JOURNAL OF APPLIED POLYMER SCIENCE VOL. 20, 1475–1482 (1976)

Effect of Surface Exposure Time on Bonds to Aluminum

DAVID W. LEVI, WILLIAM C. TANNER, MARIE C. ROSS, RAYMOND F. WEGMAN, and MICHAEL J. BODNAR, *Picatinny Arsenal, Materials Engineering Division, Dover, New Jersey 07801*

Synopsis

Lap joints with AF-126 adhesive were prepared from surfaces of 2024-T3, 2024-T3 alclad, and 6061-T4 aluminum alloys treated by either FPL etch, sandblasting, or vapor degreasing. The strength data were described by a two-parameter Weibull distribution. Allowing between 1 hr and 30 days to elapse between surface preparation and actual bonding had no appreciable effect on bond strength. This was true for all three alloys surface treated in each of the three ways as well as for bonds either tested at ambient conditions or aged for 30 days at 120°F and 95% R.H. 2024-T3 aluminum, both bare and alclad, formed bonds that showed better strength than 6061-T4 aluminum.

INTRODUCTION

Although the strength of adhesive bonds to aluminum has been studied,[1–7] in discussions with numerous aircraft manufacturers it was found that, while FPL etch was a common means of treating aluminum surfaces for superior durable adhesive bonds, there was a wide range of opinions and shop practices in the maximum times allowed between the acid etch of the substrate and the subsequent adhesive bonding step. Some felt that 24 hr was the maximum safe time for the surface exposure time (SET), since the surface was subject to too much oxidation and this led to a bonding surface giving too much scatter in the results. Others indicated SET times varying from days to as long as a month. In order to determine the maximum safe time of exposure of the surface to oxidation, SET was varied from as little as 1 hr to a maximum of 30 days in the present work.

Since many armament items, for varied reasons such as proximity to explosives or propellants, do not lend themselves to acid bath etching, there are times that the generally less desirable solvent degreasing or mechanical sanding surface treatments must be used. We wanted to determine change in SET with these surface preparations also, especially since sanding presents a new aluminum surface which promptly begins to oxidize. Degreasing had been reported as an unreliable method for long-term durability around 1958.[8]

EXPERIMENTAL

Materials

The three aluminum alloys, 2024-T3 alclad, 2024-T3, and 6061-T4, were manufactured by Alcoa in sheets 0.963 in. thick. AF-126 is a supported-tape, modified epoxy adhesive manufactured by the Minnesota Mining and Manufacturing Company. It is cured at 250°F within 1 hr; 50 psi pressure is required during the cure period.

Preparation and Conditioning of Specimens

The aluminum sheets were cut into 12 by 4 in. panels. Two panels were bonded with a ½-in. overlap along the 12-in. edge. All bonded panels were cut into 1-in.-wide lap shear specimens on a Do-All band saw. A light feed rate was used in conjunction with a fast blade speed (4500 ft/min). The blade was a Do-All standard carbon precision band saw blade, ½ in. wide, with 24 teeth/in. The bonded panel was shimmed during cutting to prevent any flapping or vibrational motion of the unsupported half, thereby minimizing any potential damage by unnecessarily stressing the bond area.

After cutting, every other specimen was conditioned for seven or more days at 73°F and 50% R.H. before testing. The other half of the specimens were conditioned for 30 days at 120°F and 95% ± 5% R.H. in a Blue M Counter-Flow Combination Temperature and Humidity Cabinet before testing.

Surface Preparation

Vapor Degreasing. The panels were washed with acetone and degreased in perchloroethylene vapor, using a Crest Ultrasonic Degreaser, Model 2001.

Sandblasting. After washing the panels with acetone, sandblasting was accomplished by using a laboratory sandblast unit containing 20–40 mesh silica sand and 90 psi air pressure. The dust was removed by brushing the sandblasted faying surfaces with a short-haired still brush.

Acid Etching (FPL Etch). The panels were washed with acetone and treated by immersion of the bonding surface into a solution containing 1 pbw sodium dichromate, 10 pbw concentrated sulfuric acid, and 30 pbw deionized water. The solution temperature was 140°F, the immersion time, 10 min. The treated panels were rinsed for 1–2 min in running tap water at 104°F, rinsed with deionized water at room temperature, and dried in an air-circulating oven at 140°F.

