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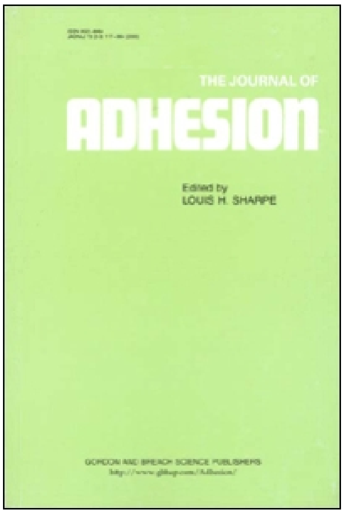

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The Use of the Short Beam Shear Test Method on Metal-to-Metal Adhesive Bonds

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Unless a large amount of degradation has occurred in the interfacial region of a metal-to-metal adhesive bond, the T-peel and wedge opening test usually provide only mode I information on the bulk adhesive. The single-lap shear test shows primarily mixed-mode failure, however, the thickness of the adherend must be carefully considered in order not to have a large amount of mode I failure. When these three tests failed consistently to find defects that were incorporated into the interfacial region of test specimens we decided to use the short beam test geometry for our adhesive bond program. This initial study, utilizing the short beam shear test geometry, has shown that several pieces of information may be obtained from the experimental data. The initial portion of the test curve appears quite linear and the slope of the curve seems directly related to the bulk properties of the adhesive. The portion of the curve where a definitive failure occurs appears directly related to interfacial failure of the bonded joint. Because this is the initial study using this test geometry the data obtained was used only in a qualitative manner in screening the various tests employed.

INTRODUCTION

Historically the effects of stress and environment on the overall strength of metal-to-metal adhesive bonds has been determined primarily by three mechanical tests; (1) T-peel, (2) wedge opening, or (3) lap shear. Attempts to correlate data from accelerated laboratory experiments with actual service life, using these mechanical tests, have met with only marginal success.[1–4] Actual in-service failures of adhesive bonded structures are reported to occur in the region of the adhesive–oxide interface. A recent study using surface instrumentation techniques[5] reports failure sites also occurring at the oxide–metal interface. Therefore, the interfacial region of the adhesive–oxide–metal should be of primary concern in laboratory studies of adhesive bond line failure. Since the geometry of the mechanical tests mentioned above provide primarily mode I type information it is difficult to obtain information in the interfacial region of interest when only minor defects in that region are being studied. Kaeble previously pointed out this problem with test geometry when he stated,[6] "A test for adhesion must, by definition, result in interfacial failure. Tests of adhesive joints are not adhesion tests when cohesive failure is obtained." This was the problem we faced with our adhesive bond program using the standard mechanical tests. When minor defects were introduced into the bond line our major failure mode was cohesive in nature.

Some of these programs were concerned with the potential problem of bond line corrosion. When dealing with copper-containing aluminum alloys the aspect of corrosion has to be considered. In order to study the possible subtle effects of surface preparation and corrosion films on the strength of the adhesive–oxide and/or oxide–metal interfaces, our testing program was directed to a test geometry that would be simple to fabricate, and would load the bond line with the proper stress to generate interfacial failure even in good bonds.

We chose the short beam shear test for our study and the purpose of this paper is to illustrate the general aspects of this test method on metal-to-metal adhesive bonds. This study, to our knowledge, is the initial use of this test method on metal sandwich structures.

EXPERIMENTAL

1. Aluminum alloy specimens

Rectangular specimens were cut from a sheet of 7075-T6 bare aluminum alloy. Each specimen was 10 × 15 × 0.35 cm. All of the specimens were degreased with an acetone wipe, then alkaline etched with 0.1 N NaOH for three minutes at room temperature. Specimens were deoxidized with a solution of 5:1 HNO_3—HF for two minutes at room temperature, then desmutted with 50% HNO_3 for thirty seconds.

Duplicate panels were then anodized in a 1.0 M H_3PO_4 bath according to the conditions described in Table I. Each pair was then bonded with FM 123-2† adhesive (in tape form), cured at 250°F and 25 psi, without the aid of a primer. Specimens 3.8 × 1.3 × 0.7 cm were cut from the bonded panels. Duplicate specimens of each surface preparation were subjected to environmental conditions described in Table II.

