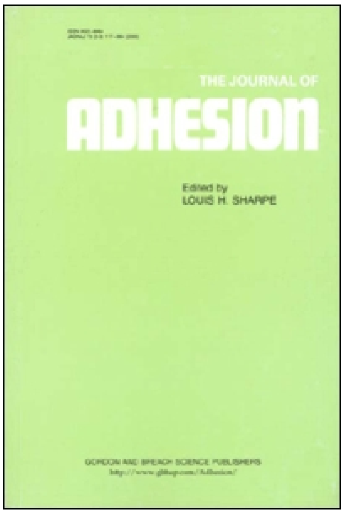

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Torsional Test Method for Adhesive Joints

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A modified tubular butt assembly for testing adhesive joints was found to yield greater precision and sensitivity than previous methods. Effects due to modifying the surface pretreatment of aluminum-epoxy joints could be identified and analyzed statistically by the Wilcoxon–Mann–Whitney test. The use of a very short tube integrally mounted on a solid rod minimizes axial stress components. The joints are easily handled and tested, and due to their greater reproducibility, fewer tests are necessary.

INTRODUCTION

Although bonding of materials is today an important industrial practice the methods of testing adhesive joints lead only to general conclusions on the effects of adhesive-adherend interaction and adherend preparation on bond strength. This has been primarily due to joint design limitations which do not separate effects such as shear, peeling, and substrate deformation. In general, the preparation of the substrate is held constant, and the adhesive mixture varied to achieve maximum bond strength. In the case of differing surface treatment, only gross effects have been identified. In this paper, not only is good reproducibility found, but subtle effects due to varying the surface treatment can also be detected.

A torsional test method used by Lin and Bell[1] subjects an epoxy–aluminum bond to almost pure shear stress. The adhesive joint, shown in Figure 1, is a modified form of a tubular butt. The use of a very short tube integrally mounted on a solid rod minimizes axial stress components in the joint that are due to distortion of the tube. The joints are easily handled and tested, and due to their greater reproducibility, fewer tests are necessary.

The purpose of the present work was to verify the reproducibility found for this method for a particular surface treatment by earlier experiments,[1] and also to find out if this testing procedure can detect more subtle effects due to changes in surface preparation. Previous work on varying surface pretreatment using lap-shear joints is limited in that there is too much scatter in the data, which can lead only to general conclusions on bond strength.

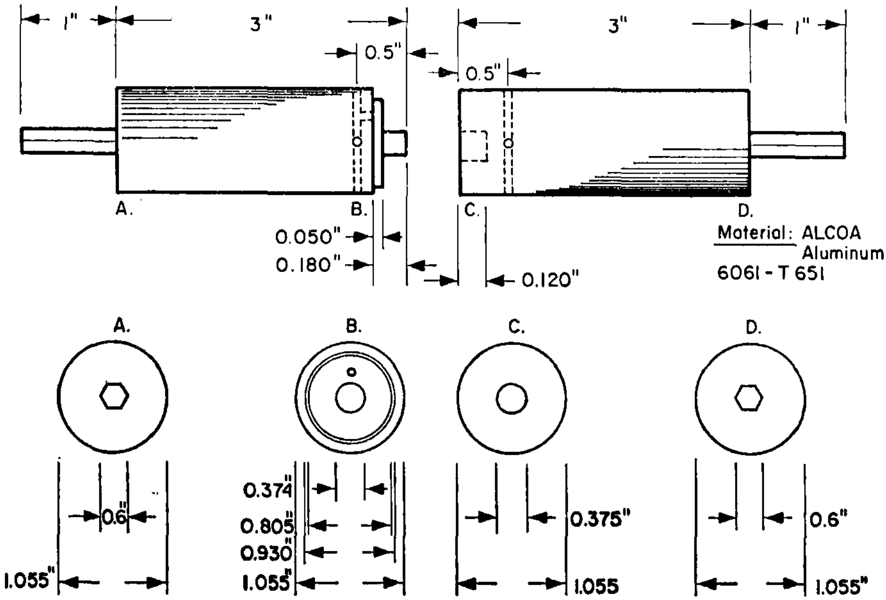

FIGURE 1 Modified tubular butt joint.

EXPERIMENTAL

The details of the adhesive joint and test procedure have been previously described.[1] In order to achieve good reproducibility, it was found that the dimensions of the annular ring (Figure 1) must be controlled to a tolerance of ± 0.001 inch. Care was taken during polishing to prevent rounding of corners and to assure that the tolerances were not exceeded due to removal of metal. The thickness of the adhesive ring should be within ± 0.0005 inch and the amount of resin must be weighed to ± 1.0 mg. The amount of resin needed to occupy a ring joint with the dimensions 0.805 inch inside diameter, 0.930 inch outer diameter and 0.010 inch thickness was found to be 44 mg.