Surface Exposure Before Bonding

Panels that had undergone surface preparation were conditioned in a controlled environment of 73°F and 50% R.H. for periods of 1–4 hr, one day, two days, seven days, 14 days, and 30 days. At the end of each time period, a group of panels was removed for bonding.

Bonding Procedure

Panels to be bonded were removed from their conditioning environment no sooner than 30 min before bonding. A strip of AF-126 film adhesive (at room temperature) approximately ¾ in. wide and 13 in. long was placed on one panel with the tape protruding over the 12-in. edge by approximately ⅛ in. The mating panel then was placed on top of the first one with a ½-in. overlap.

TABLE I
Correlation Coefficients for Linear Weibull Distribution Plots

	Sandblasted		Vapor degreased		FPL Etch	
Al Alloy	Ambient	Aged	Ambient	Aged	Ambient	Aged
6061T4	0.977	0.984	0.973	0.946	0.981	0.984
2024T3	0.985	0.973	0.943	0.985	0.983	0.982
2024T3 Alclad	—	—	0.993	0.962	0.989	0.982

In all cases, the overlap was controlled since all panels were previously scribed and drilled at the edges to permit the use of flush aluminum rivets in order to prevent dislocation of the panels. The assembled panels were placed in a hydraulic ram press, 50 psi pressure was applied, and heated to 250°F. Cure was for 1 hr at 250°F and 50 psi pressure.

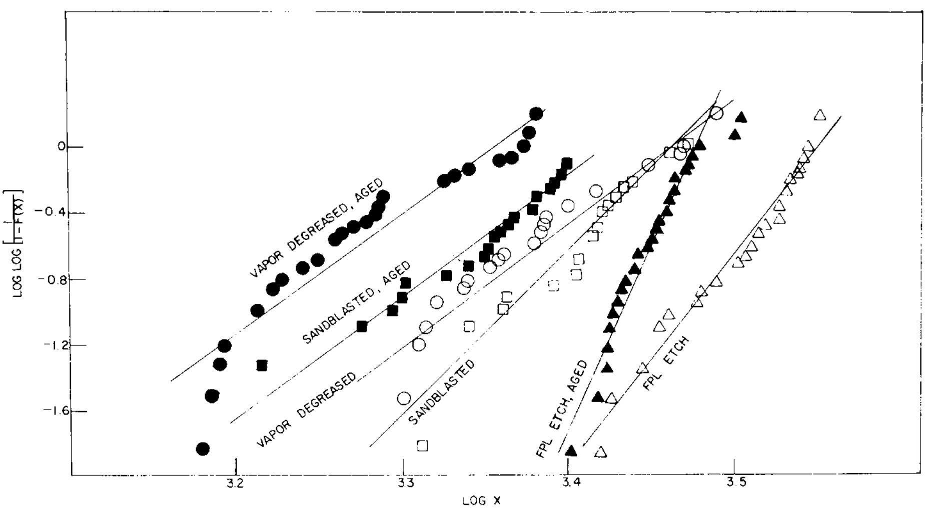

Fig. 1. Linear Weibull distribution plots for 6061T4 Al bond strengths after various surface treatments.

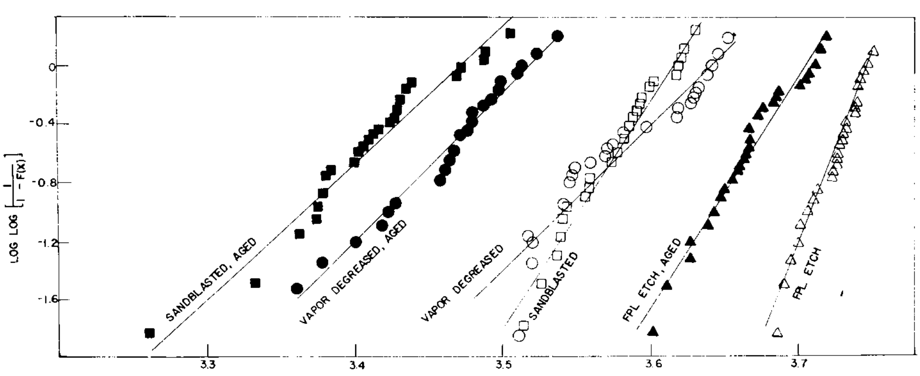

Fig. 2. Linear Weibull distribution plots for 2024T3 Al bond strengths after various surface treatments.

