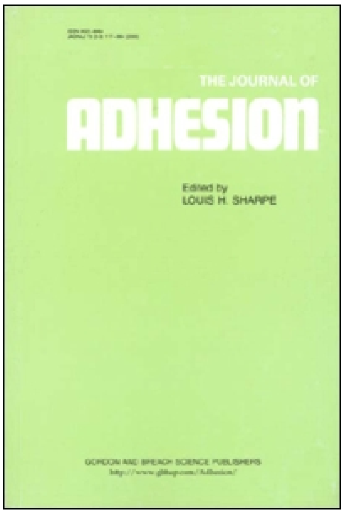

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Surface Roughness and Bond Strength of Adhesives†

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INTRODUCTION

The degree of roughness of a metallic adherend is frequently a design option for an adhesive joint. But which direction should be taken for optimum joint strength? Should the surface be polished or macroscopically roughened, or is roughening inconsequential? In practice, roughening is generally recommended, but upon what basis is this recommendation made and for which adherends and adhesives is it valid?

Garnish and Haskins,[1] in lap shear tests on a one-part hot-curing epoxy bonded to aluminum and steel adherends, found higher strengths for shotblast than degrease treatment. For aluminum adherends, a chromic acid etch provided higher strengths than shotblasting treatment. With a cold-curing amine-epoxy adhesive, shotblasting was more effective for steel adherends than a phosphoric acid in alcohol etch. No information was presented on adherends with both shotblast and acid-etch treatment.

Chessin and Curran,[2] in studying the effect of surface preparation on the lap shear strength of Epon 934 epoxy to 2024T3 aluminum joints, found a sodium dichromate-sulfuric acid treatment to have greater effect on bond strength than gritblasting. Chromate treatment after gritblasting gave a small but definite increase in bond strength over a chromate treatment to an as-machined surface. Joint strength increased with the size of the grit (90 to 24 mesh) used in roughening the surface. The effect of gritblasting was attributed to surface cleaning and surface roughening.

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Rogers[3] reported greater strengths for lap shear joints with modified epoxy and gritblasted 2024T3 aluminum when sharp cutting abrasives were used than when rounded blasting media, such as glass beads or metal shot, were used. Bikerman,[4] in reviewing the procedures for cleaning adherend surfaces (including sandblasting) says, "The contradictions between test results of different investigators may be due partly to the difference between contaminants on the specimens used and partly to the ambiguity of terms describing cleaning operations and subsequent determinations of joint strength." He further states,[5] "A cruder method of accounting for the absence of true interfacial ruptures is based on surface roughness. Because of roughness, a butt joint in the immediate vicinity of the interface is a multitude of scarf joints. As scarf joints are relatively strong, adhesive breaks in a space in which a butt joint still may be considered as such, i.e., where rupture still may progress approximately normally to the external force."

Wake[6] suggests that the efficiency of sandblasting or shotblasting a surface before bonding could, in addition to cleaning, be related to a change in surface topography whereby sharp-edged, deep pits or those with recessed angles are replaced by shallow open structures, which are more likely to be interconnecting or, in any case, more easily filled with resin. The effect of roughening to increase the spreading tendency of a liquid adhesive on an adherend is discussed by Kaelble.[7] However, the danger of trapping air in a rough surface has been pointed out by deBruyne.[8]

Zisman[9] states that the most probable effect of poor wetting is the development of air pockets or voids at the adhesive-adherend interface. Even when $\theta = 0$, there may be gas pockets formed at the adhesive-adherend interface around which stress concentrations can build up. If the adhesive is too viscous when applied, it may never penetrate the accessible surface pores before polymerizing. This situation is aggravated by a larger contact angle or a rougher surface. He justifies the practical use of roughening by the following considerations. If the gas pockets formed in the surface depressions of the adherend are nearly in the same plane and are not far apart, there may be crack propagation from one pocket to the next, and the joint may break as if it had a built-in "zipper." Therefore, if roughness must be accepted, the roughness should be random so that the bubbles cannot form a single line of weakness in the joint. In fact, more may be gained by minimizing the tendency to form interfacial occlusions than by maximizing the specific work of adhesion.

