produce a sharp crack[11] but are relatively difficult to fabricate. On the other hand, DCB (double cantilever beam) specimens are very simple and inexpensive to fabricate and are excellent for screening adherend surface quality. For either type of specimen the results may not be valid if extensive plastic deformation of the adherends occurs when testing a tough adhesive[12] and the standards for linear elastic fracture mechanics (LEFM) are not met.

In the present work, DCB specimens were chosen to evaluate the fatigue crack growth behavior of metal/adhesive/metal joints in Mode I (opening mode) under cyclic loading conditions. Joints were tested before and after exposure to hot water and with and without the use of a coupling agent.

Previous work[13–16] has shown that mercaptoester coupling agents produce a substantial improvement in the adhesion durability of epoxy/metal bonds. These coupling agents provide a thiol group which can bond both to several metals and to adhesives containing thiol-reactive moieties such as epoxy groups, double bonds, etc. The specific coupling agent used in the present work was pentaerythritol tetra 3-mercaptopropionate (PETM). Use of PETM for static test specimens (epoxy/steel) has been reported previously.[14–16]

For aluminum joints the fatigue crack growth rate was evaluated with and without the mercaptoester coupling agent. The results were compared with a phosphoric

acid anodized (PAA) surface treatment, now used by a large number of aircraft manufacturers to provide improved bond durability.

The effects of the bondline thickness and the cyclic loading frequency were also investigated, using the room temperature cured adhesive system.

Some steel adherends were treated with a PETM coupling agent following an ammonium citrate treatment. The ammonium citrate is believed to remove the oxide layer and to activate the surface by other mechanism(s) that are not completely clear.[17]

EXPERIMENTAL

Materials

Two epoxy systems were used: The first was a room temperature-curing epoxy composed of a standard diglycidyl ether of bisphenol A resin (DGEBA), Epon 828*, blended with an aliphatic diglycidyl ether epoxy resin, Epon 871*. The Epon 871* was added to increase the ductility of the system. The epoxy mixture was cured with the common polyamide curing agent V-40*. This three component curing system was prepared by mixing the Epon 828, Epon 871 and V-40 in a ratio of 4:3:3 by weight at room temperature. The mixture was degassed for 25 minutes at 50°C. The second epoxy system was FM-73** film adhesive, a 250°F (121°C) curing structural adhesive, that is a toughened, general purpose epoxy widely used in the aerospace industry. The FM-73 film adhesive, applied in one layer, was cured according to the manufacturer's recommended curing cycle, 60 minutes at 250°F (121°C) and 40 psi (0.28 MPa), following a 6°F (3.3°C)/min heating rate.

Steel (SAE1018), of interest in automotive and general structural applications, and aluminum (6061T-6), of interest in aerospace and naval applications, were selected as substrate materials.

Test Specimens

The DCB specimens used throughout this work were of the type described in the ASTM D-3433 procedure: "Fracture Strength in Cleavage of Adhesives in Bonded Joints." Both aluminum and steel adherend beams had dimensions of $16.0 \times 2.54 \times 1.27$ cm. A drawing of the specimen assembly is given in the Appendix. The strain energy release rate, G, was calculated for a double cantilever beam (DCB) in accord with ASTM D-3433:[23]

$$G = \frac{4P^2(3a^2 + h^2)}{Eh^3B^2} \tag{1}$$

For cyclic loading the corresponding expression for ΔG is

$$\Delta G = \frac{4(P_{max}^2 - P_{min}^2)(3a^2 + h^2)}{Eh^3B^2} \tag{2}$$

*Trade name of Shell Chemical Company.
**Trade name of American Cyanamid Company.

Here P is the applied load, a is the crack length, h is the half-height of the double cantilever beam, E is the Young's modulus of the adherend and B is the thickness of the beam. Although this method of calculating ΔG has been used in other studies of the characteristics of adhesive joints, it is noted that it does not represent the actual strain energy release rate of the adhesive joint inasmuch as the characteristics of the adhesive joint itself, *i.e.*, its modulus and thickness, are not reflected in the above equation for ΔG. ΔG as defined above is used herein only as a characterization parameter for purpose of comparison. It is noted that in the calculation of ΔG the presence of the adhesive bond is not taken into account.

Preparation of Joints

The cleaning procedure for both adherends consisted of grit blasting with a 600 grade grit followed by a solvent wipe by a paper towel soaked in acetone. The adherends were then degreased for an additional 30 minutes in acetone at room temperature.

The phosphoric acid anodization for the aluminum specimens, applied after the previous cleaning procedure, was carried out according to Boeing specification BAC 5555 (Boeing Aircraft, Seattle, WA, USA). The anodizing solution contained 100 g/l phosphoric acid and 7 g/l aluminum. The anodization was done for 22 minutes with 12 volts applied at room temperature. After this procedure the metals were rinsed in tap water for 12 minutes by immersion and then warm air dried for 60 minutes at 40°C. The adhesive was applied within less than 72 hours after this procedure.

The PETM coupling agent on the aluminum adherends was applied in the following manner: the specimen surfaces were prepared by grit blasting as described above, the surface was wiped with a methanol-soaked, lint-free cloth and then soaked in a methanol-bath for ½ hour. After this cleaning procedure the adherends were placed in a resin kettle containing 2.5×10^{-3} mole/liter PETM in methanol. The solution was refluxed for 15 minutes. After the solution was cooled, the specimens were removed and air dried for ½ hour. The PETM for the steel adherends was applied in the same manner but following an immersion for 10 minutes at 80°C in an ammonium citrate solution (30 g/liter of citric acid solution in water, neutralized to pH 7 by adding ammonium hydroxide), a distilled water bath for 10 minutes and a methanol bath for 5 minutes. The cleaned and treated adherends were taped from the side with a ½-inch (1.27 cm) wide Teflon® or polyimide* tape to prevent runout of excess resin which could cause additional bonding on the adherend faces. Before fatigue testing, the Teflon® or polyimide adhesive film was easily removed, and the specimen bond line was painted with a white coating (typewriter correction fluid) and marked at 5 mm increments to aid in crack detection and crack length determination.