Preparation of aluminum surface

The joints, prior to each use, were machined on a lathe to the proper specifications. After resurfacing, the joints were buffed on a polishing wheel with

5.0 μ, then 0.3 μ alumina suspended in distilled water, followed by washing with distilled water, then acetone. After air drying, the joints were degreased in refluxing trichloroethylene for approximately $\frac{1}{2}$ hour. All joints were treated in this manner. When the hot joints were removed from the degreaser and had cooled to 60 to 65°C, they were immersed in a 60 to 65°C solution of chromic acid for 10 minutes. The chromic acid consisted of a 30 : 10 : 1 by weight solution of distilled water, sulfuric acid, and anhydrous potassium dichromate. A 45 : 15 : 1 by weight solution of distilled water, sulfuric acid and chromium trioxide was also used to generate chromic acid. After etching, the joints were either rinsed with, or rinsed with and immersed in, 10°C tap water. At 10°C the aluminum surface undergoes reaction with water very slowly.[2] The joints were air dried at room temperature for 1 hour at about 55% relative humidity before resin was applied. The resin was applied and cured as described by Lin and Bell[1] to keep the polymer structure constant, except that the curing schedule was modified to 30 minutes at room temperature, 80°C for 1 hour, then 150°C for $2\frac{1}{2}$ hours. The cured specimens were broken at 0.05 inch/min. cross-head speed (0.4%/min. strain rate) with the force to break recorded in pounds. The torsional testing was conducted on an Instron® Universal Testing Machine, Model TM–S. The device used to apply torque to the joint is manufactured by Instron® as part of the Instralab® series.

Applied torque, Mt, is related to the maximum shear stress, τ_{max}, as shown below:

$$\tau_{max} = \frac{16 . Mt . D_0}{\pi(D_0^4 - D_i^4)}$$

where D_0 and D_i are the outer and inner diameters of the annular ring, respectively. The angle of twist is given by

$$\theta = \frac{32Mt . L}{\pi(D_0^4 - D_i^4)G}$$

where G is the modulus of rigidity of the resin and L is the resin thickness.

The Wilcoxon–Mann–Whitney test,[3] a simple statistical analysis for a small population of data, was applied to the experimental data. The test is used to determine whether or not two sets of data are significantly different. Each value in the first set is assigned a symbol $x_1, x_2, \ldots, x_n$, and each value in the second set is assigned a symbol $y_1, y_2, \ldots, y_n$. The test compares every x-value with every y-value, counting the total number of times each x-value surpasses y-values (U_x), or the total number of times each y-value surpasses x-values (U_y). U_x and U_y can be used to test the null hypothesis (H_0) that there is no significant difference between two series of joint strengths. If the null hypothesis is true, x- and y-values can be expected to be similar, and U_x and U_y will also be similar. If y-values tend to be greater than x-values, then U_x should be small and U_y large. This suggests rejection of the null hypothesis (H_0), i.e., the two series of joints are different, when the smaller one of U_x and U_y is sufficiently small.

Assuming two independent sets of values, an x-set consisting of m number of values, and a y-set consisting of n number of values, $U_x + U_y = mn$. Let U be equal to the smaller of U_x and U_y. The Wilcoxon–Mann–Whitney test rejects the hypothesis that the x- and y-values are equivalent in a two-sided test if U is smaller than a value d, where d is listed[4] for particular m and n. A significance level is obtained,[4] for a given value of U and d, for the rejection of the null hypothesis for a two-sided test and a one-sided test. The two-sided test is used when the two series appear similar. In the one-sided test, U_x is used if x-values appear to be smaller than y-values, or U_y is used if y-values appear to be smaller than x-values. The two-sided test can determine if two series are similar, and the one-sided test if two are different. A sample calculation is shown in Appendix 1.

RESULTS AND DISCUSSION

The reproducibility of chromic acid etched joints

The data for bond strengths of joints prepared as previously described and etched in a 30 : 10 : 1 by weight solution of distilled water, sulfuric acid,

TABLE 1

Potassium dichromate–sulfuric acid etched aluminum–epoxy bond strength

Force to break in lbs.		
Series 1	Series 2	Series 3
293	300	290
301	295	303
295	297	301
296	300	294
		296
296 average	298 average	297 average

and potassium dichromate are shown in Table 1. After etching at 60 to 65°C for 10 minutes, the joints were washed in cold tap water and allowed to air dry. Three series of five joints each were made on three different days, and the data for joints that were found to have a gap in the annular ring of resin are not reported. The joints in series 1, 2, and 3 exhibited mixed adhesive/cohesive failure. The ring of cured resin was largely intact with the bulk of the resin adhering to one side of the joint. However, a small amount of the resin was present on the opposite face.