Thus, roughening an adherend can exert a positive effect on joint strength by cleaning contaminants from the surface, increasing surface bonding area, providing a scarf-like surface geometry, and increasing the tendency of the adhesive to spread on the adherend surface. Negative effects can arise if abrasive particles are left on the surface or if wetting is incomplete and an entrapment of bubbles or voids is produced at the interface. These flaws or discontinuities can be detrimental in that they can serve as sources of stress concentration or points of weakness within the adhesive adherend interface.

This investigation is directed toward providing additional information on the relationship between surface roughness and joint strength. Butt tensile and a few bulk shear strengths are being measured for different surface preparations of aluminum and stainless steel substrates. These tests, chosen because they are commonly used, are thought to be simpler in terms of stress analysis. Surfaces are examined before and after fracture by visual, optical, beta-backscatter, radiochemical evaporative rate analysis, and electroplating techniques. A few surfaces were examined with a profilometer and by ellipsometry.

EXPERIMENTAL

The adherends are 6061 aluminum or 304 stainless steel cylindrical plugs, 1 inch in diameter and 1 inch long. The as-machined finish on these specimens has small but visible tool marks. Before sandblasting, the surfaces are abraded through 600 SiC paper to obtain a flat surface. A 40- to 50-mesh (10 to 15 mil) SiO_2 grit is used in sandblasting the bonding surfaces. Excess grit is blown free with dry air. Polished surfaces are obtained by abrading through various abrasive papers and finishing with oil-wetted 6μ and 1μ diamond-dust paste. The cleaning procedure consists of water and acetone or alcohol wash and a degrease in trichlorethylene. The specimens are boiled in water at 93°C (199°F) and either oven dried at 74°C (165°F), platen heated in air to approximately 300°C (574°F), or chromate etched. Aluminum specimens are etched per ASTM D2651 Method 5.1. The stainless steel specimens are immersed for 20 min at 71–77°C (160–170°F) in a solution consisting of 1.75 parts (by weight) sodium dichromate, 1.75 parts distilled water, and 100 parts concentrated sulfuric acid.[10] After the etch, the specimens are rinsed in tap water, then by distilled water, and evacuated under a bell jar to 0.2 Torr.

The adhesives being evaluated are: DER 332, a 172–178 equivalent weight diglycidyl ether of bisphenol A with DEAPA (diethylaminopropyl amine), DEA (diethanol amine), *Z* (a proprietary mixture of aromatic amines), or Versamid 140 (a liquid polyamide curing agent), Metlbond 1301 (a nylon epoxy low-modulus film adhesive), RTV 630 and DC 93-083 (silicone adhesives). The adhesive constituents are degassed, weighted to 0.01 gram, and mixed warm (50–70°C), when possible. The mix was degassed and applied to heated (50–70°C) aluminum or stainless steel surfaces with a spatula. Ten joints were bonded at one time by using the alignment jig described by DeLollis.[11] A spacer gage was used to adjust the bondline thickness to 0.005 inch. Specimens were measured with a micrometer before and after bonding to establish actual bondline thickness. Exuded adhesive was wiped from the specimen surfaces prior to cure, which was at 74°C (165°F) for 16 hours unless indicated otherwise. The specimens were released from their clamped position in the jig at curing temperature after cure and oven cooled to room temperature. A bulk sample of the polymer was cured along with the specimens and checked for hardness with a Shore D durometer. Relative humidity of the laboratory during bonding and curing was 22 to 40%.

The tensile plugs were pulled to failure on a Tinius Olsen testing machine according to ASTM D2095. Temperature, unless indicated otherwise, was 23°C (73°F). Rate of head travel was 0.05 inch/min, corresponding to 65 to 95 psi/sec for most specimens. The joint strengths represent averages of five or more determinations. Standard deviation varies from 5 to 15% and the scatter is, in some instances, up to ±20% of the average.

