Investigation of Outlife Time on the Environmental Durability of P2-Etched, Adhesively-Bonded Aluminum Alloys Using the ASTM Wedge Test

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Abstract

P2 etchant is an environmentally-friendly aluminum etchant which has the potential to replace the Forest Products Laboratory (FPL) etchant as the industry standard. Environmental durability of adhesively-bonded aluminum surfaces etched using a paste version of the P2 etchant were tested using the Boeing-developed wedge test (ASTM D3762 - 03(2010)). This project specifically aimed to examine the relationship between outlife time (the time between etching and adhering) and the ability of bonded aluminum samples to pass the wedge test. Two aluminum alloys, 2024-T3 and 7075-T6, were wedge tested and the etched surfaces examined with an atomic force microscope (AFM) and a scanning electron microscope (SEM). The etchant improved durability of the bonded specimens and helped produce passing 2024 specimens for times ranging up to one week. Results of the 2024 testing demonstrated slightly decreased bond durability on average with increased outlife times, while the results of the 7075 testing were less conclusive and require more investigation to make meaningful conclusions. With more 2024 testing, the data could ideally be used to find a consistent critical outlife time near where bond durability decreases below the minimally-acceptable value. The results of this study may help Raytheon Company to improve their manufacturing procedures by defining a broader range of acceptable outlife times.

Keywords: etchant, aluminum, adhesive, wedge test, environmental durability, P2, FPL, oxide

1. Introduction

The project aim is to find a statistically significant relationship between outlife time (the time between etching and adhering) and the rate of aluminum-bonded samples passing the ASTM Wedge Test for environmental durability. Ideally, the test data can be used to find a critical outlife time near where bond strength significantly decreases. Two aluminum alloys, 2024-T3 and 7075-T6, will be wedge tested and their surfaces examined under atomic force microscope (AFM) and scanning electron microscope (SEM). Differences in oxide layer formation between the alloys may affect their critical outlife times. The results of this study may help Raytheon Company to improve their manufacturing procedures by defining a broader range of acceptable outlife times.

2. Literature Review

This section focuses on the wedge test for determining environmental durability, as defined by ASTM D3762-03, including the origins of the test and its purpose. The types of aluminum alloys to be tested, the adhesive used, common surface preparation procedures, oxide layer formation mechanisms, and other factors surrounding the Forest Products Laboratory (FPL) and P2 etchants are also covered.

2.1 Broader Impacts

In the spring of 1988, a Hawaiian island-hopping 737 heading from Hilo to Honolulu suffered catastrophic fuselage failure that resulted in the death of a stewardess. The adhesive connecting aluminum sheets to one another broke down and passed the majority of stress onto the rivets. The stress concentrated at these rivets caused cracks to form and propagate in the airplane shell. The most likely causes of this adhesive failure stems from corrosion damage which was exacerbated by Hawaii’s humid atmosphere. The lack of uniformity and consistency of aluminum surface treatments has also been thought to have made matters worse [1]. Leaving a cleaned and/or etched metal subject to normal environmental conditions may result in dust or other particles settling on the metal surface, obscuring the desired porous oxide for bonding. Last year’s Materials Engineering senior project team, working with Raytheon, sought to examine the relationship of the time between etching and bonding aluminum and the shear strength of those bonds through the ASTM D1002 lap shear test. This year, the project is to examine the durability of the adhesive bonds in humid environments.

The etchant used, P2, provides an alternative to the more prevalent FPL etchant. Because of the hexavalent chromium and other ingredients found in FPL, this etch is considered to be both carcinogenic and toxic [2]. P2 etchant cuts out the carcinogenic risk and minimizes toxicity for a far more environmentally-friendly experience.

2.2 Aloha Airlines Disaster Reveals Need for Improved Durability Test

Adhesive bonding of aluminum is an important industry process for fabricating seamless joints without the need for rivets or bolts. This is especially important in the aircraft industry for producing thinner lap joints on planes that help reduce manufacturing costs and overall weight [3]. With the average plane running tens of thousands of cycles in its lifetime, it is crucial that the durability of these bonds can be simulated in a short period of time before a finished aircraft is assembled. Early test methods for evaluating aluminum bond durability were proven insufficient in 1988 when Aloha Airlines Flight 243 experienced explosive decompression mid-flight and the upper lobe of the fuselage was torn off, sweeping one flight attendant overboard [3]. The failure was the result of low bond durability in a lap joint, which allowed significant debonding, corrosion, and premature fatigue cracking to occur. According to Boeing, the bonded joints had passed existing accelerated fatigue test methods, but the testing did not take into account in-service environmental effects such as humidity. The Wedge Test (ASTM D3762) was later developed to more reliably test the durability of adhesively-bonded aluminum joints.

