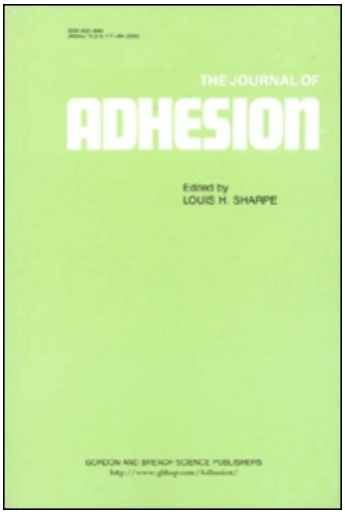

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Experimental Investigation of Aluminum/Epoxy Interfacial Properties in Shear and Tension

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This paper presents the reults of comprehensive testing to characterize the effect of several different surface treatments on shear and tensile bond strength between 7075-T6 aluminum and two epoxy systems: EPON 815/V40 and EPON 828/Z. A rod pull-out test was used to determine interfacial shear strength, modeled after similar tests on reinforced concrete. The tensile bond strength was characterized using a tension test fixture designed in this study. Overall, the interfacial shear strengths were higher than the tension strengths. Surface knurling gave the highest interfacial shear strength, representing a 72% increase over untreated specimens. Phosphoric acid anodization (PAA) was also quite effective in shear. In tension, the highest strength was obtained from specimens treated with the PAA process along with a silane coupling agent. These specimens showed an increase in interfacial tensile strength by a factor of 5.6.

KEY WORDS: Adhesive bonding; aluminum/epoxy interface; aluminum surface treatments; phosphoric acid anodization; silane coupling agent; adhesive films; interfacial shear strength; interfacial tensile strength.

1 INTRODUCTION

Structural weight is of primary importance in the design of aircraft and spacecraft. Since the 1970's there has been a concerted effort to utilize polymeric adhesives, rather than mechanical fastures, for bonding structures.[1] For example, the McDonnell-Douglas AV-8B Harrier forward fuselage assembly is a monolithic, cocured, molded composite structure using 88 parts and 2450 fasteners; a conventional riveted aluminum structure would contain 237 parts and 6440 fasteners. The wing structure of the Harrier is also a cocured composite structure, and saves 330 pounds over an all-aluminum wing.[2] In addition to the weight reduction by replacement of mechanical fasteners, many industries (automotive, sporting goods, medical equipment, and construction) are examining the feasibility of using hybrid composites composed of metal and polymer composites. These hybrid materials are generally mechanically coupled, but other designs utilizing polymer/metal interfacial bonding are being developed.[3–6]

To fully realize the potential of hybrid systems there are several technical issues which must be addressed. Thermal expansion and stiffness mismatch at the interface

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contribute to poor adhesive bonding. However, the most significant problem with bonded joints is the strength of the polymer/metal bondline. An untreated metal surface, such as 7075 aluminum, does not exhibit adequate bond strength with an epoxy. Good bond strength between the epoxy and aluminum is required for adequate load transfer across the bondline. These bonds have the additional requirement that they be hydrothermally stable to prevent environmental degradation. A better understanding of the aluminum substrate surface and its interactions with a polymeric (epoxy) adhesive is important for the aerospace industry and general manufacturing technologies.[3]

Several surface treatments were investigated to increase the shear and tensile bond strengths between 7075-T6 aluminum and two epoxy systems: EPON 815/V40 and EPON 828/Z. For interfacial shear testing a rod pull-out test was used, modeled after similar tests for reinforced concrete.[7,8] This type of test was chosen for ease of manufacture and to correlate with hybrid rod specimens previously tested.[9] The tensile bond strength was characterized by a modified tensile butt joint test as opposed to other popular adhesive tests, such as T-peel tests and single, double, and cross lap shear tests, to avoid bending effects and the complex calculations necessary to derive a tensile strength value from the test data.[10] The ASTM standard for adhesive tension testing was also not used due to the necessity of manufacturing the test grips and difficulty in making the specimens.[11] Initially a butt-type joint test fixture was attempted for testing, as discussed by Anderson *et al.*[12] However, the untreated aluminum/epoxy joints were so weak in torsion that they failed while loading them in the test frame. A modified tensile test fixture was created assuming that interfaces in tension would break before interfaces in shear would. Upon testing it was found that some failures were initiated by shear failure along the side walls. The test fixture mold was then modified to include a taper. This effectively prevented the shear failures previously experienced. Only the EPON 828/Z system was used in tension testing because it was found to have greater repeatability in tension.