Beta-backscatter measurements were made on a Twin City Betascope using a C^{14} (0.155 MeV) source. A rough calibration of film thickness against change in beta-backscatter count was made using thin Mylar film. Rate of desorption of radiochemical from the bonding surface was measured on a MESERAN Model 720 evaporative rate analyzer.[12] Approximately 0.05 microcurie of C^{14} labeled diethylsuccinate, 2-ethyl butyric acid, or NN-dimethyl *n*-decylamine, dissolved in 0.02 ml of cyclopentane, was added to approximately 1 cm^2 of bonding surface. The amount retained is measured at periodic intervals. Copper is plated from a Rochelle salt bath[13] at 30 to 60 amp/ft^2 for 30 sec on the fracture surfaces to distinguish the epoxy regions from the interfacial regions. Copper was also deposited by immersion from a copper sulfate hydrofluoric acid solution on a few of the specimens.

RESULTS

Butt tensile strengths of bisphenol A epoxy joints as shown in Table I are consistently higher for sandblasted than polished surfaces, regardless of the cleaning or etching treatment. Similar radiochemical adsorption was shown by both surfaces before bonding. Brittle fracture patterns were observed for joints tested at room temperature, regardless of the surface preparation. Both cohesive and interfacial failure modes were observed. Fracture patterns for joints with polished surfaces frequently showed bare regions and epoxy regions. The bare part of one face matched the epoxy part of the other, thus indicating a failure path that fluctuated between the interfaces. A specimen

TABLE I
Adhesive joint strengths with 6061 aluminum

Adhesive	Weight ratio	Cure °C/hrs	Surface treatment	Butt tensile Joint strength (Psi)	
				Sandblasted	Polished
DER 332-DEAPA	100/7	74/16	WBPH[a]	5940	3740
DER 332-DEAPA	100/7	74/16	CE[(b)]	6260	3240
DER 332-DEA	100/12	74/16	WBOH[c]	6730	2120
DER 332-DEA	100/12	74/16	CE	9850	7700
DER 332-Z	100/20	121/16	WBPH	7020	5560
DER 332-Z	100/20	121/16	CE	7550	6140
DER 332-Versamid 140	70/30	74/16	WBOH	6610	3200
DER 332-Versamid 140			CE	8300	3950
	60/40	74/16	WBOH	6140	
		74/16	CE	7920[d]	4720
		25/2100	CE	7710	3270
		74/16 + 25/2100	CE	7180	5840
		74/16 + 150/2	CE	7250	5220
		74/16	CE + P[e]	7855	4715
			A[f]	7505	3750
	50/50	74/16	WBOH	6430	2160
			WBPH	6990	3680
			CE	8400	5440
Metlbond 1301		138/2	CE + P[g]	8850	8970
DC 96-083		74/16	CE	840	1000
RTV 630 (A4094 Primer)		74/16	CE	550	490
				Compression Shear	
DER 332-DEAPA	100/7	74/16		4480	3260
DER 332-Versamid 140	60/40	74/16		6670	5990

[a] Waterboil, platen heat to ~300°C.
[b] Chromate Etch, ASTM D2651-5.1A.
[c] Waterboil, oven heat 177°C/0.5 Hr
[d] Average of 50 specimens.
[e] A187 (epoxy terminated silanol) primer.
[f] Sulfuric acid anodize
[g] 6706 primer.

was said to fail interfacially when beta-backscatter could not distinguish between the surface prior to bonding and the bare region of the surface after fracture. Evaporative rate analysis and an ellipsometry analysis indicated that there was some organic material on the bare region; whether this was residual adhesive or contamination could not be established. The focal point for fracture initiation in brittle cohesive failures was usually 0.01 to 0.08 inch from the specimen perimeter.

As has been previously reported,[2] the chromate etch gave stronger bonds although no significant change in contact angle or radiochemical adsorption was noted. The water boil treatment was evaluated because surfaces so treated were able to retain adsorbed C^{14}-labeled esters, acids, or amines for extended periods. Up to 95% of the diethylsuccinate deposited was retained after 1 year of open storage. Over 65% of this ester was still adsorbed on a polished surface after 5 months in an oven at 74°C. The greatest desorption occurred during the first hour at 74°C. Saturation of a sandblasted surface after water boil required approximately twice as much of the labeled diethylsuccinate as a polished surface. The sandblasted surface retained 67% and the polished surface 55% of this initial adsorption over a 10-month interval. Most of the desorption occurred during the first few days.