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† American Cyanamid Co., Stamford, CT, U.S.A.

TABLE I

Anodization conditions for 1.0 M H_3PO_4

Surface preparation	Voltage	Time in bath	Average oxide thickness
1	10	0.5 min	225Å
2	10	2.0	1000
3	10	10.0	3200
4	10	16.0	3600
5	40	2.0	1200
6	40	5.0	3000

TABLE II

Type of exposure for test specimens

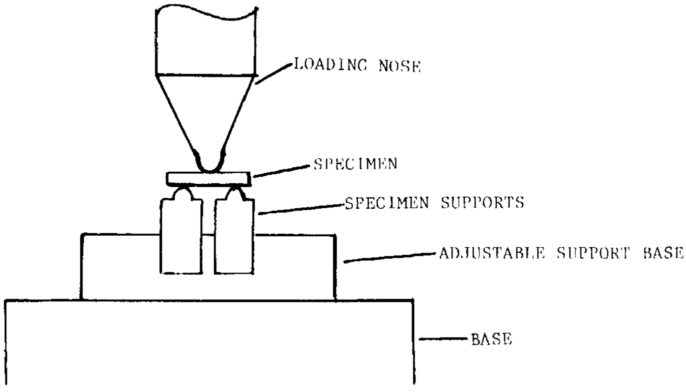
Test	
A	specimens cured then tested within 24 hours
B	specimens cured then post cured at 220°F for 8 hours
C	specimens cured then stored in desiccator under ambient conditions for 90 days
D	specimens from Test C subjected to dry heat, 100°C, for 63 hours
E	specimens from Test C subjected to SO_2 environment at R.T. for 2160 hours
F	specimens from Test C subjected to SO_2 environment at 100°C for 72 hours
G	specimens from Test C stressed to 1000 lb for two minutes then subjected to 100% R.H. and 100°C for 8 hours

2. Mechanical test

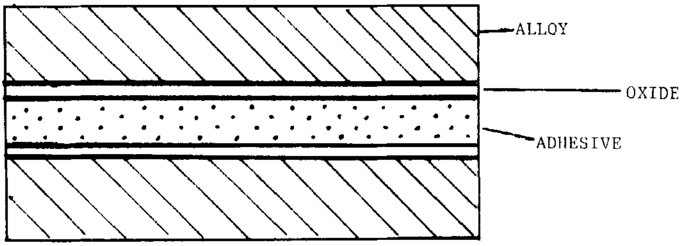
The data was obtained from each specimen using an Instron test machine. The short beam shear test procedure (similar to ASTM D-790) aligns the specimen so that the resultant of the applied load is perpendicular to the adhesive bond line (Figure 1) in a configuration often used to determine flexural properties of materials. The support noses were adjusted to a span 4 times the average specimen thickness. The test recording was obtained by loading each specimen at a crosshead speed of 0.02 inch per minute until interfacial failure occurred. All tests were stopped after 6 minutes. All tests were performed at room temperature.

ANALYSIS OF DATA

The short beam shear test is one of the most widely used test methods for evaluation of the shearing strength of composite materials and flexural strength of polymers. Several assumptions are made in applying beam theory to this test, one of which is that the shear stress S is distributed across the



SHORT BEAM SHEAR TESTING FIXTURE



SPECIMEN

FIGURE 1 Illustration of short beam shear test geometry.

transverse face of the specimen with a maximum at the center according to the following equation

$$S = 0.75(L/wt) \tag{1}$$

where L is the failure load applied at the center of the beam, w is the width of the beam, and t is the thickness. The failure load (L) is obtained from the test record, and the value of S generated from the equation is assumed to represent the shearing strength of the composite in pounds per sq. inch.

We have utilized this same test in order to study the sandwich structure of metal-to-metal adhesive bonds. We believe this is the first attempt to apply this geometry to metal-sandwich adhesive joints. Because of the complexities of the stress and strain distribution across the bond line this test procedure was only used as a screening method to determine if we could differentiate between the interfacial properties of various surface preparations that had been subjected to changing temperatures and/or environments.