Immediately after applying the adhesive, the two members of the double cantilever beam were clamped together, with the thickness of the adhesive joint being set by Teflon® shims. These shims were inserted at each end of the double cantilever

*Scotch Electrical Tape of Minnesota Mining and Manufacturing Co.

beam. The length of the shim at the end where the load was applied was 5.08 cm, and the length at the opposite end was 0.63 cm. The clamped adherends were then placed in a six-cavity mold and cured at room temperature or at elevated temperature (FM-73) with subsequent slow cool to room temperature.

Fatigue Tests

The fatigue tests were conducted using an Instron (Model No. 1350) servohydraulic test machine with a suitable pair of self-aligning pinned grips to hold the specimen in alignment when the load was applied. A stress ratio (ratio of minimum to maximum stress in a cycle) of 0.05 was used. The cyclic loading frequencies were between 1 and 5 Hz with a sinusoidal load waveform.

For most toughness measurements or fatigue crack growth evaluation, it is essential to start with a sharp, well-defined crack. To produce a sharp, natural starting crack the specimens were precracked under a static load before the start of the cyclic loading. The precracking procedure involved loading the specimen under displacement control until a crack appeared. The fatigue crack growth test was performed at a nominal load approaching about one-half of the load value used to introduce the crack. The crack tip was observed with a X20 movable telescope.

RESULTS AND DISCUSSION

Aluminum Joints

1. Surface Treatment The fatigue crack growth for the aluminum adherends with and without coupling agent or phosphoric acid anodization (PAA) surface treatments was evaluated for the two adhesives before and after water immersion. The bondline thickness was 0.10–0.13 mm for both adhesives, except as noted on Figures 1 and 5. The cyclic loading frequency was 1 Hz, which precluded the obtaining of data at very low crack growth rates, *i.e.* below 10^{-4} to 10^{-5} mm/cycle, because of the great amount of time required. The lines drawn through the data points on Figures 1–8 were determined by a least-squares fit of the data, using two different computer programs; fitting was done between the limits of points with the lowest and the highest values of ΔG_I for each case. Because there is scatter in the data, the amount of which varies among the various experimental conditions, one should not put too close an interpretation on the Paris Law constants; the relative differences between the sets of data are large enough to be visibly apparent on the figures, however. The lowest rate of fatigue crack growth measured in these experiments was 10^{-5} mm/cycle, well above the generally-accepted threshold level of 10^{-8} mm/cycle.[9] For purposes of comparison between samples, we will define the ΔG level corresponding to a growth rate of 10^{-3} mm/cycle as ΔG_{-3}. Values of ΔG_{-3} are given in Table I. We will also designate, as a matter of interest, an upper bound to the fracture toughness, G_c, as G_{UB}. The value of the upper bound, G_{UB}, was obtained by taking the crack length in a fatigue crack growth test at a given load amplitude as equal to the distance between the load line and the far end of the specimen, *i.e.*, 15.0 cm. This assumed crack length is, of course, larger than the

TABLE I

Adherend and adhesive	Exposure	Surface treatment	Bondline thickness [mm]	Test frequency [Hz]	ΔG_{-3} [J/m^2]	ΔG_{UB} [J/m^2]
Aluminum and FM-73		without CA	0.127	1	60	600
	R.T.	PETM	0.127 Fig. 1	1	100	550
		PAA	0.178	1	550	1800
	8 days	without CA	0.127 Fig. 2	1	~7	200
	water, 57°C	PETM	0.127	1	70	1000
Aluminum and R.T. curing epoxy		without CA	0.127	1	19	160
system	R.T.	PETM	0.127 Fig. 3	1	30	470
		PAA	0.127	1	180	525
	8 days	without CA	0.127	1	20	35
	water, 57°C	PETM	0.127 Fig. 4	1	9	175
		PAA	0.127	1	65	1030
			0.127	1	19	160
	R.T.	without CA	0.254 Fig. 5	1	40	370
			0.381	1	~60	370
			0.127	1	19	160
	R.T.	without CA	0.127 Fig. 6	3	24	245
			0.127	5	29	370
Steel and R.T. curing epoxy	R.T.	without CA	0.127 Fig. 7	1	30	70
system		PETM	0.127	1	70	315
	8 days	without CA	0.127 Fig. 8	1	40	525
	water, 57°C	PETM	0.127	1	80	1000

actual crack length at the point of unstable fracture. Values of G_{UB} are given in Table I.

a. Joints with FM-73 Structural Adhesive The fatigue data obtained with aluminum adherends for the structural adhesive FM-73 with different surface treatments at 1 Hz frequency are given in Figure 1. Approximate constants, ΔG_{UB} and ΔG_{-3}, are given in Table I.

While the data show considerable scatter, all specimens fall in the same general region. Inspection of the failed joint surfaces in all cases revealed resin on both sides, with less than 5% visually-bare area. Of the three samples, the aluminum without CA appeared to have more bare area than the other two, and in a separate