The application of primer A 187 (epoxy terminated silanol) to chromate etched surfaces provided no increase in joint strength, nor did anodizing aluminum surfaces in sulfuric acid rather than subjecting them to a chromate etch. Compression shear strength was also higher for the sandblasted than polished surfaces.

As contrasted with the rigid epoxies, the lower modulus adhesives, Metlbond 1301, DC 96-083, and RTV 630 showed little difference in room temperature joint strength between sandblasted and polished surfaces.

Although the chromate treatment has a strong oxidizing action, one could always conceive of some contaminant from the polishing operation to persist through all treatments. To test this, polishing paste and oil were rubbed onto the sandblasted surfaces and both polished and sandblasted specimens were treated together. The joint strengths still showed the typical difference between the two surfaces.

Tables II and III show that the joint strength of a Versamid epoxy adhesive depends on the degree of abrasion. The polished and sandblasted surfaces represent the extremes in roughness. In order to evaluate the effect of increased surface area of the sandblasted over the polished surface, a different surface geometry was tried. Grooves 0.005 and 0.010 inch deep were machined into the bonding faces of Al and SS specimens. The joint strengths, shown in Table III, were not as high as those for sandblasted surfaces with the same epoxy adhesive and a similar cleaning treatment. Fracture occurred on planes

TABLE II
Joint strengths with abraded and chromate etched 6061 Al adherends and DER 332-Versamid 140 (60/40) adhesive

	Adherend Surface	Butt tensile strength (Psi)[a]
A.	Polished, 1 μ diamond dust	4180 ± 1020
B.	Abraded through 600 paper	4480 ± 1120
C.	Abraded through 280 paper	5650 ± 990
D.	Abraded through 180 paper	5330 ± 1090
E.	Sandblasted (40–50 grit)	7030 ± 1020

[a] 5 groups, each consisting of A through E were solvent cleaned, chromate etched, bonded, and cured together.

TABLE III
Effect of surface geometry on butt tensile strength of DER 332-Versamid 140 (60/40) epoxy joints[a]

		Adherend Surface[b]	Butt tensile strength (Psi)
6061	Al	polished	4720 ± 1000
6061	Al	0.005 inch grooves, negative bondline[c]	5050 ± 760
6061	Al	0.005 inch grooves	6420 ± 500
6061	Al	0.005 inch grooves, sandblasted	7020 ± 1120
6061	Al	Sandblasted (40–50 grit)	7920 ± 530
6061	Al	Sandblasted (10–20 grit)	7680 ± 360
304	SS	polished	4030 ± 840
304	SS	lapped to 2λ[d]	4720 ± 850
304	SS	0.010 inch grooves, negative bondline[c]	5060 ± 340
304	SS	0.010 inch grooves	5110 ± 1020
304	SS	0.010 inch grooves, sandblasted	5510 ± 770
304	SS	Sandblasted (40–50 grit)	7750 ± 840
304	SS	Sandblasted (10–20 grit)	9120 ± 470

[a] 74°C/16 hours cure
[b] Adherend surfaces were chromate etched
[c] Grooves meshed; all other joints had 0.005 inch bondline
[d] Surface not polished

across the ridges. The epoxy in the valleys was intact and not pulled out.

Bond strengths for aluminum and stainless steel surfaces sandblasted with 40–50 size grit were about the same; however, with a coarser grit the stainless steel joints were stronger. For polished surfaces, the aluminum adherends had slightly higher strengths than did the stainless steel. Profilometer measurements before and after chromate etch treatment, Figures 1 and 2, show the aluminum to be etched more than the stainless steel. This microroughening could have contributed to the greater strength for the aluminum specimens. The microroughening of the chromate etch is not visible on the sandblasted surfaces because of the large change in profilometer sensitivity.