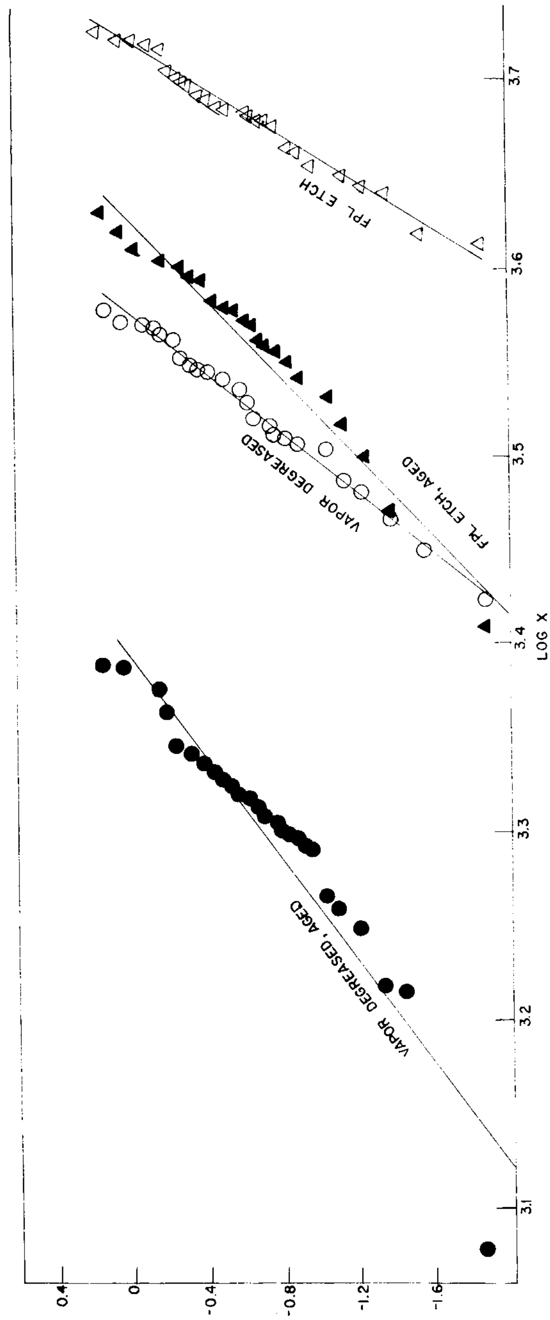

Fig. 3. Linear Weibull distribution plots for 2024T3 alclad Al bond strengths after various surface treatments.

Testing

The specimens to be tested were first conditioned for at least 24 hr at 73°F and 50% R.H. Tests were made at 73°F and 50% R.H. using a Baldwin Universal Test Machine. The load rate was 2400 psi/min.

Adhesive Glueline Thickness

Panels were assembled and cured as described above, except that Teflon tape was placed between the adhesive and the adherends. After removal of the adherends and the Teflon tape, the adhesive glueline was measured with a micrometer. The glueline thickness was 2.1–2.7 mils.

RESULTS AND DISCUSSION

In order to facilitate comparison between the effect of different processing parameters and between the different aluminum alloys, the data were fitted with a Weibull distribution function. This function[9,10] was used in the form

$$\log \log \frac{1}{1 - F(X)} = -\log \alpha + \beta \log (X - \gamma) \tag{1}$$

where $F(X)$ is the distribution function, i.e., the fraction of samples failing at a shear strength (psi) of X or less; X corresponds to the shear strength values; and α, β, and γ are the parameters of the function. A plot of the left-hand side of eq. (1) versus $\log (X - \gamma)$ should give a straight line. γ is selected on an iterative basis by making trial plots[1]; α and β may be evaluated from the slope and the intercept.