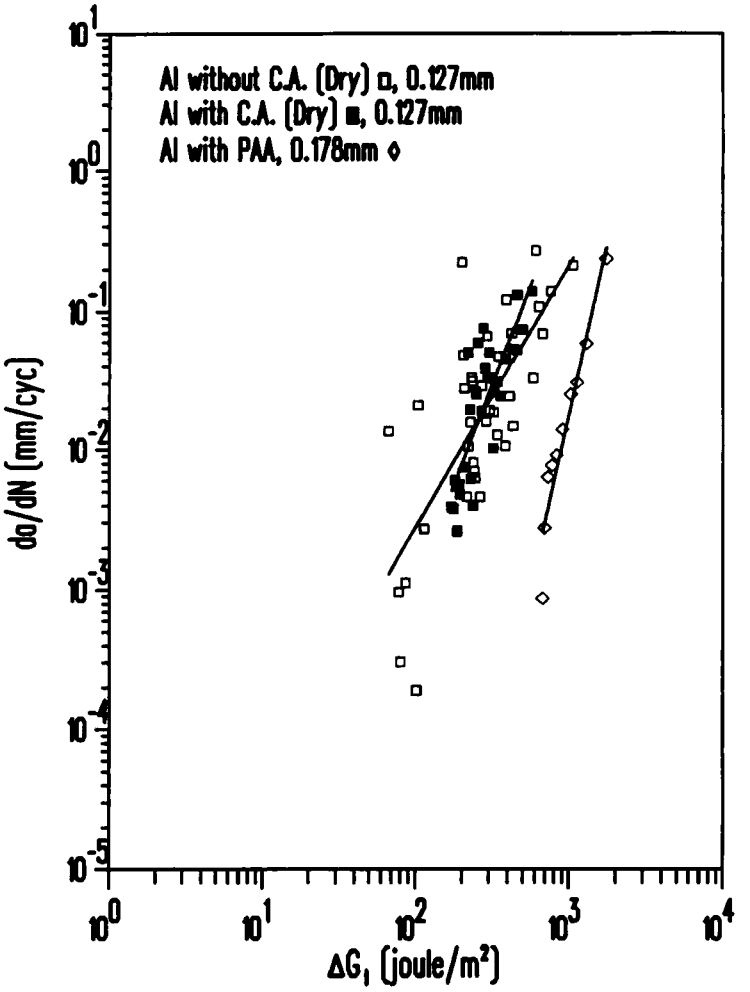

FIGURE 1 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with FM-73 structural adhesive and different surface treatments.

test using a different joint the failure of the untreated aluminum FM-73 joint was clearly adhesive ($\Delta G_{-3}=20$ J/m^2). The phosphoric acid anodized sample shows higher ΔG, for a given da/dN on Figure 1, but this may be due to the greater joint thickness, as will be discussed in a later section. The value of ΔG_{UB} for the dry PAA-treated samples was the highest of all samples tested (1,800 J/m^2).

Because, as reported in previous work,[13-16] a significant improvement was obtained in shear and peel strength of steel joints using mercaptoester coupling agents, especially after water exposure, the fatigue crack growth rate was also evaluated after 8 days of water immersion at 57°C following the same surface treatments. There is an even greater scatter in the data, particularly for the control sample (without coupling agent). The sample with coupling agent exhibited cohesive failure; the data for this sample are also shown as circles on Figure 2. The data fall

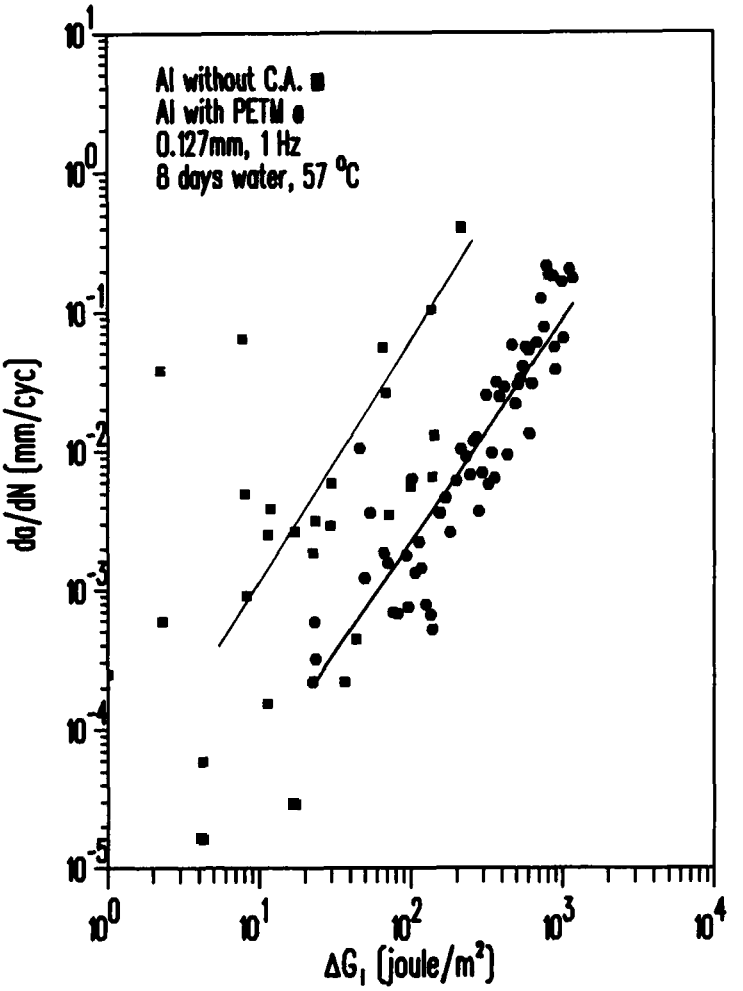

FIGURE 2 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with FM-73 structural adhesive without and with a mercaptoester coupling agent after water immersion.

in the same general region as the dry sample data on Figure 1. It is clear from Figure 2 that the control is not only far more erratic in crack growth, but also ΔG_1 for a given crack growth rate is, on the whole, considerably lower. Inspection of the water-soaked controls after failure revealed primarily adhesive failure, with the aluminum metal having a dull, greyish appearance.

b. Joints with room temperature cure epoxy system Figure 3 shows the crack growth rate *vs.* the opening mode strain energy release rate, ΔG_1, for the room temperature curing epoxy system and aluminum adherends. Table I shows approximate ΔG_{-3} and ΔG_{UB} for these different surface pretreatments.