2.3 The Wedge Test and its Purpose

The wedge test utilizes elevated temperature and humidity to determine the environmental durability of adherend surface preparations far more reliably than conventional lap shear or peel tests [4]. After two 1” x 8” x 0.125” aluminum coupons have been joined by appropriate surface preparation and bonding procedures, a wedge is driven into the bondline longitudinally and the specimen is exposed to a standard test environment for one hour or more. A common test period is a 1 hour exposure to over 95% relative humidity at 122°F [5]. The initial crack length along the specimen is measured on both sides and averaged before exposure and the change in length (crack growth) is recorded likewise at the end of the test period. The coupons are then pulled apart and the failure mode is reported as mostly cohesive (bond separates from itself) or mostly adhesive (bond separates from coupon surface). Initial crack length, crack growth and joint failure mode are all functions of the adherend and surface treatment being considered, so acceptance criteria must be established accordingly [4]. An adhesive failure or a large crack growth usually indicates that the test specimen has failed and is indicative of poor surface preparation and a resultant poor resistance to extended periods of exposure to stress in humid environments.

The purpose of the wedge test is to both quantitatively and qualitatively describe a bonded joint's durability and to verify that the proposed surface preparation has been done properly. Lap shear tests can also be performed to assure proper mixing and curing of adhesives used by testing if bond strength is near the theoretical strength. A failed specimen indicates that the aluminum surface oxide layer or the adhesive agent were inadequate for proper bonding, which would result in premature failure of a part in service.

In this study, the wedge test was used on aluminum specimens with variable times between P2 etching and adhesive application to evaluate the maximum time after etch when a bond can be made without compromising its durability. The results were expected to yield an optimal or maximum acceptable time after etching that Raytheon Company can bond its 2024-T3 joints.

2.4 Aluminum Alloys Used

Due to their common use in the aerospace industry, aluminum alloys 2024-T3 and 7075-T6 were subjected to the wedge test. 2024 consists of high amounts of copper and magnesium while 7075 contains zinc, magnesium, and lesser amounts of copper. Both alloys are solution-treated, cold-worked, and aged (natural aging for 2024-T3 and artificial aging for 7075-T6). Other notable differences in the properties of the two alloys are the higher yield strength and ultimate tensile strength of 7075 and the higher % elongation of 2024 (Table I).

Table I: Properties of aluminum alloys used in wedge test

Aluminum alloy	2024-T3 [6]	7075-T6 [7]
Primary alloying elements	Cu, Mg	Zn, Mg, Cu
Density (g/cm^3)	2.78	2.81
Elastic modulus (GPa)	73.1	71.7
Yield strength (MPa)	324	503
Ultimate tensile strength (MPa)	469	572
Elongation (%)	19	11
CTE (µstrain/℃)	23.2	23.6

2.5 Surface Preparation and Oxide Formation

In adhesive bonding of aluminum, the two most important factors to consider are the bond strength and durability. Both are directly related to the chemistry of the adhesive and how well it bonds to the surfaces. Since the adhesive in this study is fixed, Loctite EA 9394, the focus will be on the factors surrounding bonding surface (substrate) quality. There are many aluminum pre-treatment procedures including mechanical abrasion, vapor degreasing and alkaline cleaning [8]. None of these treatments, however, produce suitable oxide layers for adhesive bonding. The surfaces are usually left inactive and oxide structure is typically rough at the micro-scale and above. Electrochemical treatments can be used to etch away the relatively thick oxide layer that is weakly adhered to the aluminum surface, leaving a thin, porous, well-adhered oxide layer that is chemically active to form good adhesive bonds. These treatments work by attacking regions of high electrochemical potential in the aluminum alloy surface, usually around the alloying elements. The phosphoric acid anodizing (PAA) process is a commonly used electrochemical method, but requires immersion of the aluminum in an electrically charged bath which limits its application for many types of assemblies.

2.6 Environmentally Friendly P2 Paste Etch

The ability of aluminum to almost instantaneously form a thin oxide layer over its entire exposed surface represents one of its most important traits. The oxide layer protects the metal from some deformation and keeps it stable in variable conditions by increasing thickness as humidity increases [9]. Accordingly, the thickening passivation layer provides a key defense against corrosion.

Applying etchants to aluminum surfaces reshapes the oxide layer into a more porous and thinner coating which makes it more advantageous for adhesives to bond with it. The porosity gives the adhesive more areas to fill while the reduced thickness allows the oxide to better resist shearing forces. The Forest Products Laboratory (FPL) etch morphs the aluminum oxide into a “fine finger-like structure” around 400 Å tall and 50 Å thick (Figure 1) [10].