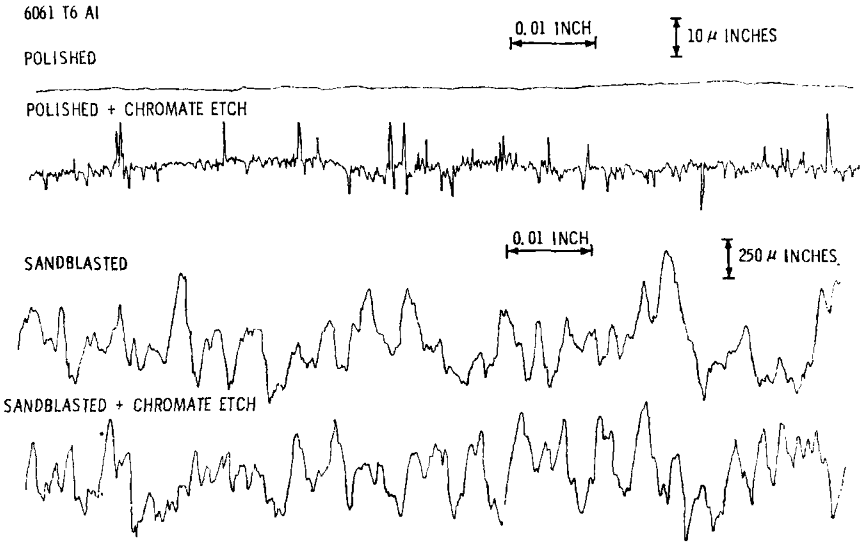

FIGURE 1 Talysurf profilometer traces for 6061 T6 aluminum surfaces

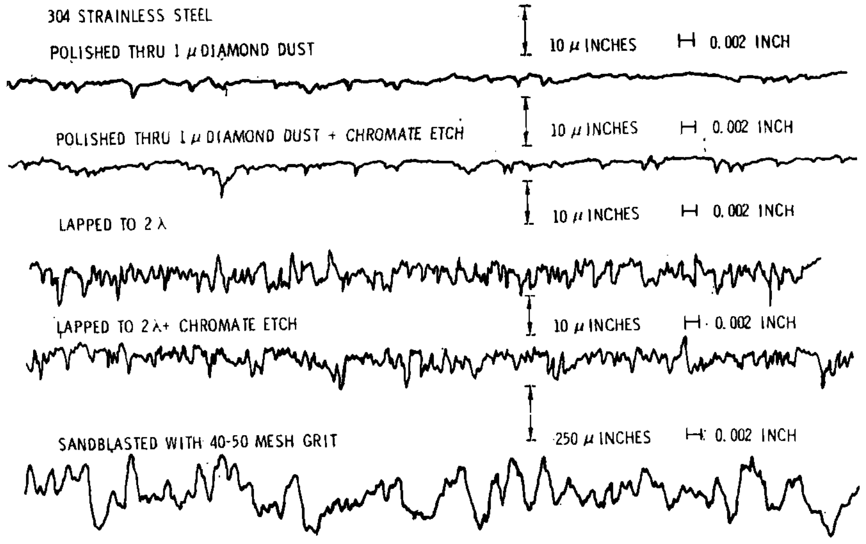

FIGURE 2 Talysurf profilometer traces for 304 stainless steel surfaces

Table IV shows that the metallurgical state of aluminum adherends influences joint strength. For all conditions the chromate etch provides superior joint strength. The T6 or hard condition is preferred for 6061 while for 2024 there was a much greater degree of etching for the annealed or O state which probably accounts for their higher strengths. Copper plating the specimens after fracture showed 95 to 99% cohesive failure for both sandblasted and polished specimens. Copper was observed at the focal point of the conchoidal fracture pattern, thus indicating fracture initiation at an interfacial region of one of the adherends.