The samples with coupling agent were prepared differently from those used with FM-73 adhesive; although 0.07 wt % coupling agent solution was used, the ratio of

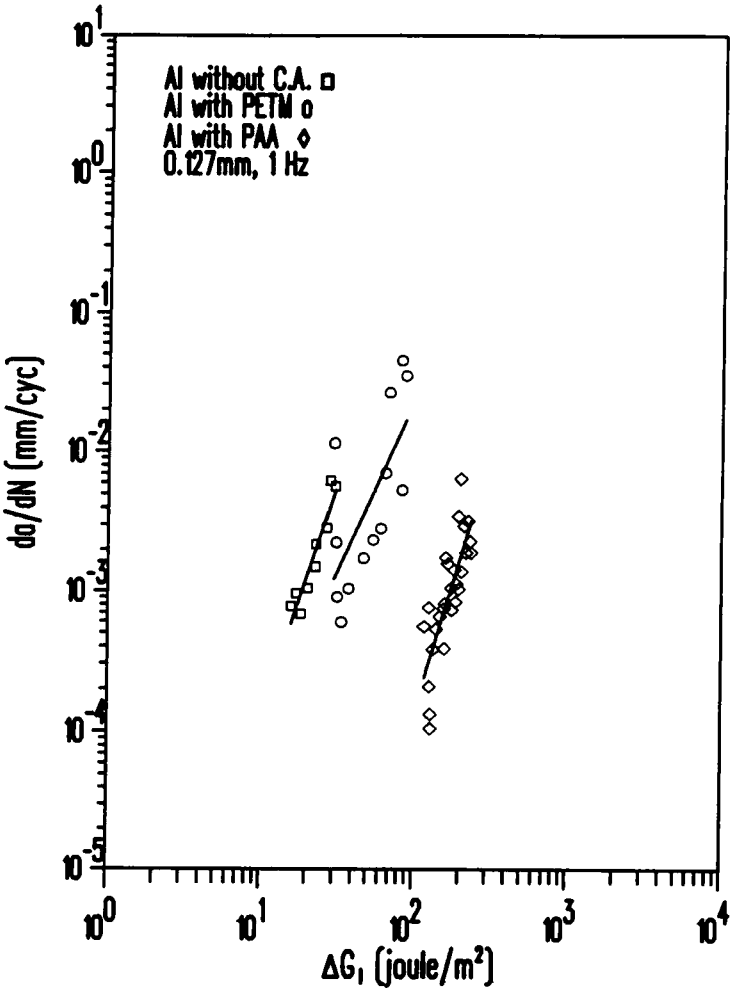

FIGURE 3 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with a room temperature curing epoxy system for different surface treatments.

metal (geometric) surface area (cm^2) to coupling agent solution volume (cm^3) was much higher, ~0.5 cm^{-1}, as compared with approximately 0.25 cm^{-1} reported in Figure 2. The higher surface-to-volume ratio gave a coupling agent thickness of *ca.* 50 Å, as compared with 150 Å at the higher solution volume. The lower coupling agent thickness very probably resulted in less ΔG_I improvement than was observed for FM-73 adhesive (Fig. 1).

The phosphoric acid anodized (PAA) sample with the room-temperature curing adhesive would be expected to exhibit strong bonding. The PAA results (Fig. 3) are in the same ΔG_I range (at a given da/dN) as the dry samples with FM-73 adhesive, again indicating cohesive failure. Inspection of the fractured PAA joints also indicated cohesive failure. For this resin system the control was significantly poorer than for the phosphoric acid anodized sample, and the coupling agent did not result in as much improvement, probably because of the application procedure (high surface/volume ratio) as discussed above.

Since the failure of the PAA-treated specimens was cohesive, the G_{UB} value should approximate the critical strain energy release rate of the pure resin. For comparison, G_{Ic} for the anodized specimen was also evaluated by a static fracture test (ASTM D-3433). The G_{Ic} value was found to be 490 J/m^2, in very good agreement with the 525 J/m^2 value obtained from the fatigue test. A good correlation between the average static fracture energy and the fatigue fracture energy also has been reported by Mall and Johnson for a graphite/epoxy composite system, bonded with a rubber-toughened epoxy and determined using DCB specimens.[18]

After 57°C water soak, the fatigue debond rate as a function of ΔG is shown in Figure 4. As can be seen from the ΔG_I scale, the fatigue crack growth rate for all the surface treatments was much higher than before water soak, with the best performance shown by the anodized specimen. No significant differences were observed in G_{-3} relative to the dry control. However, after 8 days in water at 57°C a much faster crack growth rate was detected.

The PETM treated specimen after 8 days in water followed by crack growth reached a G_{UB} value of 175 J/m^2, a value higher than for the non-PETM treated specimen (35 J/m^2). The G_{UB} value for the anodized specimen which was not given a water soak was 525 J/m^2, much lower than the value obtained after the 57°C water soak (1033 J/m^2). The failure, as estimated from visual inspections, was approximately 50% cohesive and 50% interfacial. The increase in the G value after water immersion is related to a toughening (plasticization) effect of the water and was obtained also with the steel adherends.

2. Bondline thickness Grit-blasted, acetone-degreased DCB aluminum specimens were used to examine the effects of the bond thickness. The procedures are described in the experimental section. The measured debond growth rate *vs.* the strain energy release rate ΔG_I (crack driving force) is shown in Figure 5 and the corresponding approximate ΔG_{-3} in Table I. It seems that the greater the thickness, the higher is the fatigue resistance. For the highest thickness (0.38 mm), the crack growth rates at $\Delta G > 10$ J/m^2 were always high; the crack tip was difficult to follow and fewer experimental data were obtained.

Most of the data reported in the literature regarding the influence of the bondline thickness deal with the effect on the fracture toughness under static load.