Figure 2 represents the test record of a completely nonbonded specimen.

This specimen was prepared by physically laying a film of FM 123-2 adhesive between two adherends of 7075-T6 aluminum. All of the figures reproduced in this paper were generated by a Tektronix data curve processor with points taken from the Instron generated curves. Some of the reproduced figures appear to be nonlinear; however, this is due only to the small number of data points taken from the actual curves. The initial fifteen seconds of the actual recorded data is not reproduced because it represents instrument take-up. The linear portion of this figure represents the resistance that the two adherends, plus the nonbonded adhesive film, offer the loading nose of the Instron machine. As this resistance or stiffness is overcome the next portion of the curve shows a more gradual take up of force. The load value at this point represents a slight metal deformation mixed with the energy required to overcome the friction of the adhesive tape opposing the slide of the two opposite faces of the adherends. The displacement of the specimen from the applied load is plotted as a function of time. The load value obtained from the curve after one minute of applied force was chosen to represent the stiffness of

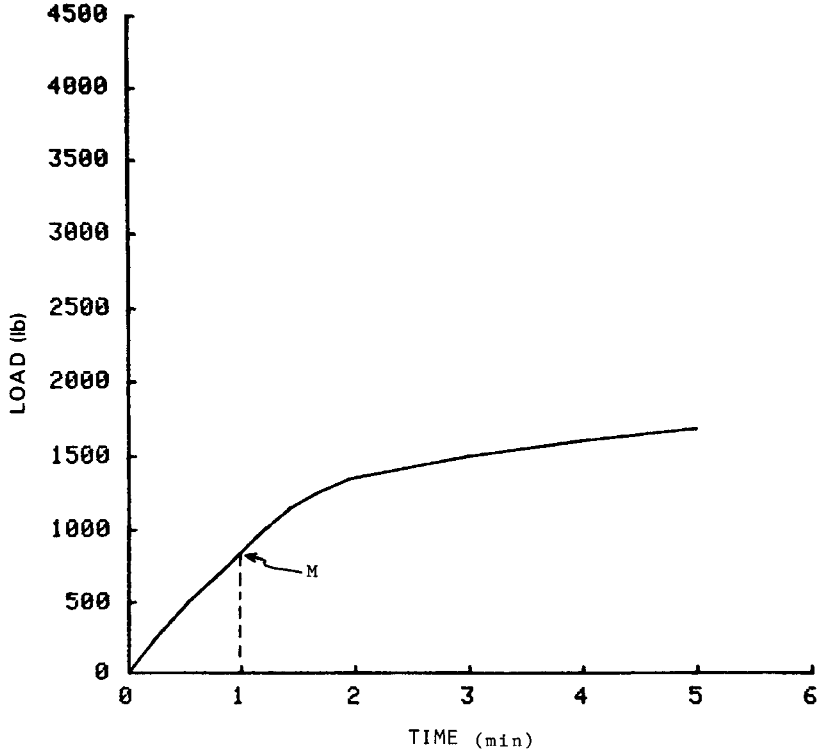


FIGURE 2 Load-displacement curve for non-bonded specimen.

the specimen. This value would represent the slope of the curve and is designated as M. The load value supplied by the curve after one minute (Figure 2) represents our data point for a completely nonbonded specimen and is recorded as M_r.

Figure 3 represents the data obtained from a specimen where the adhesive film has been fully cured and has not been subjected to any environmental tests. The load value is obtained from this curve in the same manner as for Figure 2 and is designated M_x for an experimental data point. From the linear portion of our reference curve we obtain M_r and from the experimental curve we obtain M_x. From these data points we can calculate what we have chosen to call the bond line stiffness efficiency (BLS).

$$\text{BLS} = \frac{M_x}{M_r} \tag{2}$$

The upper portion of the curve (Figure 3) obtained from the bonded specimen is distinguished by a definitive break in the test record. The load