Joints in Series 1, 2 and 3 (Table 1) were compared with the following results, using the Wilcoxon–Mann–Whitney test, assuming a null hypothesis (H_0).

Series 1 *vs.* Series 3. Accept H_0 (two-sided test), where $U > d = 4$, for $m = 4, n = 5$.

Series 1 *vs.* Series 2. Accept H_0 (two-sided test), where $U > d = 3$, for $m = n = 4$.

Series 2 *vs.* Series 3. Accept H_0 (two-sided test), where $U > d = 4$, for $m = 4, n = 5$.

The comparisons of Series 1, 2 and 3 indicate that all three series may be treated as one group of data, as there is no significant difference between the three. The reproducibility of the test is qualitatively apparent by a comparison of the average value for each of the three series.

The effect of tap water immersion pretreatment on unetched aluminum-epoxy joint strength

The effect of immersing aluminum in cold tap water prior to bonding with epoxy resin is demonstrated by the data presented in Table 2. The joints were

TABLE 2

The effect of water immersion pretreatment time on unetched aluminum–epoxy bond strength

Force to break in lbs.			
Series 4 Quick wash	Series 5 ½ hour H_2O exposure	Series 6 2 hour H_2O exposure	Series 7 6 hour H_2O exposure
270	286	309	323
274	283	301	325
276	290	312	319
278	280	307	315
276	280	292	319
274	273	300	312
275	—	300	307
274	282 average	—	—
—		307 average	317 average
275 average			

prepared as previously described except that the joints were polished with 5 μ alumina suspended in distilled water, and not acid etched. After degreasing, the joints were allowed to cool to room temperature, then were immersed in 10°C tap water for varying times. The joints were then air dried for 1 hour before the resin was applied. The joints in Series 4, 5, 6 and 7 exhibited the full range of adhesive failure. Series 4 and 5 showed primarily adhesive failure with the resin ring remaining intact on either face of the joint. The joints in Series 6 showed mixed adhesive/cohesive failure. Series 7 samples failed primarily cohesively, as the annular ring of resin was shattered. However, more resin was observed on one face of the joint than on the opposing one, indicating that the failure was not entirely cohesive. No preference was shown for either joint face.

The null hypothesis (H_0) was rejected for each series presented in Table 2 in a one-sided test to determine which set of bond strengths was the strongest. The results were as follows:

Series 4 *vs.* Series 5. Reject H_0 at significance level 0.021 (one-sided test), where $U_4 < d = 9$, for $m = 6, n = 8$, $U_4 < U_5$.

Series 5 *vs.* Series 6. Reject H_0 at significance level 0.004 (one-sided test), where $U_5 < d = 4$, for $m = 6, n = 7$, $U_5 < U_6$.

Series 6 *vs.* Series 7. Reject H_0 at significance level 0.004 (one-sided test), where $U_6 < d = 5$, for $m = n = 7$, $U_6 < U_7$. Series 4, 5, 6 and 7 each come from different populations, and bond strength increases in the order of Series 4, 5, 6, 7. For these unetched joints, the strength increases with time of immersion in 10°C tap water for times up to 6 hours.

The effect of resin thickness on bond strength

Etched aluminum joints were prepared as described, except that the joints were chemically treated in a 45 : 15 : 1 by weight solution of distilled water, sulfuric acid, and chromium trioxide. After etching, the joints were washed in 10°C tap water and allowed to air dry. Table 3 shows the data on the effect of resin thickness on bond strength. Series 8 consists of joints machined such that the thickness of the resin ring was 0.008 inch, and Series 9 are standard joints (0.010 inch thick). The surface treatment of the aluminum

TABLE 3

The effect of resin ring thickness on bond strength

Force to break in lbs.	
Series 8 0.008 inch resin thickness	Series 9 Standard thickness (0.010 inch resin thickness)
333	295
322	297
348	295
320	303
—	297
326 average	295
	—
	294 average

and the amount of applied resin were held constant for both series. The joints in Series 8 and 9 showed mixed adhesive/cohesive failure. The statistical analysis is shown below:

Series 8 *vs.* Series 9. Reject H_0 at significance level 0.005 (one-sided test), where $U_9 < d = 1$, for $m = 4$, $n = 6$. The effect of changing resin ring thickness without changing the amount of resin applied will be to yield a significant change in sample strength as compared to standard joints as shown by the comparison of Series 8 with Series 9.