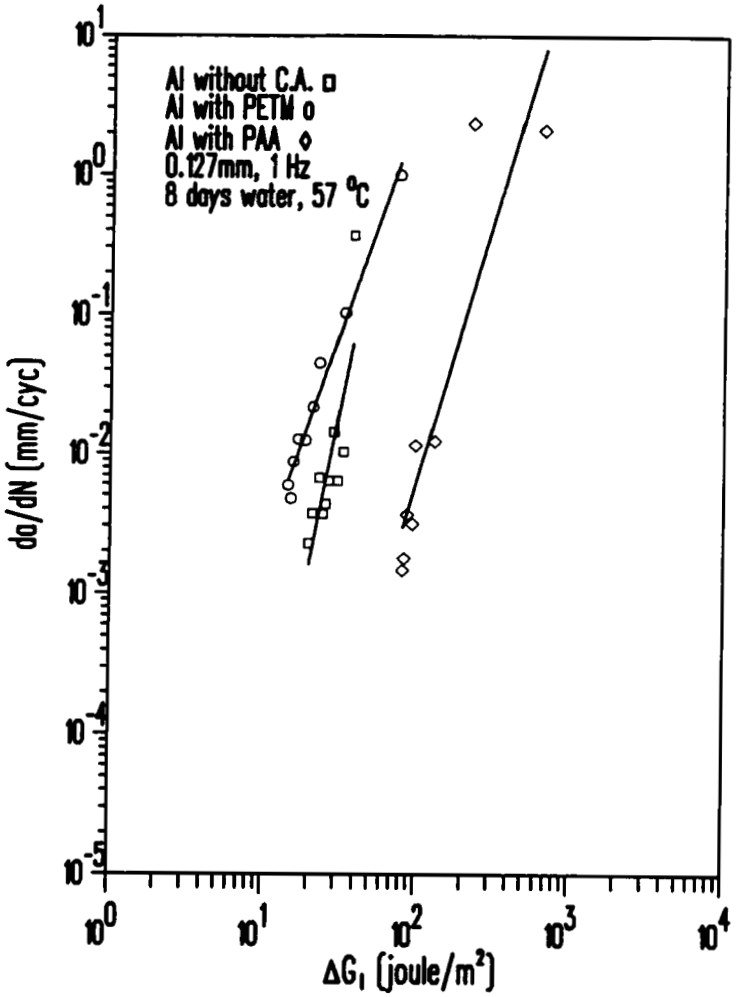

FIGURE 4 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with a room temperature curing epoxy system for different surface treatments after water immersion.

Spingarn,[4] using aluminum chevron-notched specimens with a nylon-modified epoxy adhesive, found that the fracture toughness increased and reached a maximum value at 75 μm, and then decreased to a plateau at about 250 μm. Kinloch,[19] referring to various workers (Mostovoy and Ripling, and Bascom *et al.*), showed that for adhesives possessing low values of G_{1c} or K_{1c} the effect of the adhesive bond thickness is minimized over the common range of thickness employed, say from about 0.08 to 1 mm, in tests using TCDB aluminum joints with DGEBA/piperidine-cured adhesive. The picture is different with a toughened adhesive (CTBN). A maximum G occurs when the bond thickness and the plastic zone diameter are approximately equivalent, about 0.6 mm. Beyond a thickness of 1 mm, G was independent of the bondline thickness.

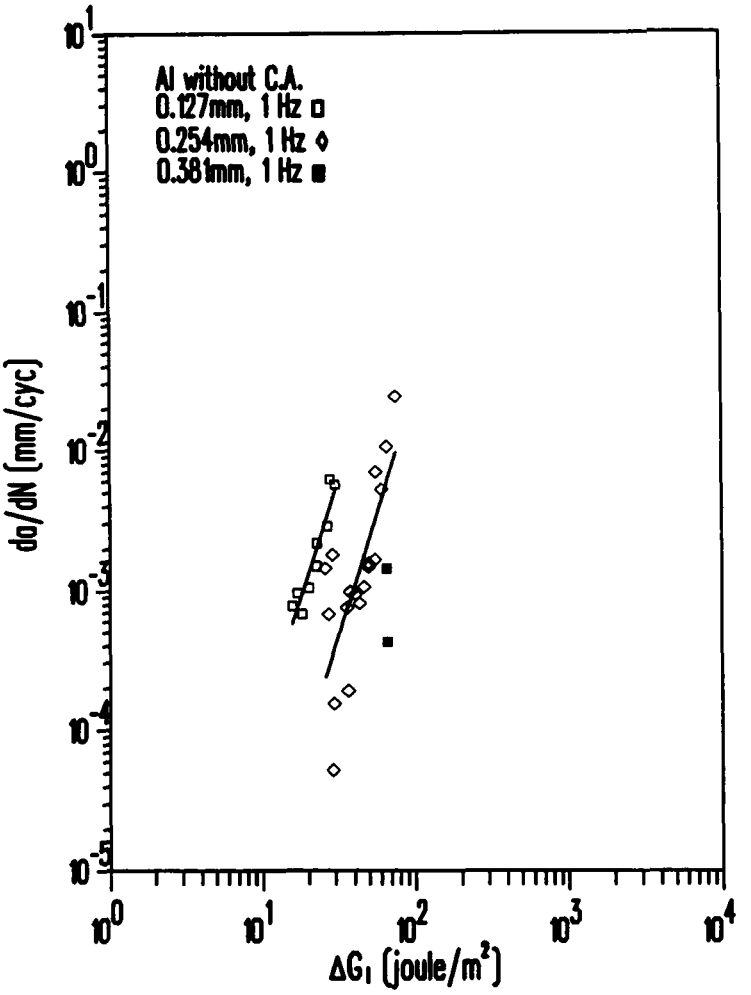

FIGURE 5 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with a room temperature curing epoxy system and different bondline thicknesses.

In contrast to many experimental findings, Crews *et al.*[20] using a finite element analysis concluded that the adhesive thickness has very little influence on the stress intensity factor of the crack driving force for the DCB aluminum/epoxy specimen. Mall and Ramamurthy,[21] using graphite/epoxy DCB specimens bonded with a rubber-toughened structural adhesive, found an increase in the critical strain energy values of 50% for a 0.51 mm bondline thickness compared with 0.10 mm and 0.25 mm thickness. They found that the fatigue resistance improved considerably with a 0.51 mm thick bondline at higher debond rates. However, this was not the case at lower debond growth rates (or near the threshold region), where the fatigue resistance was almost the same. In our case, we had a similar improvement in the fatigue resistance for the higher bondline thickness (0.38 mm) with highest differences in

the ΔG_{-3} values (19, 40 and 60 J/m^2 for the 0.13, 0.25 and 0.38 mm thicknesses, respectively). Schmueser and Johnson[22] found the same trend of increase in the strain energy release rate values. Their data were obtained from mixed-mode cyclic loading applied to primed, cracked lap-shear specimens. The bondline thickness varied between 0.305 and 1.27 mm.