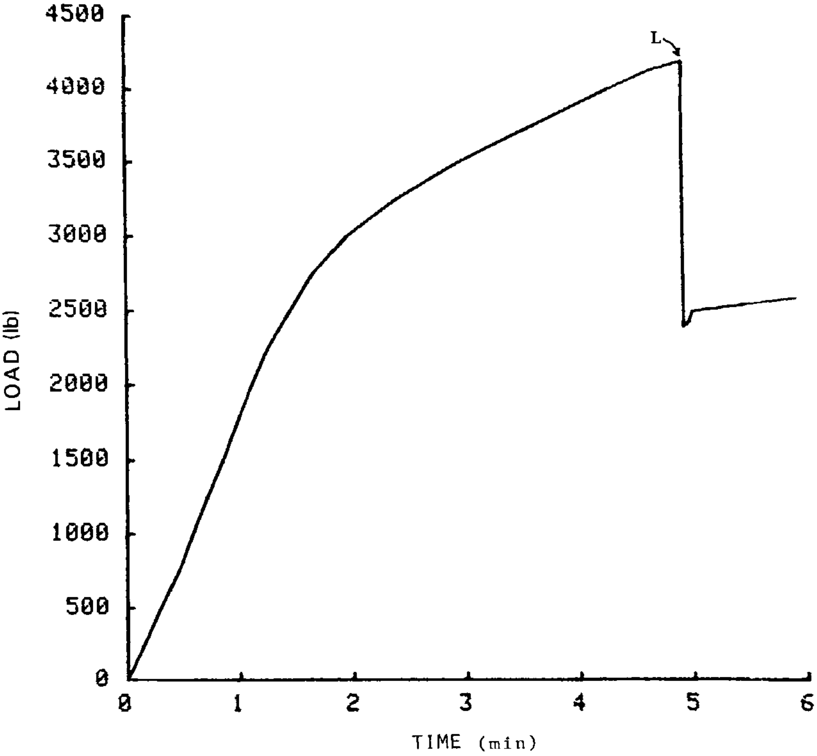


FIGURE 3 Load-displacement curve for bonded and curved specimen.

value obtained from this point (*L*) is taken as the yield strength of the bond line (YSI). All of the YSI values were obtained directly in pounds since the dimensions of the specimens used in this study were the same in each case. After the break in the upper curve another curve is generated where a gradual increase in load value is observed.

Figure 4 is representative of a bonded specimen that has been subjected to environmental tests. The YSI break in the curve is still present but at a much lower value. The last type of curve recorded from this study is shown in Figure 5. This represents a bonded specimen that has no indication of a value for *L* and has lost all bond line strength due to severe environmental testing.

DISCUSSION

We believe that two types of definitive information can be obtained from the test records generated by this study. One piece of information may be obtained

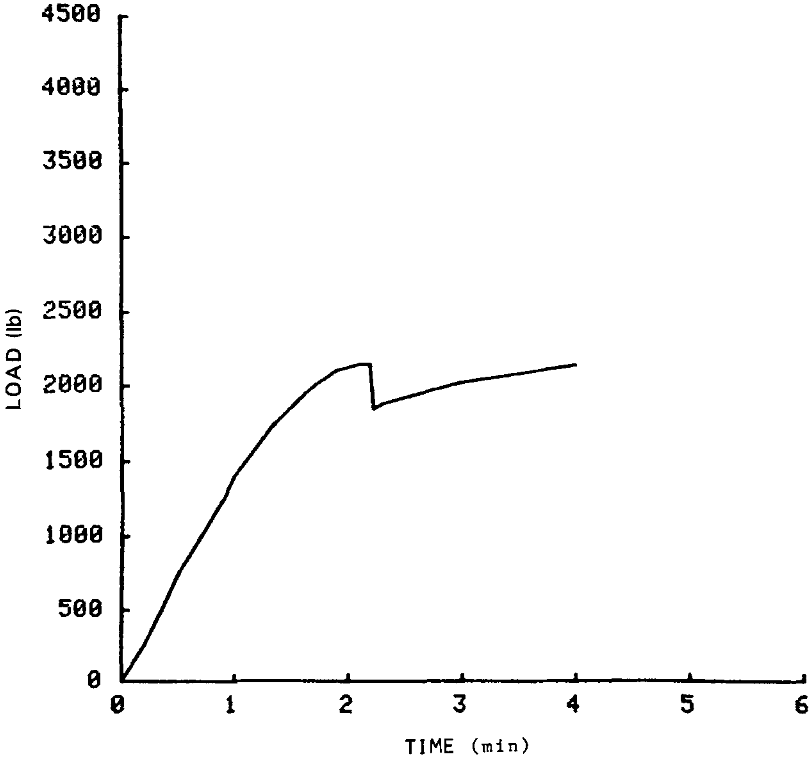


FIGURE 4 Load-displacement curve for test specimen with low strength interface.