The present study was undertaken as a complimentary analysis to development work on hybrid composites combining aluminum with graphite/epoxy materials.[9] The failure mode of these hybrid composites was (near) interfacial without exception. In order to optimize their design and the resulting mechanical behavior, a comprehensive experimental study was undertaken to characterize the effect of various surface treatments or combinations thereof on bond strength between aluminum and epoxy. Both shear and tension modes were investigated because the initiation of failure in the hybrid specimens occured in regions of combined loading.

This work is extremely important and useful to those interested in hybrid materials combining aluminum with polymers and polymer matrix composites. It is also pertinent in the design of adhesive joints in composites structures where combined loading and thermal residual stresses are problematic.

2 EXPERIMENTAL PROCEDURE

A comprehensive test plan was undertaken to fully characterize the interfacial shear and tensile strength of 7075-T6 aluminum/epoxy bondlines. As with almost all interfacial experiments, the failure plane is not truly interfacial, but is confined to a very

TABLE I
Surface treatments and designations for interfacial shear and tension testing

Surface Treatment	Shear Tests		Tension Tests	
	Number	Code	Number	Code
Untreated	10	Uxxx[1]	10	UT
Untreated, Knurled	5	UKxxx	–	–
Adhesive Film w/Etch	5	AFxxx	5	AFT
Adhesive Film w/Etch, Knurled	5	AFKxxx	–	–
Adhesive Film w/Etch, BR primer	5	AFBxxx	5	AFBT
Silane Coupling Agent	14	SCxxx	10	SCT
Phosphoric Acid Anodize (PAA)	5	PAAxxx	5	PAAT
PAA w/BR primer	5	PBRxxx	5	PBRT
PAA w/Silane Coupling	5	PSCxxx	5	PSCT
PAA w/Adhesive Film, BR primer	5	PABxxx	5	PABT
PAA w/Adhesive Film, Silane Coupling	4	PASxxx	5	PAST

[1] xxx denotes epoxy system used (828 or 815)

small region near the aluminum/epoxy interface. Many different types of aluminum surface treatments were examined for increasing the inherent bond strength. A test matrix of all the experiments conducted is shown in Table I. A rod pull-out fixture was used to characterize interfacial shear strength, whereas a specially designed butt tensile fixture was used to obtain interfacial tensile strengths.

Humidity, temperature, and pH levels can significantly affect aluminum oxide formation. The aluminum surfaces were prepared at room temperature (22°C), except when high temperature was required by the surface treatment, and in a range of relative humidities between 20% and 60%. Adhesive bonding took place within 30 minutes after surface treatment, and testing occurred within 24 hours. Higher humidity can cause lower bond strength by causing a greater conversion of the oxide to the bayerite form, which is more brittle than boehmite.[13]

2.1 Interfacial Shear Testing

A schematic of the rod pull-out specimen and test fixture is shown in Figure 1. A 7075-T6 aluminum rod, 19.1 mm in diameter, was tapered down to a 12.7 mm diameter inside the test fixture. An aluminum mold with 6.4 mm wall thickness was used to cure an epoxy plug around the aluminum rod. Epoxy plugs were 50.7 mm in diameter by 101.6 mm long. The top plate of the mold was made from 6.4 mm thick aluminum stock. The test fixture was fabricated from mild steel in two halves, and was held together by four bolts. A 19.1 mm diameter steel bolt was attached to the bottom grips of a test frame and the aluminum rod was placed in the upper grips.

Once the aluminum rod was surface treated and the epoxy plug cured, the specimen was placed inside the test fixture. The test fixture was then transferred to a Riehle uniaxial test frame. The specimen was loaded in displacement control at a crosshead rate of 0.51 mm/min until failure was detected. All testing was conducted at room temperature. The rod was subsequently removed for post failure analysis. The average