CONCLUSION

The torsional test method developed by Lin and Bell[1] can yield more useful information on bond strengths than current methods, such as lap joints, circular butt, or tubular butt joints. Table 4 shows the standard deviation

TABLE 4

A comparison of various joint test methods

Type of Joint	Coefficient of variation[a]	Worker
Epoxy-aluminum lap joints	14	R. F. Wegman[5]
Epoxy-steel: circular butts in simple shear	11	Bryant and Dukes[6]
Epoxy-steel: circular butts in torsion	4	Bryant and Dukes[6]
Epoxy-steel: tubular butts in torsion	5	Bryant and Dukes[6]
Epoxy-aluminum: modified tubular butts in torsion (no acid-dichromate treatment)	3	Lin and Bell[1]
Epoxy-aluminum: modified tubular butts in torsion (acid-dichromate treated)	1	Lin and Bell[1]
Epoxy-aluminum: modified tubular butts in torsion		This work
Series 1	1.1	
Series 2	0.8	
Series 3	1.8	
Series 4	0.8	
Series 5	2.1	
Series 6	2.6	
Series 7	1.9	
Series 8	3.9[b]	
Series 9	1.4	

[a] $\frac{\text{Standard deviation for the series}}{\text{Average value for the series}} \times 100.$

[b] Not simple shear.

of each series presented in this paper and a comparison with other test methods. The modified tubular butt method exhibits much less variance than other test methods, therefore simpler statistical methods can be used to analyze the results.

The testing of joints with identical surface pretreatment will give identical results, as shown by the bond strengths of the three series of potassium dichromate–sulfuric acid etched joints done on different days. If the surface treatment is made dependent on one variable, the test method can give useful data illustrating the effect. The bond strength of unetched aluminum pretreated by immersion in cold tap water for varying times is shown graphed in Figure 2. The mean value for a particular series is used as a locus for the line. In this case, the bond strength increases as a function of tap water immersion time.

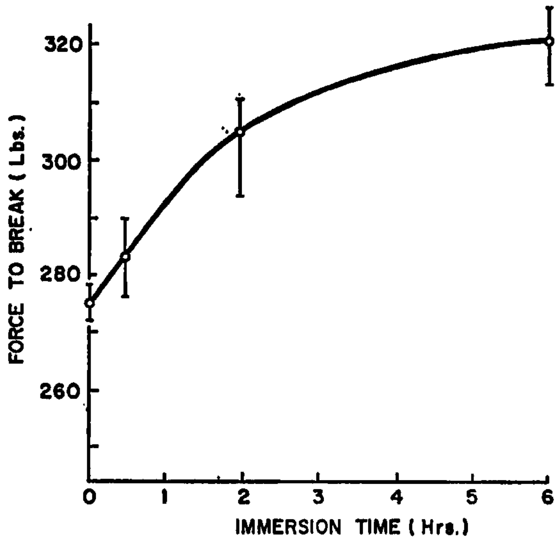

FIGURE 2 Effect of tap water immersion pretreatment time on bond strength. Range of experimental values indicated by vertical bars.

APPENDIX 1

SAMPLE CALCULATION TO DETERMINE A SIGNIFICANCE LEVEL USING THE WILCOXON-MANN-WHITNEY TEST

x-values	y-values
x_1 270	y_1 286
x_2 274	y_2 283

x_3 276	y_3 290
x_4 278	y_4 280
x_5 276	y_5 280
x_6 274	y_6 273
x_7 275	
x_8 274	

x_1 surpasses 0 y-values	$Ux_1 = 0$	y_1 surpasses all x-values	$Uy_1 = 8$
x_2 surpasses y_6	$Ux_2 = 1$	y_2 surpasses all x-values	$Uy_2 = 8$
x_3 surpasses y_6	$Ux_3 = 1$	y_3 surpasses all x-values	$Uy_3 = 8$
x_4 surpasses y_6	$Ux_4 = 1$	y_4 surpasses all x-values	$Uy_4 = 8$
x_5 surpasses y_6	$Ux_5 = 1$	y_5 surpasses all x-values	$Uy_5 = 8$
x_6 surpasses y_6	$Ux_6 = 1$	y_6 surpasses x_1	$Uy_6 = 1$
x_7 surpasses y_6	$Ux_7 = 1$		
x_8 surpasses y_6	$Ux_8 = 1$		$\Sigma Uy_i = U_y = 41$

$$\Sigma Ux_i = U_x = 7$$

$m = 8, n = 6$ equivalent to $n = 8, m = 6$ since assignment of x and y to the two populations was arbitrary.

$Ux + Uy = 48 = mn$

One-sided test for $Ux = 7$ to determine if Ux is significantly smaller than Uy (Table G, Ref. 4). Reject the hypothesis H_0 at significance level 0.021, where $U < d = 9$. Since the H_0 hypothesis states that the populations are the same and the significance of this hypothesis is very low, the populations must be different.

Two-sided test for $U = Ux = 7$: Reject the hypothesis H_0 at significance level 0.042, where $U < d = 9$.

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