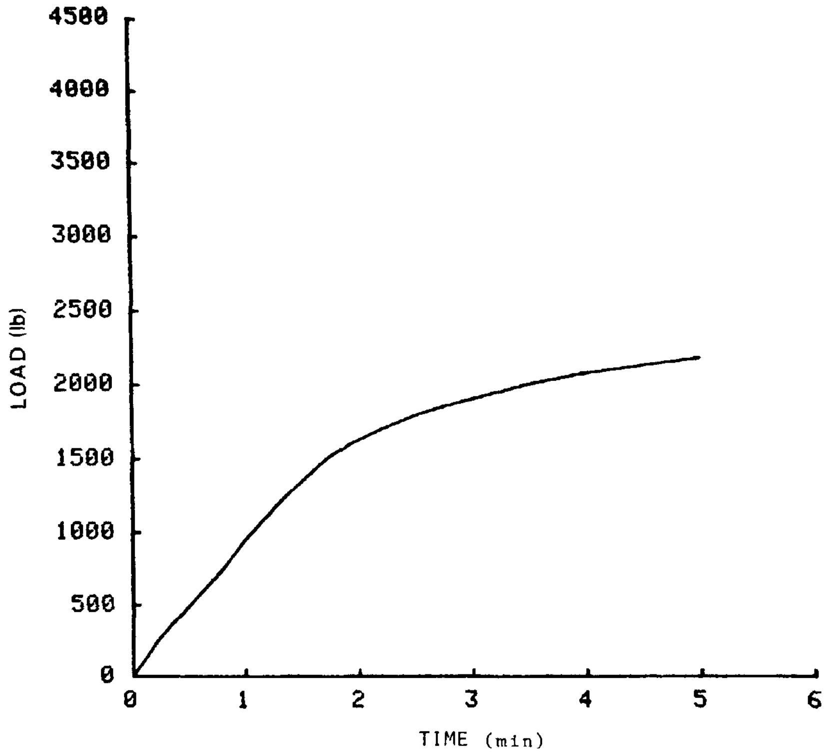


FIGURE 5 Load-displacement curve for test specimen with adhesively failed interface.

from the initial linear portion of the curve and is designated as BLS in Tables III and IV. The second piece of information may be obtained from each curve where a definitive break in the record occurs. These values are notated as the yield strength of the bond line (YSI) in Tables III and IV.

The fact that the stress generated by this test geometry is driven primarily to the interfacial region can be observed by a visual inspection of the failed specimens. Figure 6 shows the surfaces of three specimens that were fully opened after mechanical testing. The test records shown in Figures 3, 4, and 5 were obtained from the specimens shown in Figures 6a, 6b, and 6c respectively. Figure 6a shows the surface of a failed specimen considered to be a good bond. Two other specimens were taken from the same panel from which the specimen shown in 6a was obtained. These two specimens were cut to the appropriate size for a wedge opening test and a lap shear test. Both specimens were taken to failure without being subjected to any environmental tests. Both tests showed the specimens to fail 100% cohesively as observed by visual inspection. The short beam shear test specimen shown in Figure 6a definitely

TABLE III

Data from control specimens

Surface prep	Controls A		B		C	
	BLS	YSI (lb)	BLS	YSI (lb)	BLS	YSI (lb)
1	1.53	2825	2.06	3050	1.84	2758
2	1.59	3012	2.15	3552	1.80	2793
3	1.55	3200	2.12	4120	1.86	3222
4	1.58	3175	2.12	4202	1.92	3202
5	1.53	3025	2.01	3725	1.84	2900
6	1.59	2875	1.98	3650	1.85	2940