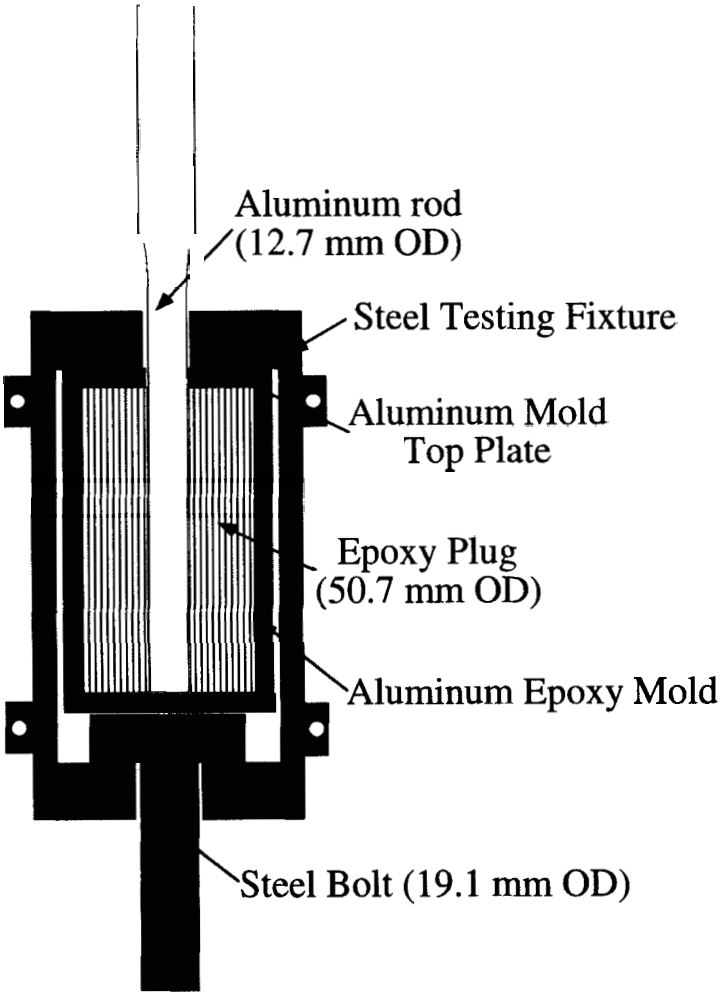

FIGURE 1 Interfacial shear strength (rod pull-out) test fixture (not to scale).

shear strength at failure at the interface was calculated from

$$\tau_{ave,f} = \frac{P_{max}}{2\pi rh} \tag{1}$$

where P_{max} is the failure load, r is the radius of the aluminum rod, and h is the length of the bondline.

For each surface treatment the aluminum was first degreased by immersion in 1,1,1-trichloroethane for five minutes and wiped dry with a clean cloth. Subsequent preparation of the aluminum differed according to the surface treatments desired. The knurled specimens (UK and AFK) were made by mechanically deforming the surface of the rod in a criss-cross pattern to a depth of approximately 0.40 mm. This was

accomplished by placing the rod in a lathe and pressing a knurling tool with three rotating ball cutters into the surface of the rod. The adhesive film specimens (AF, AFK, and AFB) were made using American Cyanamid FM-300 adhesive film. These specimens were cleaned with a room temperature alkaline cleaner and then acid etched using a chromic-sulfuric acid solution according to American Cyanamid specifications,[14] followed by placement of the adhesive film on the surface. For the AFB specimens, Cyanamid's BR-127 corrosion-inhibiting primer was applied using an aerosol sprayer before placing the adhesive film on the surface. BR-127 was chosen because of its compatibility with FM-300 and due to its proven strength characteristics.[15] The SC specimens utilized Dow-Corning Z-6040 silane primer, which was made according to specifications[16] and applied with an aerosol sprayer. The ASTM Standard D 3933-80 was followed for the PAA procedure and used for all PAA specimens.[17]

Each test specimen was made using either Shell's EPON 815 epoxy/curing agent V-40 or EPON 828 epoxy/curing agent *Z* systems. These are two-part epoxies and were mixed with a 40% and 20% by weight curing agent concentrations, respectively. A 24-hour room temperature cure was followed by a one-hour postcure at 90°C for the 815 system. The 828 system was cured two hours at 80°C followed by two hours at 177°C.[18] A Lab-Line Imperial III remote-control radiant heat oven (Lab-Line Instruments, Melrose Park, IL 60160 USA) was used for all heated cure cycles. The molds were coated with Trewax clear paste wax (Grow Group, Inc., City of Commerce, CA 90040, USA) that contains carnauba wax as a mold release agent. The mold was assembled and the rod inserted. The mold was then held upside down on a stand so that the liquid epoxy cured flush to the mold top. The epoxy was poured into the mold and the bottom plate was placed on the mold. The aluminum rod was aligned with a recessed circle in the bottom plate. The specimens were placed under −381 mm Hg vacuum for 15 minutes to remove any air bubbles trapped at the interface. Once the epoxy was cured, the bottom plate and mold were removed before testing.