3. Cyclic loading rate The influence of the cyclic loading frequency on the fatigue crack growth rate within aluminum/epoxy specimens was evaluated. The aluminum adherends were grit blasted and acetone degreased only. The test frequencies were 1, 3 and 5 Hz. The bondline thickness of all of the specimens was 0.13 mm. The fatigue crack growth rate as a function of ΔG is given in Figure 6 and the corresponding approximate ΔG_{-3} is given in Table I.

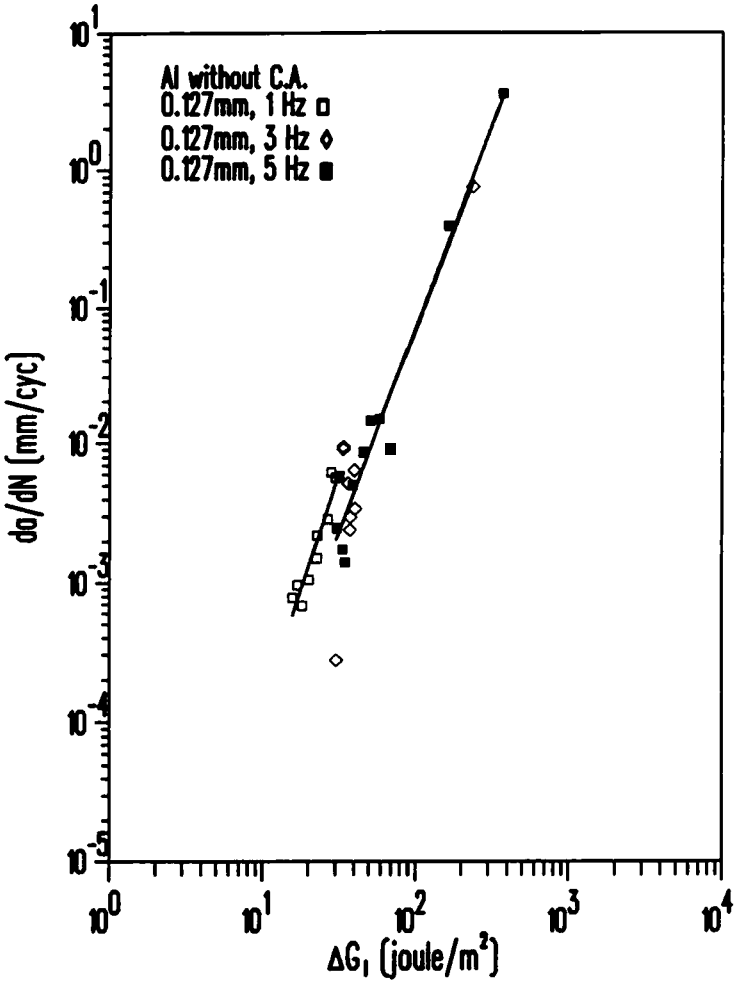

FIGURE 6 The fatigue crack growth rate *versus* the opening mode strain energy release rate for aluminum joints with a room temperature curing epoxy system and different cyclic loading frequencies.

As indicated in Figure 6, the fatigue crack growth rate is relatively frequency-independent in the 1–5 Hz range. A similar frequency independence in the range of 0.1–3.0 Hz[9] and 0.5–5 Hz[8] has been observed for other epoxy based adhesives.

Steel Joints

1. Surface Treatment The fatigue crack growth rate for steel joints with and without PETM coupling agent was evaluated using the room temperature-curing epoxy system, before and after water immersion. The PETM CA was applied using the ammonium citrate solution treatment described in the experimental section. The bondline thickness was 0.13 ± 0.025 mm and the loading frequency was 1 Hz. Figure 7 shows the crack growth rate as a function of ΔG and Figure 8 shows the

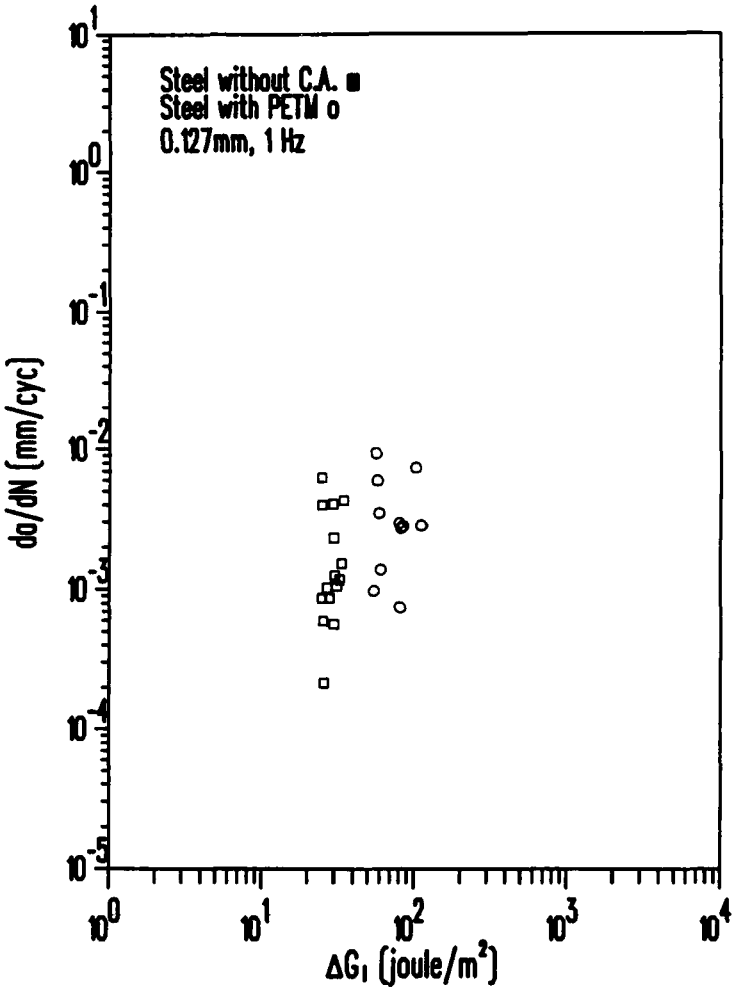

FIGURE 7 The fatigue crack growth rate *versus* the opening mode strain energy release rate for steel joints with a room temperature curing epoxy system without and with a mercaptoester coupling agent.