TABLE IV

Data from environmental test specimens

Surface prep	Environment D		E		F		G	
	BLS	YSI (lb)	BLS	YSI (lb)	BLS	YSI (lb)	BLS	YSI (lb)
1	1.53	3100	1.76	2520	—	NIF†	1.62	2455
2	1.62	2960	1.82	2760	1.76	2475	1.70	2590
3	1.60	2780	1.81	2945	1.70	2820	1.73	3045
4	1.65	3210	1.82	3145	1.70	2900	1.53	2500
5	1.59	3000	1.88	2910	1.70	2515	1.56	2640
6	—	NIF†	1.82	2400	1.69	2690	1.65	2847

† No definitive interface failure (see Figure 5).

has a reasonable amount of adhesive failure. All of the specimens designed to be good bonds had a definite amount of adhesive failure when failed by the short beam shear test. For this reason we believe this test geometry allows more stress to be applied to the interfacial area of the bond line. Figure 6b shows the surface of the specimen that has a low value at failure. This specimen has considerably more interfacial failure than the specimen in 6a. It had been exposed to environment testing. Figure 6c shows the surface of a specimen having a very large amount of adhesive failure, so much so that point L was not obtainable from the test record.

The reported test data were generated from two groups of specimens. Table III reports the data from Tests A, B, and C. Each group was cured under various conditions but was not subjected to environmental tests. The yield strength of the bond lines (YSI) of Tests A and C are quite comparable; however, the BLS values vary about 19% on the average for the different cycles. The post anneal cure (Test B) shows an increase in both the YSI and BLS

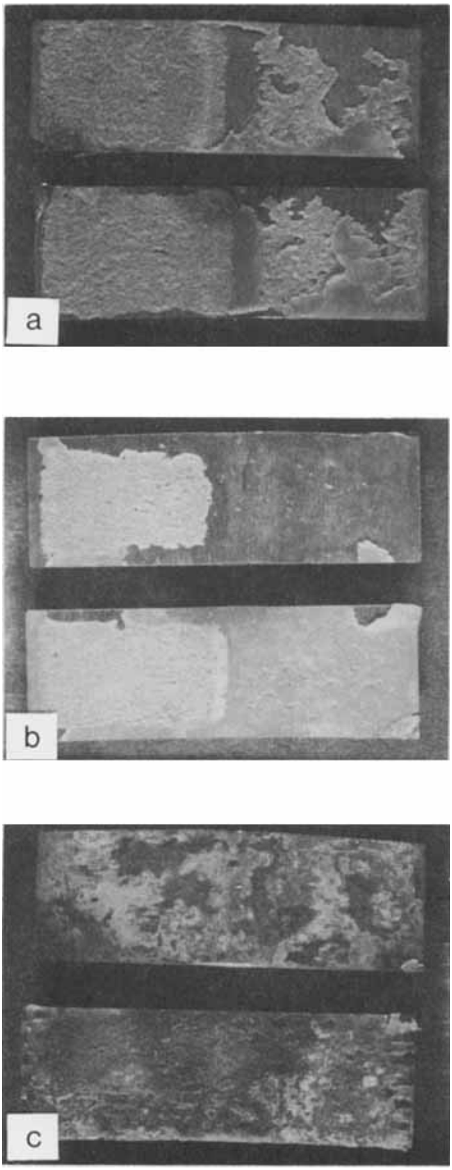


FIGURE 6 Low magnification photographs of test specimens (a) specimen from Figure 3, (b) specimen from Figure 4, (c) specimen from Figure 5.

values when compared to the other control specimens. The average of the YSI values for groups A and C is taken as the control reference. These data (including the data spread) are shown in Figure 7 plotted against the surface preparation (shown in Table I). The data population is small for these groups but the trend shows the 10 volt–10 minute, and 10 volt–16 minute anodizations provide the higher strength specimens.

Data from the specimens subjected to the environmental tests are shown in Table IV. The data from the more severe tests (E, F, and G) were averaged and plotted against surface preparation (Figure 8). The data population is small; however, a definite trend is indicated. The strength values overall are lower than the control specimens but the curve reproduces the trend shown by the control 10 volt–10 minute, and 10 volt–16 minute surfaces.