2.2 Interfacial Tension Testing

The tension test fixture that was used in this work consisted of two main parts, a mold bottom (aluminum bonding specimen) and an upper mold, which tapered into the mold bottom (see Fig. 2). The aluminum specimen was made from 7075-T6 alloy with a 3167 mm^2 exposed surface area for bonding. The upper mold was made from stock aluminum. The tapered region had a 23° slope running from the aluminum/epoxy interface. At the juncture between the two parts of the mold, a high-temperature O-ring was used to prevent the epoxy from leaking during cure. For alignment purposes, and to hold the specimen intact during test set-up and handling, four steel pins (6.4 mm diameter) were inserted into the mold. A stock aluminum mold cap was threaded into the mold top for testing, and the tensile force was transmitted through a steel universal joint to eliminate any moments that might be induced due to improper alignment. The bottom part of the fixture was loaded by a 19.1 mm steel bolt that was threaded into the aluminum specimen. A 6.4 mm diameter, 88.9 mm long steel rod was then threaded into the bolt to help decouple tension forces and bending moments.

Surface treatment procedures for the tensile and shear specimens were the same. After the appropriate surface treatment was performed, the inside of the mold and the

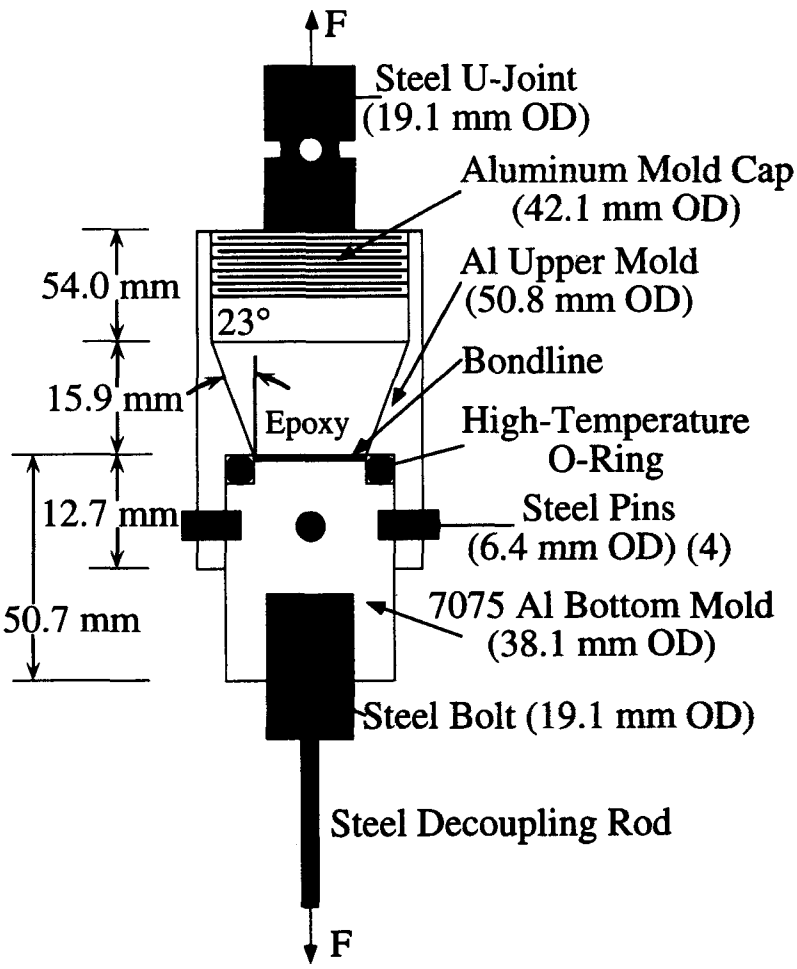

FIGURE 2 Interfacial tension test fixture (not to scale).

steel alignment pins were coated with Trewax as a mold release agent. The treated aluminum specimen and mold were assembled, and 15 grams of epoxy were poured into the mold. The specimens were then placed under −381 mm Hg vacuum for 15 minutes to remove any air bubbles trapped at the interface, and placed in an oven and cured for two hours at 80°C and two hours at 177°C.