TABLE IV

Butt tensile strengths of joints with der 332-versamid 140 (60/40) and Al adherends

	Butt tensile strength (Psi)	
	Polished	Sandblasted
A. *6061 Aluminum*		
1. O-Condition (Annealed)		
Chromate Etched	4780 ± 400	5100 ± 230
No Etch[a]	3490 ± 530	4950 ± 110
2. T6-Condition		
Chromate Etched	4920 ± 600	8210 ± 560
No Etch[a]	3410 ± 790	5570 ± 570
B. *2024 Aluminum*		
1. O-Condition		
Chromate Etched	7410 ± 500	7970 ± 670
No Etch[a]	3910 ± 840	6010 ± 240
2. T4-Condition		
Chromate Etched	4830 ± 550	7810 ± 580
No Etch[a]	3620 ± 500	4810 ± 740

[a] Degreased in trichlorethylene and washed in distilled H_2O at 66°C (150°F)

Variation in joint strength with temperature is shown in Figure 3 for an Epon 815-Versamid 140 (60/40) adhesive. Epon 815 is an epichlorohydrin/bisphenol A-type epoxy resin containing a reactive diluent. Tensile strength data of Ishai[15] for bulk specimens of this same system are included for comparison. Although the strengths are comparable at room temperature, at higher temperatures the adhesive joint is stronger than the bulk polymer. Lewis and Ramsey[16] reported similar behavior for a bisphenol A epoxy cured with diethylentriamine. The difference in joint strength between sandblasted and polished adherends is shown for DER 332-Versamid 140 and Metlbond 1301 in Figures 4 and 5 to depend on the test temperature. Greater strengths for the sandblasted surfaces coincide with temperature regions where brittle fracture patterns were observed.

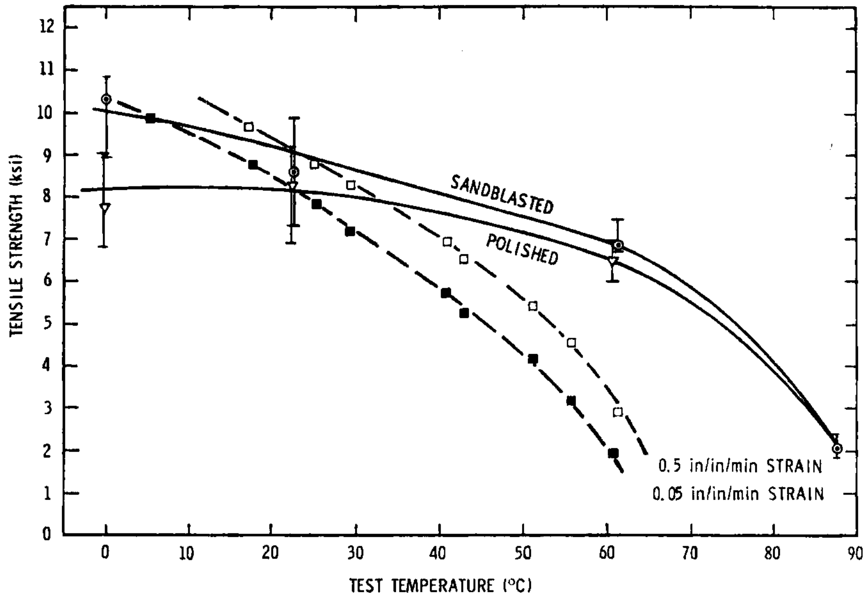

FIGURE 3 Variation of joint strength with temperature for EPON 815-Versamid 140 (60/40) adhesive and 6061 T6 Al adherends. Dashed lines indicate bulk polymer data of Ishai[15]