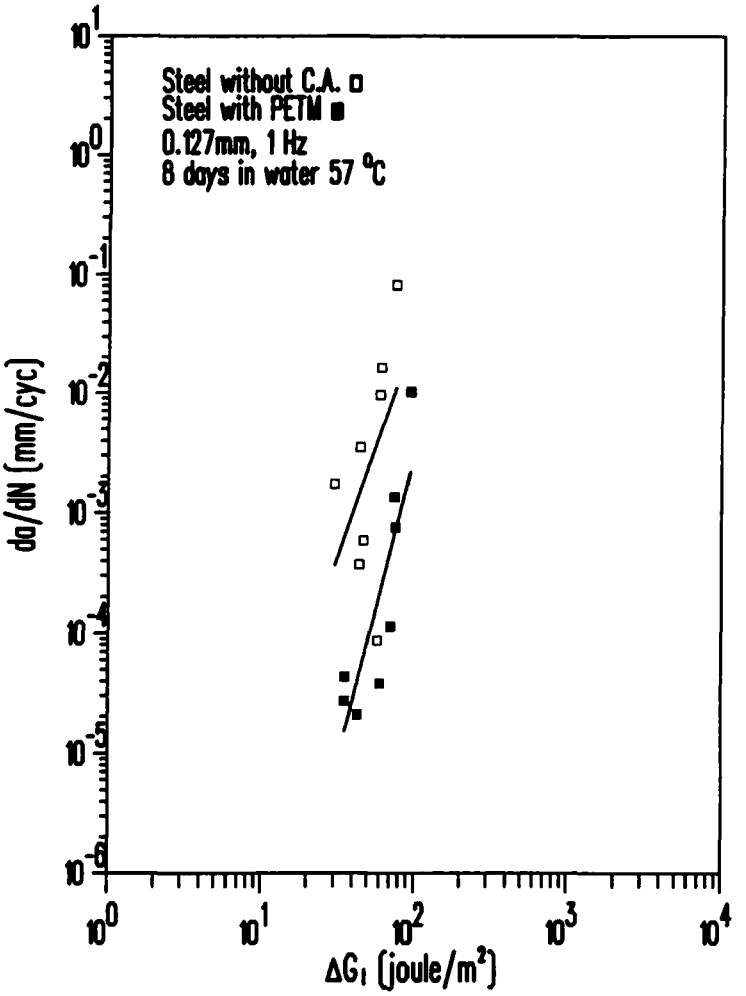

FIGURE 8 The fatigue crack growth rate *versus* the opening mode strain energy release rate for steel joints with a room temperature curing epoxy system without and with a mercaptoester coupling agent, after water immersion.

effect of 8 days of water immersion at 57°C on the fatigue crack growth rate. ΔG_{-3} values are given in Table I.

First of all, as can be seen from Table I, the fatigue crack growth rate is much higher for the steel than for the aluminum without CA and with the same resin system. The fatigue crack growth rate for the steel specimens is lower for the specimen with the PETM CA. ΔG_{-3} with the CA is higher than without the CA (70 J/m^2 and 30 J/m^2) as is the G_{UB} value (315 J/m^2 and 70 J/m^2, respectively). A comparison between the dry and wet data shows that the water improved the fatigue performance somewhat both with and without CA. This apparent toughening of the joints by water is associated with the higher G_{UB} values: 525 J/m^2 instead of 70 J/m^2 for the specimen without CA and 1000 J/m^2 instead of 315 J/m^2 for the speci-