Once the aluminum specimen was surface treated and the epoxy cured inside the mold, the aluminum mold cap with attached U-joint was screwed into the fixture top and the steel bolt screwed into the fixture bottom. All tension testing was done on an MTS 880 hydraulic testing system that included a 464.80 Data Display, 410.80 Function Generator, 448.85 Test Controller, and a 413.81 Master Control. All specimens were loaded at 741.4 N/min, and all tests were performed at room temperature. Maximum failure load was recorded and the average tensile bond strength at failure

was calculated from

$$\sigma_{ave,f} = \frac{P_{max}}{\pi(r-t)^2} \tag{2}$$

where P_{max} is the failure load, r is the radius of the bottom mold, and t is the compressed O-ring thickness. The thickness of the O-ring was measured after testing, as indicated by a thin epoxy film that remained under the O-ring.

3 RESULTS

3.1 Interfacial Shear Testing

The results of the rod pull-out tests are shown in Table II and Figure 3. The untreated knurled (UK) specimens had the highest average strength value, while the untreated specimens with 828 (U 828) had the lowest. The specimens fractured in three characteristic ways. Many had large "thumbnail" cracks extending from the rod surface into the epoxy plug at an angle of approximately 45°. This phenomenon was shown by Vinson to occur in pull-out tests of E-glass fibers embedded in epoxy.[19] Figure 4 is a photograph of these cracks in specimen PAS828-4. Others, such as the AFK, UK, and AFB specimens, exhibited complete failure of the epoxy plug. Some epoxy was left adhered to the rods afterwards. A few of the low strength specimens did not exhibit any cracks in the epoxy plug and the rods simply pulled out cleanly.

3.2 Interfacial Tension Testing

The results of the interfacial tension tests are shown in Table III and Figure 5. The PAA-silane coupler (PSCT) specimens had the highest average strength value, while the untreated specimens (UT) had the lowest. Most of the specimens showed some

TABLE II
Interfacial shear test experimental results

Code	Average Shear Strength (MPa)	Standard Deviation (MPa)
U815	9.27	1.02
U828	4.65	1.10
UK815	16.20	2.08
AF828	5.89	0.60
AFK828	15.97	2.28
AFB828	13.34	0.50
SC815	6.18	2.07
SC828	7.60	1.69
PAA815	15.23	0.89
PBR815	14.12	1.35
PSC828	10.62	1.81
PAB828	13.26	0.36
PAS828	8.83	0.79

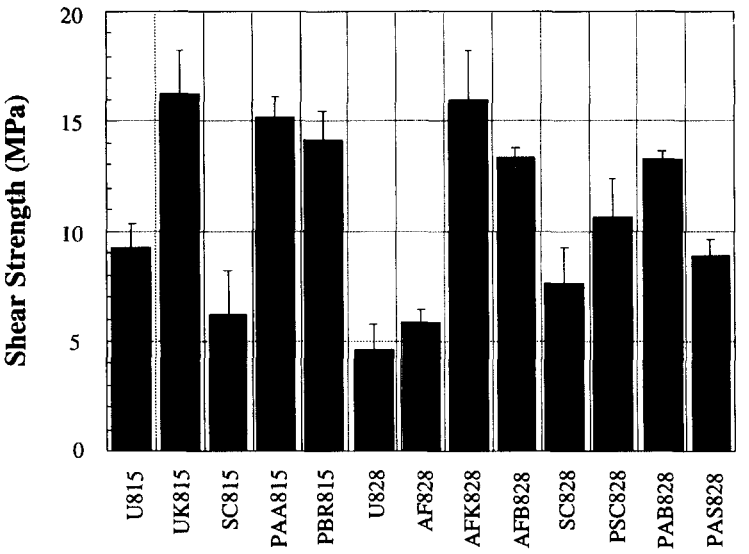

FIGURE 3 Interfacial shear test experimental results.