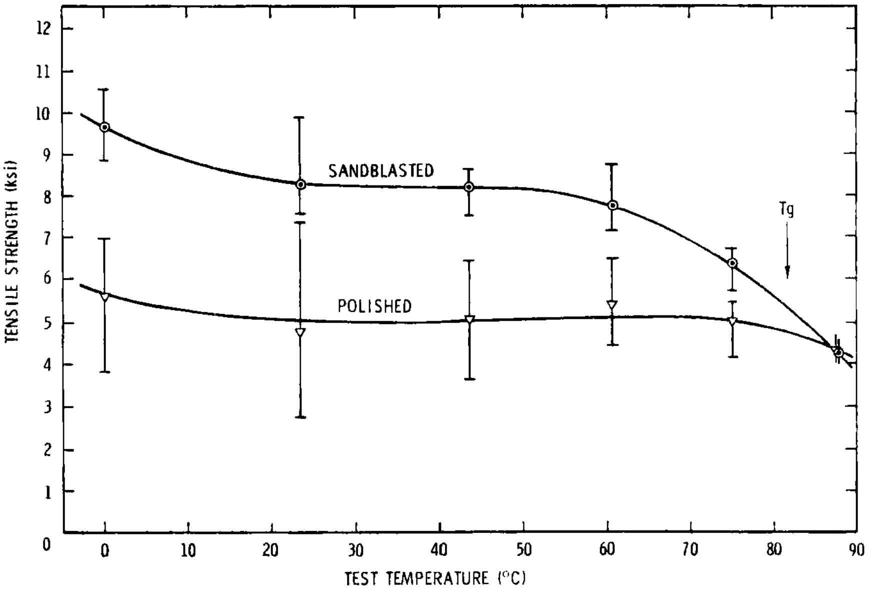

FIGURE 4 Variation of joint strength with temperature for DER-332-Versamid 140 (60/40) adhesive and 6061T6 Al adherends

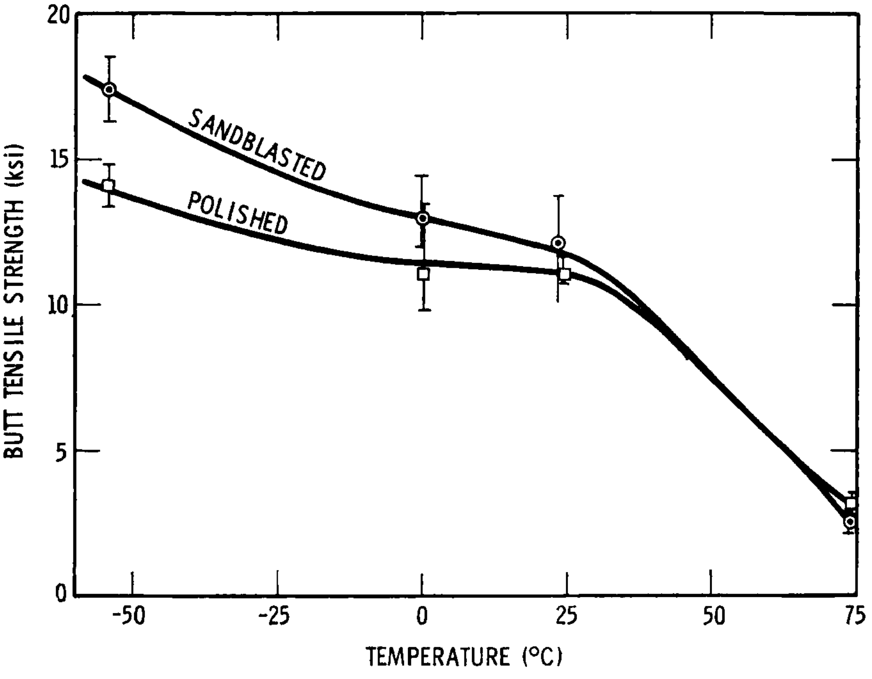

FIGURE 5 Variation of joint strength with temperature for Metlbond 1301 adhesive and 304 stainless steel adherends

DISCUSSION

The improved joint strength with gritblasted surfaces has often been attributed to surface cleaning. This effect although important to surfaces given no further treatment is of less importance to surfaces subjected to additional solvent and chemical treatment. The similar radiochemical adsorption for both sandblasted and polished surfaces and the gradation in behavior with degree of abrasion would be difficult to explain by surface cleaning.

The increase in adhesive spreading and surface area caused by roughening is difficult to assess. The surfaces were water-break-free and the water contact angle was very low (less than 10°) after cleaning and chemical treatments. Adhesive spreading on the two surfaces could not be differentiated visually. The specimens with grooved faces had greater macroscopic bonding areas yet showed no increase in joint strength. Adhesive was not pulled out of the valleys; therefore, one would have to assume that if there was inadequate wetting, it was on the ridges. Sandblasted specimens that were copper plated after fracture showed by optical and scanning electron microscope examination much more copper on the peak regions than in the depressions. Although the increase in wetting and surface area are important, one must still explain the smaller difference between the sandblasted and polished surfaces at higher temperatures, the lack of effect with the low-modulus adhesive, and variation in strengths obtained by changing the hardness condition of the aluminum.