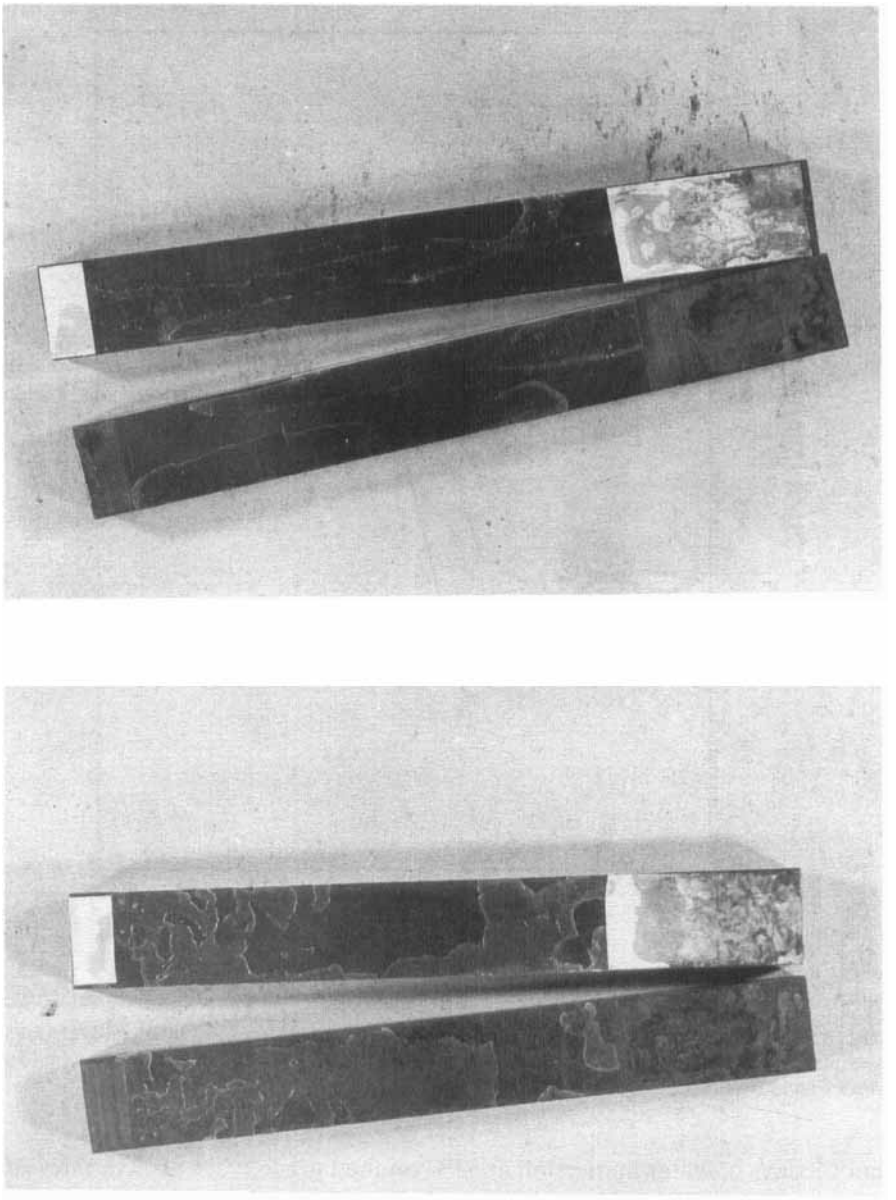

FIGURE 9 Failure surfaces for steel adherends with a room temperature curing epoxy system without (upper picture) and with a mercaptoester coupling agent (lower picture) after water immersion.

mens with the CA. Similar toughening after water exposure was observed by Mostovoy and Ripling[9] for an EPON 826/MPDA system and also for a nitrile-phenolic film adhesive.

The significant improvement in the fatigue behavior for CA relative to controls, both before and after water immersion, is accompanied by a completely different failure mode; while the failure for the specimens without CA appears interfacial with long strips of debonded material, the specimen with CA is still mostly interfacial but with many islands on both sides (see Fig. 9).

CONCLUSION

The fatigue crack growth rate of aluminum and steel joints with epoxy adhesives was evaluated on a comparative basis using simple DCB specimens.

An improvement was found for aluminum joints in fatigue behavior using a mercaptoester coupling agent (CA) for both adhesives tested: FM-73 film adhesive and a room temperature curing epoxy system. For the anodized specimen with the room temperature-curing epoxy system, good agreement was found between the G_{UB} value evaluated from the fatigue test and G_{1c} from a static fracture toughness test. A significant change in the fatigue behavior was found as a function of the bondline thickness. The higher the thickness, the higher was the ΔG_{-3} value. The highest thickness used in this study was 0.38 mm although higher thickness may be of interest, especially to the automobile industry. No significant change was found in the fatigue crack growth rate between 3 and 5 Hz frequencies. The growth rate at 1 Hz appeared slightly higher, but the significance of the difference is questionable.

For FM-73 adhesive the coupling-agent-treated samples showed cohesive failure in the FM-73 layer, both before and after 8 days of 57°C water exposure. The room temperature cured adhesive data did not show as large of an improvement, most probably because the different CA application method used for these samples resulted in a different CA thickness.

An improvement in the fatigue behavior for CA-treated steel joints was also maintained after water soak. After water immersion the fatigue performance for the steel joints was even better than before exposure, probably due to the plasticizing effect of the water on the room temperature curing epoxy system.

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APPENDIX

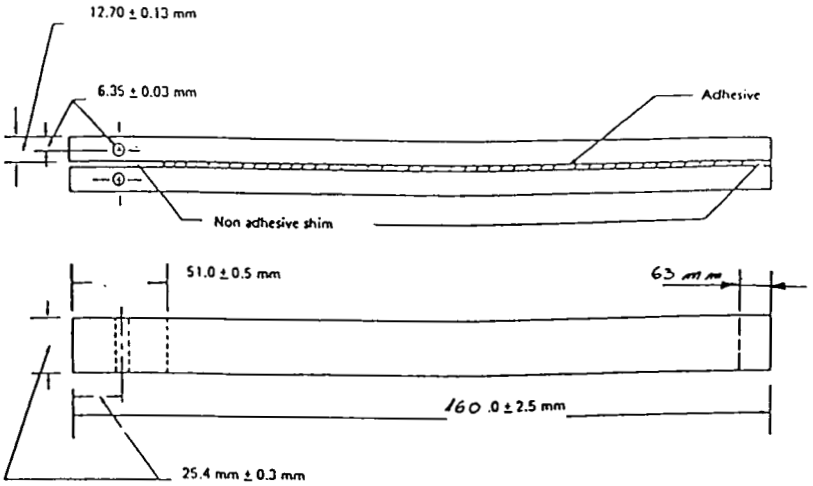

FIGURE A1: DCB Test Specimen

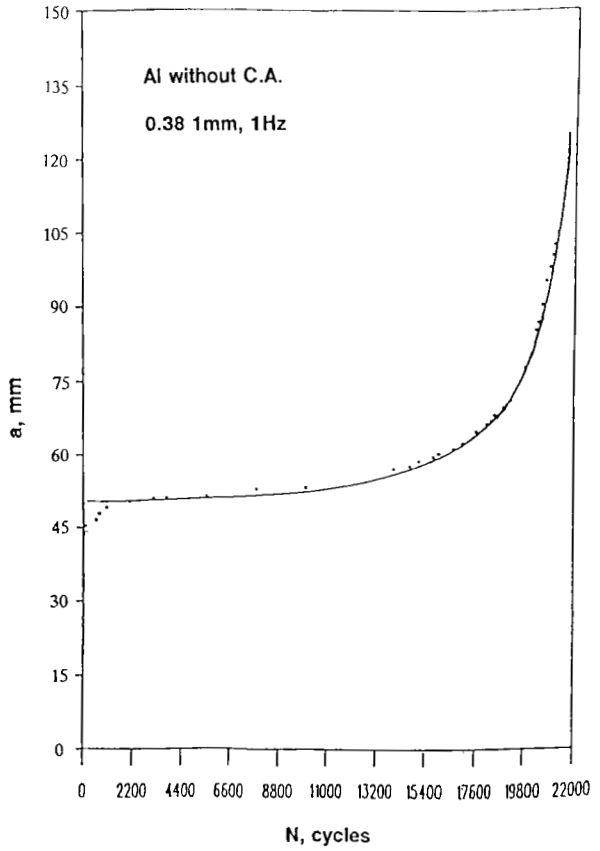

FIGURE A2: Typical debond growth values as a function of number of fatigue cycles for aluminum joints with a room temperature curing epoxy system.