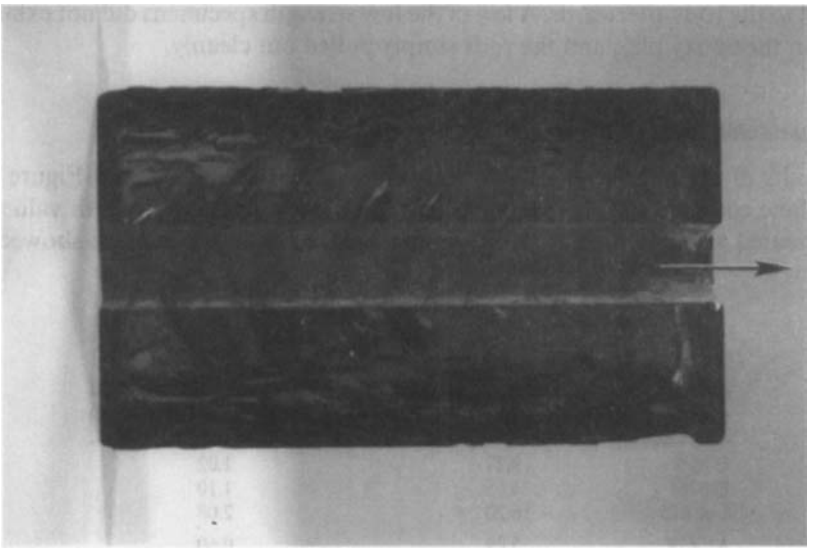

FIGURE 4 Photograph showing interfacial shear test bond surface and cracks. See Color Plate I.

degree of cohesive failure, shown in Table IV. The untreated and silane coupled specimens experienced failures that were almost entirely adhesive. Cohesive failures occurred either near the center or as a ring around the outside of the specimen. The area percentages were determined using a Kontron Image Processing System (IPS) (Eching,

TABLE III
Interfacial tension test experimental results

Code	Average Tensile Strength (MPa)	Standard Deviation (MPa)
UT	1.88	0.54
AFT	9.74	1.57
AFBT	3.68	0.42
SCT	4.45	1.48
PAAT	7.45	1.39
PBRT	6.76	0.89
PSCT	10.57	3.76
PABT	9.48	0.11
PAST	9.95	0.19

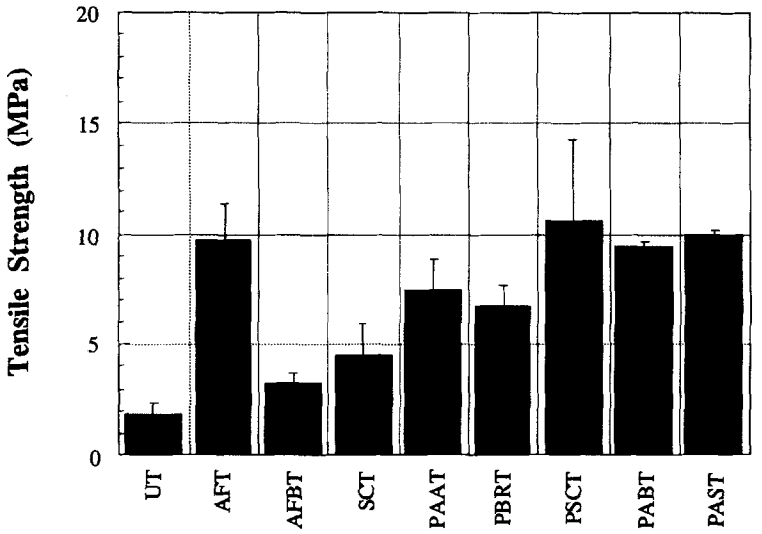

FIGURE 5 Interfacial tension test experimental results.

Germany), version 4.4, by dividing the cohesive area on the specimen surface by the total surface area.

4 DISCUSSION

4.1 Interfacial Shear Testing

It is interesting to note that the U 815 specimens had a higher strength than the U 828 specimens. This may be caused by several factors in addition to the difference in resin chemistry. The U 828 specimens were cured at a temperature higher than the U 815 specimens and the aluminum may have formed larger grains on the surface, disrupting the adhesion process.[20] Also, larger residual stresses are developed at the interface in the 828 specimens and this may have caused the U 828 specimens to fracture at a lower

TABLE IV
Interfacial tension test cohesive failures

Code	Average % Cohesive Failure
UT	< 1.0
AFT	13.8
AFBT	9.3
SCT	< 1.0
PAAT	34.1
PBRT	50.5
PSCT	37.5
PABT	19.2
PAST	8.2

stress level. Since both epoxy systems were tested with untreated rods, basic comparisons between all the surface treatments can be made.

Overall the UK815 specimens showed the highest shear strength. This is not surprising since the mechanical interlocking between the epoxy and aluminum in shear is high. This interlocking mechanism is enhanced by the high wettability of aluminum by epoxy, which allows all the grooves in the surface to be filled with the epoxy prior to gelation. The AFK828 specimens failed at essentially the same stress level as the UK815 specimens. Evidently the mechanical interlock mechanism is dominant and resin chemistry has little effect. The AF828 specimens were stronger than U828, but lower than U815. Using the BR-127 primer with the adhesive film more than doubled the interfacial shear strength, as demonstrated by comparing AF828 and AFB828 specimens.