Another way roughening can affect joint strength is by altering the stress distribution at the adhesive-adherend interface. In an adhesive joint, residual stresses are present. The principal one arises from the difference in thermal strain of the adherend and adhesive. Kobatake and Inoue,[17] studying the effect of thermal stress on the mechanics of an adhesive joint state, "Stress distribution changes sharply in the vicinity of the peripheral edge of the bonded plane where some stress concentration occurs. Such a disorderliness of stress distribution, however, diminishes within a short distance from the free edge, and beyond this region the stress distribution in the adhesive layer becomes uniform just as in the case of uniaxial tension." This disorderliness of stress distribution is also thought to be a function of surface roughness.

Brittle adhesives, and most adhesives become brittle as temperature is lowered, fail by a flaw-initiated crack mechanism. The fact that a joint fails cohesively in the polymer does not imply that it fails at the bulk strength of the polymer or at a fixed strength. The ultimate cohesive stress can vary with the adhered, its metallurgical state, and surface preparation. Similarly, specimens have shown partial interfacial failure at stress levels above those expected for the bulk polymer. Figure 3 shows joint strengths at higher temperatures to be significantly greater than the reported bulk strengths of the same polymer. Similar results of Lewis and Ramsey for an Epon 828-DET adhesive have already been mentioned. The joint strengths reported by Wegman and Tanner[18] for a Versamid epoxy at different test temperatures are similar to those shown in Figure 4. Their results and those reported here are estimated to be within a factor of 2 of a 0.05 min^{-1} strain rate.

Hughes and Rutherford[19] using a high-precision microstrain technique to characterize Versamid epoxy joints found the effective modulus of the adhesive to be lower for aluminum than stainless steel adherends. Modulus decreased with joint thickness, and even a 0.058-inch thick joint had a greater modulus (470,000 psi) than the bulk polymer (330,000 psi).

The difference in strength of an adhesive polymer in a joint and in the bulk state can arise from several factors. There is the lateral constraint offered by the higher modulus adherend. In thin brazed and soldered joints, this constraint produces joint strengths three times that of the bulk solder or braze materials.[20-22] This would be expected to be more pronounced in adhesive joints at a higher temperature where the adhesive is in a more ductile state. Another factor is the nature of the polymer formed in the joint. Evidence that the adherend can affect polymer surface morphology has been presented by Cuthrell.[23,24] It is difficult, however, to evaluate its contribution to joint strength or experimentally separate it from the other factors. In view of the brittle failure mechanism of most epoxy adhesives, the type, number, and distribution of flaws or points of stress concentration should be a very important factor differentiating joint from bulk adhesive behavior.

If the polymer is similar, a joint should have fewer inherent or homogeneous flaws because of its smaller volume and surface. However, the most vulnerable region of a joint is at the adhesive-adherend interface, especially near the joint perimeter where combined stresses are maximum. It is here that flaws are expected to have a strong effect on joint strength. This region is also where surface roughness is expected to have its greatest effect.

If the macroscopic surface roughness is random, it can be effective, as proposed by Zisman, in preventing the flaws or points of stress concentration from aligning and propagating along lines or planes of weakness in the joint. Such alignment and propagation appear more likely for polished surfaces than for those with regular ridges.

The roughened surface can also be considered as a reinforcing medium, with the metal asperities assuming some fraction of the load in the interfacial plane. Around the tips of the asperities one would expect regions of stress discontinuity. This appears to be where many interfacial failures occur in sandblasted specimens. At higher temperature or with low-modulus adhesives, where plastic or viscous flow is possible and flaws or points of stress concentration are less important to the strength of the joint, roughness would be expected to have a minor effect.

CONCLUSIONS

1. Random roughening an adherend surface can increase the butt tensile or shear strength of a joint with a brittle or stiff adhesive.

2. The increase in strength is a function of the roughness of the adherend surface.

3. Butt tensile strength can be a function of the metallurgical state of the adherend.

4. A joint can be stronger than the bulk strength of the adhesive polymer.

5. Variation in joint strength with adherend treatment is believed to be associated with a change in stress distribution at the adherend-adhesive interface.

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