The average BLS from Test C was taken as our control value because all of the experimental specimens were subjected to the conditions of Test C for standardization purposes. Before any data were obtained from the experimental specimens it was presumed that the ratio of the slopes (M_x/M_r), which

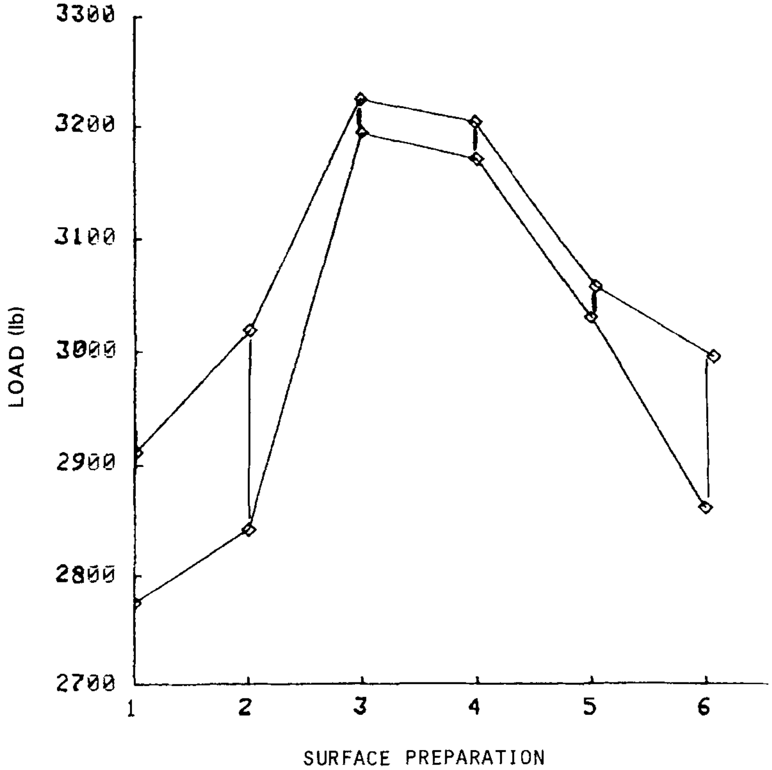


FIGURE 7 Spread of load values for control specimens.

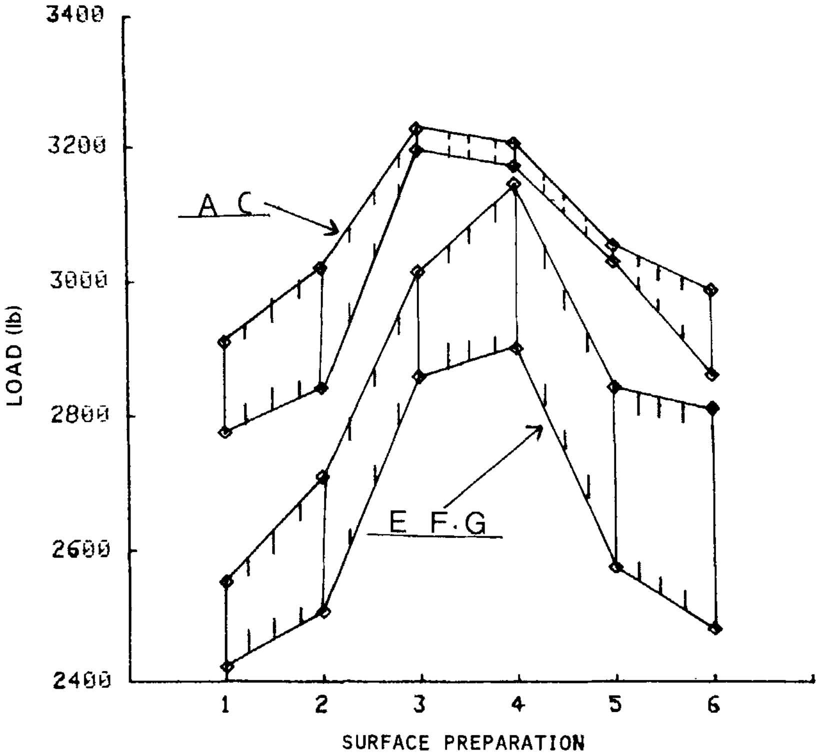


FIGURE 8 Comparison of the spread of load values for control and test specimens.