Another surface treatment that worked well was the PAA process. Even without using a primer system, the PAA815 specimens were nearly as strong as the knurled specimens. The oxide layer formed during anodization features large pores that extend into the base aluminum approximately 300 nm.[21] The micromechanical adhesion that develops at the aluminum oxide/epoxy interface mimics the mechanical interlock mechanism of knurling, but on a much smaller scale. Another approach to enhancing bond strength would be to induce both micro- and macromechanical couplings by performing a PAA process on knurled specimens.

The silane coupling agent surface treatment exhibited some unusual behavior upon initial testing. The interfacial shear strength for the SC815 specimens was found to be lower than for the untreated U815 specimens. When the 828 epoxy system was used, the opposite results were obtained, *i.e.*, the silane increased interfacial shear strength. Silane coupling agents should provide a mechanism for covalent bonding between aluminum and epoxy. While this could be inferred for the 828 specimens, the test results for 815 specimens indicate a degradation in bond strength. There are several reasons why this might be the case with the lower temperature (815) epoxy system. Other research indicates that no chemical interactions occur between epoxy and aluminum surfaces below about 170°C.[22] Above this temperature the heated aluminum substrate facilitates the breakage of the epoxide ring and the formation of a three-dimensional silane network. Since the 815/V40 system never reaches this temperature, the epoxide rings in the organofunctional silane may not have become reactive with the hydrated aluminum surface, thus preventing a covalent network from forming at the interface. Another

possible explanation is preferential diffusion of epoxide or amine. If the epoxide preferentially diffuses and builds up to a half micrometer-thick layer at the interface, this would prevent the silane network from forming.

To test these concepts, several specimens were made by doping the aluminum surface directly. The results are shown in Table V. Three specimens (SC6020) were made using Dow-Corning's Z-6020 organofunctional silane that has an amine group instead of epoxy. If epoxide preferentially diffuses to the surface, this silane coupling agent would effectively bond with it. However, these specimens failed at low stress levels (1.89 MPa). Another test was performed on two specimens using the original Z-6040 coupling agent (epoxy group) and doping the surface of the rod with curing agent V-40 prior to pouring the mixed epoxy into the mold (specimens SCdV40). Again, the strength for these specimens was very low (1.76 MPa). The results of these tests would seem to indicate that the epoxide does not preferentially diffuse to the surface of the aluminum at 90°C. A third test was performed on two specimens designated SCd815 by doping the surface of the rod with 815 epoxide. These specimens were much stronger than the other two groups (5.87 MPa), but still less than the U815 results. These results are consistent with preferential diffusion of amine groups, but the silane coupling agent/epoxide/amine network does not from completely. The results for the 828 specimens cured at 177°C support the finding that the activation energy of the organofunctional silane is such that temperatures in excess of 170°C are required before the epoxide rings open and become reactive.[22]

The four surface treatments that were combinations of PAA, BR-127, Z-6040, and FM-300 gave unexpected experimental results. All four treatments gave shear strengths lower than PAA alone (Table II). PAA with BR-127 primer surface treatment (PBR815) is 7% lower than PAA alone, while PAA with Z-6040 silane surface treatment (PSC828) is 30% lower. The addition of the adhesive film FM-300 to the previous combinations of treatments lowers the shear strength even further. FM-300 lowers the PSC828 treatment by 17%. It is important to note that the shear strength of AFB828 and PAB828 are nearly identical. Because of this, it is believed that the adhesive film exhibits a shielding effect preventing full exploitation of the PAA micromechanical interlocking. The adhesive film does not allow the epoxy to flow freely into the oxide pores, probably due to the presence of the carrier mesh so that little interlocking between the aluminum oxide and epoxy occurs.

4.2 Interfacial Tension Testing

The use of the adhesive film was shown to greatly enhance the interfacial tensile bond strength. This may be due to greater wettability at higher cure temperatures than the

TABLE V
Results of silane coupling agent study

Code	Average Shear Strength (MPa)	Number of Specimens
SC6020	1.89	3
SCdV40	1.76	2
SCd815	5.87	2

liquid epoxy. When the BR-127 chromate primer was introduced with the adhesive film (AFBT), the strength decreased by 66%. This may be due to the large chromium oxide particles present in and around the surface.[23] Microcracks present at the interface may rapidly coalesce around these stress concentrators and cause interfacial failure. The silane treatment increased the bond strength by 58%. If a non-ideal, two-dimensional silane network forms because conditions at the interface (pH, IEPS, contaminants, or uniformity of silane layer) favor oxane bonds to the aluminum surface instead of crosslinking, the interface would be more suited to carrying tensile loads since the primary bonds that develop tend to be aligned perpendicular to the surface.