In the application to the present data, all 30 data points in each case are tabulated in order of increasing bond strength. The one exception to this

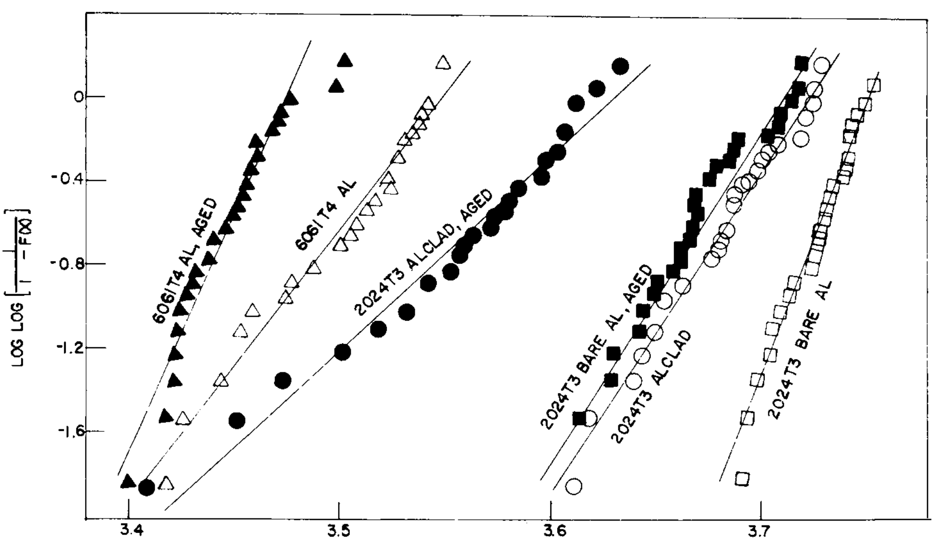

Fig. 4. Linear Weibull distribution plots for Al alloy bond strengths after FPL etch.

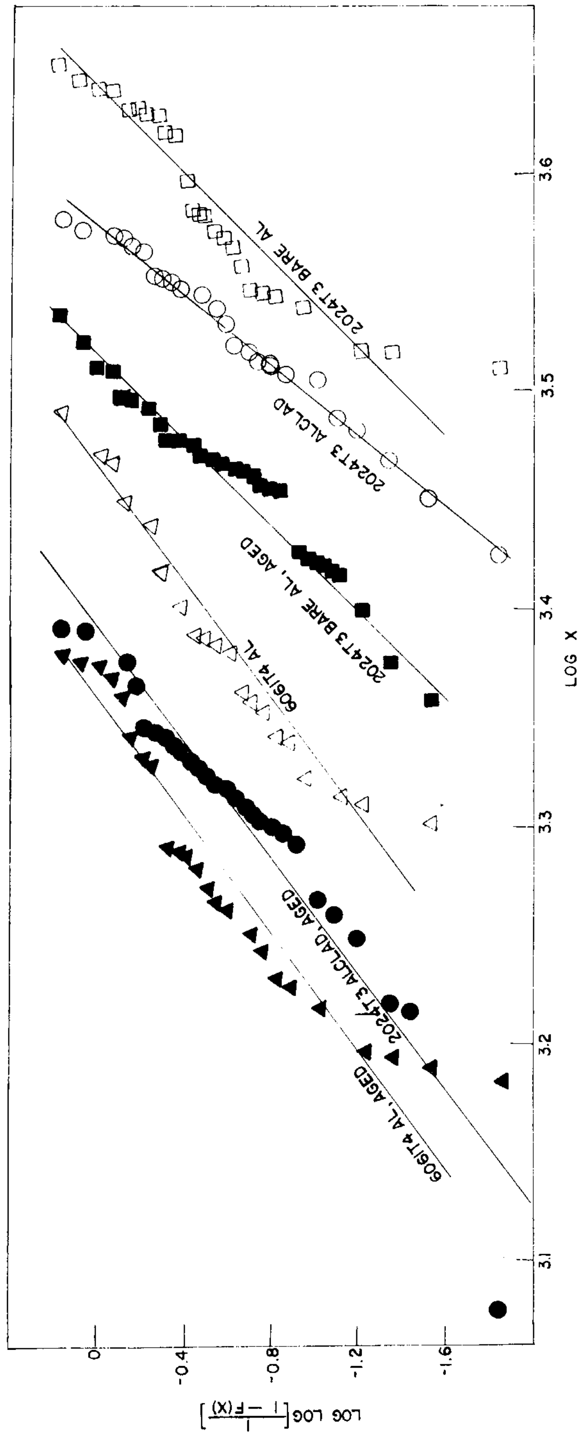

Fig. 5. Linear Weibull distribution plots for Al alloy bond strengths after vapor degreasing.