we have designated BLS, would follow the same type of plot as shown for the bond line strength values. This is not the case since the BLS values obtained for each specific test are quite constant, with test G being the one exception. The fact that the BLS values remain constant within each individual test environment but change from test to test seems to indicate that this ratio is showing the effect of the environment on the bond line, while at the same time it is not that sensitive to the varying surface preparations within each test. Therefore, we propose that the BLS number represents a figure of merit for the bulk adhesive and its response to each particular environment. Some added proof for this proposal can be shown from some data obtained previously[7] from a wedge-opening study using SO_2 as an environment. The specimens were exposed to the environment for 168 hours at room temperature. Zero crack growth was recorded and no adhesive failures were observed. Since the wedge opening geometry generates mode I type failure a zero crack growth indicates that the SO_2 environment had very little effect on the bulk adhesive.

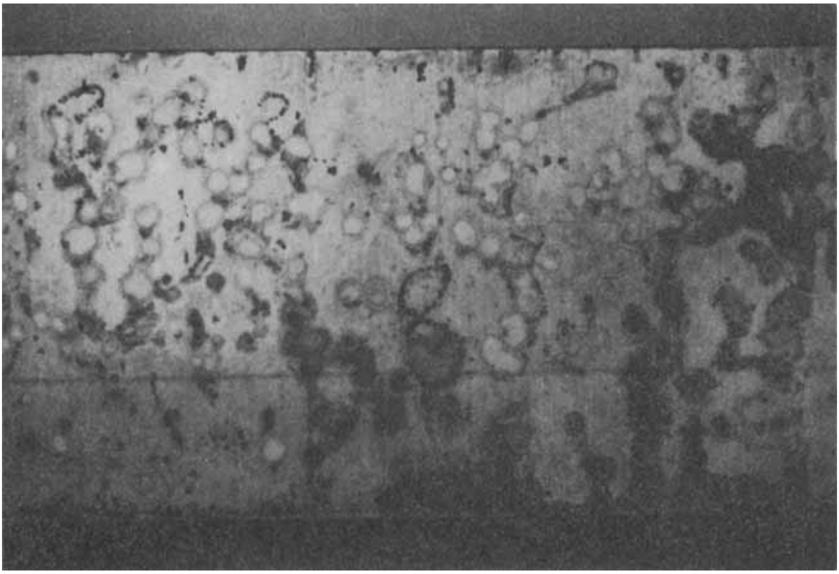


FIGURE 9 Phosphoric acid anodized surface subjected to SO_2 environment.

This same environment is quite harsh on a phosphoric acid anodized 7075 aluminum alloy surface as can be seen in Figure 9.

The same environment (Tests E and F) was used in the present study and the specimens were evaluated using the short beam shear test. Using the average value of the BLS data from Test C as our control we find a 1.7% change from the average BLS value from Test E. At the same time the average YSI value from Test E degrades by 6.4% when compared to the YSI value for Test C. We believe that these two sets of data strongly indicate the BLS data are indicative of bulk adhesive properties and the YSI data are indicative of the bond line interfacial properties. Comparing Test F to C (where F has both temperature and SO_2) we find a change of 7.5% in BLS value while the YSI value changes 9.7%. This environment is detrimental to both the bulk adhesive and the bond line interface. Obviously as both the bulk adhesive and the bond line degrade the two pieces of data will not remain independent of each other.

CONCLUSION

The weak link in most metal-to-metal adhesively bonded structures that have been subjected to a temperature-environment regime is the adhesive–oxide–metal interfacial region. When these structures are subjected to a mechanical test the test geometry must provide a stress to probe the interfacial region. The data obtained from this initial study using the short beam shear test indicate that this test is capable of following the subtle changes that may occur in the interfacial region. A second piece of information is also available from this test and it appears to be directly related to the bulk properties of the adhesive.

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