The PAA surface treatment improved the bond strength over that of untreated surfaces, but less than the adhesive film. Since the pores that develop from the anodization are perpendicular to the surface, they are aligned in the load direction in this case. Micromechanical interlock is left to that of the whiskers that form on the top of the pores, reducing some of the benefits of this surface treatment.[21] With the addition of the BR-127 primer the strength again decreases, in this case by 9%. The PABT treatment showed a marked increase in strength caused by the addition of adhesive film. Although the PSCT treatment showed the highest strength, it also had the highest percentage of deviation. The PAST treatment in nearly as strong as PSCT and has much better repeatability.

A paper by Anderson *et al.*[24] discussed the evaluation of adhesive test methods. They found that the stresses were not uniform in the test described by ASTM D 897.[11] If the thickness of the adhesive is not constant or if the grips are not perfectly aligned and rigid, the initial load is applied at only one edge of the specimen. This has two effects on the resulting bond strength data: 1) the true failure load is higher than indicated, and 2) since the magnitude of misalignment is not the same for each sample, the data scatter is much greater than for other tests. Using the ASTM standard, they found the coefficient of variation to be 36%. A modified fixture incorporating a rubber plate to reduce the effect of both shear and bending misalignment was also tested, and the coefficient of variation was determined to be 6%. An 80% increase in bond strength was also seen. In this study, coefficients of variation ranged from 36% (PSCT) to 1% (PABT). The average variation was about 18%. The variations seen with this test fixture are reasonable considering the multitude of variables that exist during specimen preparation. An improvement in the coefficient of variation could be realized through the use of a clean room during sample preparation.

5 CONCLUSION

Various pathways to improve adhesive bond strength in hybrid aluminum/epoxy systems have been tested. The strength of the interfacial bond using different surface treatments was obtained experimentally both in shear, using a rod pull-out test fixture, and in tension, using a tension test fixture.

Overall, the shear strengths are higher than the tensile strengths (Fig. 6). This conclusion is supported by the observed failure modes in hybrid aluminum/graphite/epoxy specimens.[9] Some conclusions can be drawn about each type of surface treatment technique. The adhesive film performs very well in tension but relatively

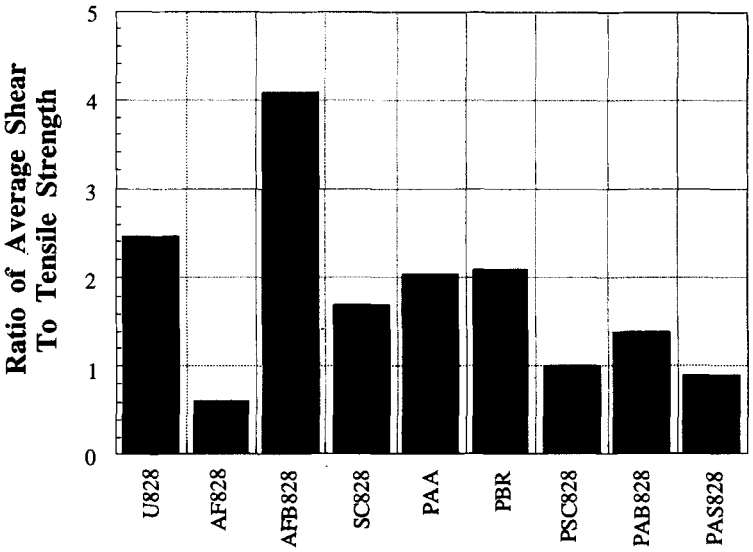

FIGURE 6 Comparison of average shear strength to tensile strength ratios for various surface treatments.

poorly in shear. The silane treatment and the resulting silane network also perform better in tension than in shear. PAA was shown to give very strong interfacial bonding in shear but has a lesser effect in tension due to the structure of the oxide produced on the aluminum surface. The chromate primer BR-127 also performed well in shear but less so in tension. Surface knurling was not tested in tension and, although highly successful in improving bond strength in shear, it is believed that the mechanical interlocking that takes place in shear would not be as great in tension.

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