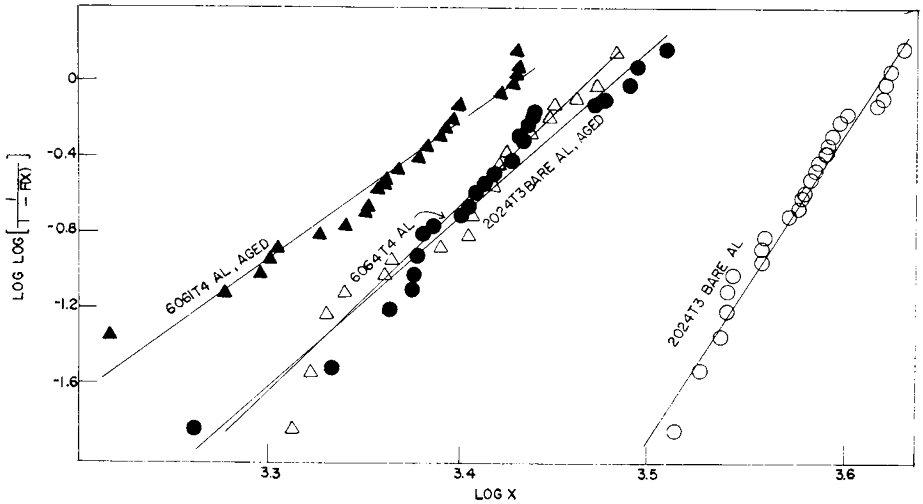

Fig. 6. Linear Weibull distribution plots for Al alloy bond strengths after sandblasting.

procedure occurred for the sandblasted, aged 6061T4 aluminum bonds where there were two very wild (low value) points. These two points were not used in the Weibull distribution computation. Had they been used, the correlation coefficient would have dropped from 0.984 to 0.866. However, the conclusions in this paper would not have been affected in any way. Plotting the data according to eq. (1) with $\gamma = 0$ and using a computer to calculate linear correlation coefficients gave the results shown in Table I. For our purposes, taking $\gamma = 0$ and hence using a two-parameter Weibull distribution appears to be satisfactory.

The fact that all of the data can be fitted to the same distribution in each case indicates that whether bonding is accomplished immediately after surface treatment or after up to 30 days makes no appreciable difference in the strength of the resultant bond. This seems to be true both for specimens tested at ambient conditions and for those aged in a harsh environment.

The superiority of chemical surface treatment is confirmed in Figures 1 through 3. It is interesting that those aged in a harsh environment FPL etch treated surfaces led to bonds that were a little stronger than the vapor degreased or sandblasted bonds kept at ambient conditions. These figures indicate that there is not a noteworthy difference in the effect of vapor degreasing or sandblasting on resultant bond strengths. As would be expected, harsh environment aging markedly lowers bond strength in every case. The alclad 2024-T3 was not sandblasted since the mechanical abrasion might have penetrated the thin alclad layer.

Each aluminum alloy used presents a slightly different surface composition.[11] The 2024-T3 aluminum is alloyed chiefly with copper and silicon. The alclad version of the same alloy shows an essentially pure aluminum surface. The 6061-T4 is alloyed primarily with silicon and magnesium. There are distinct differences in strengths of bonds formed by these alloys, as clearly shown in Figures 4 through 6. The most noteworthy feature of these plots is the clear indication that the 6061-T4 alloy forms noticeably weaker bonds than 2024-T3 after any of the treatments.

The alclad tends to give slightly lower bond strengths than the corresponding base aluminum. The lower bond strength noted for the 6061 alloy is in accord with the results of Wegman et al.[12] who related bond properties to mechanical properties of the adherend. Their *Sy* values[12] for the three adherends are as follows: 6061-T4 alloy, 8,000 psi; 2024-T3 alclad, 20,000 psi; 2024-T3 alloy, 21,000 psi. These values would indicate that the order of decreasing strength would be 2024-T3 alloy >2024-T3 alclad >6061-T4, with the alclad system showing values only slightly lower than those for base 2024-T3.

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Received June 9, 1975
Revised August 29, 1975