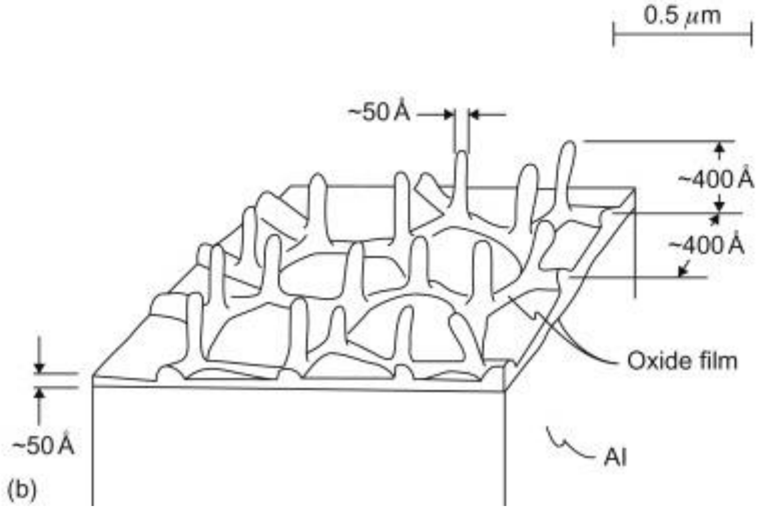

Figure 1: Representation of the newly-formed oxide layer as a result of FPL etching [10].

In addition to yielding similar results as the FPL etchant, the P2 etchant has the safety and environmental benefit of being chromate-free because it uses ferric sulfate in place of sodium dichromate (Table II). Due to the presence of chromates, common etchants like FPL have a carcinogenic risk associated with them and can also cause damage to the respiratory system. As a result, the Occupational Safety & Health Administration (OSHA) has regulated and in some cases even restricted their use where acceptable alternatives exist [11, 12].

Table II: Comparison of the ingredients in FPL and P2 etchants

FPL	**P2**
Sodium dichromate Sulfuric acid Deionized water	Ferric sulfate Sulfuric acid Deionized water

2.7 Comparison to Previous Work on Outlife Time

During the 2014-15 school year, a Cal Poly senior project group compared the lap shear strengths of FPL and P2-etched 2024 aluminum, finding that they yielded comparable results which were statistically greater than those of non-etched aluminum. The seniors also investigated the effect of outlife time on the lap shear strength of three different P2-etched aluminum alloys. Their results showed that shear strength generally decreased with increasing outlife time. The highest bond strengths were achieved when samples were bonded immediately after etching, but with higher outlife times strength decreased significantly until leveling off at about two thirds the maximum strength. The students also found that a high number of their samples debonded, or failed adhesively. These were discounted from the results on the grounds that sample construction was to blame [13].

Based on these results, it seems that outlife time does negatively impact the performance of adhesive bonds and that adhesively failed samples should be regarded with caution. In general, it seems that well-prepared wedge samples should not fail at low outlife times (less than one week). Assuming lap shear strength decreases with increasing outlife time, it is not certain that a weaker bond, about two-thirds the optimal strength, will result in a large enough crack growth to be considered failed in the wedge test. Ideally, this experiment will result in a distribution of crack growths near and beyond 0.30” (the cutoff for passing specimen) with higher outlife times, which could help determine an exact outlife time beyond which to recommend re-etching of a part. The goals here are to (1) find how closely environmental durability of adhesively-bonded aluminum correlates with outlife time and (2) determine the physical morphology of a P2-etched surface oxide to compare with published descriptions of FPL-etched aluminum surface oxides.

3. Experimental Procedure

3.1 Materials and Equipment

- Aluminum alloys 2024-T3 and 7075-T6 were both used for the wedge test. Coupons were band-saw cut from 2024 strips, sheared from 7075 sheets, and water-jet cut from 2024 and 7075 sheets.
- The P2 etchant contained, by weight, 56% deionized H_2O, 29% H_2SO_4, 10% $Fe_2(SO_4)_3$ (97% anhydrous), 5% Cab-O-Sil, and <1% methyl red.
- In addition to the etchant, specimen preparation included 7447 Scotch-Brite pads and Ajax oxygen bleach cleanser.
- Loctite Gray EA 9394 two-part epoxy adhesive (100:17 A:B ratio) was used to bond each wedge specimen and 0.005” glass beads were mixed in at 0.5% by weight to set the bondline.
- For humidity testing, a large desiccator jar partly filled with a 1 L saturated solution of potassium sulfate in water provided an enclosed humid environment, which could be heated inside a low-temperature oven.

3.2 Wedge Test Assembly Preparation

The initial set of 2024 coupons (6” long) and wedges (1” long) were band-saw cut to length from strips supplied by Raytheon. Metal belt grinders were used to machine pointed tips on each wedge. For the first set of 7075 coupons, sheets were cut to width and length using a metal shear. For the last batches of 2024 and 7075, sheets were sent to Dugandzic Design & CNC for cutting via water-jet.

Each test coupon underwent a similar, if not exactly the same, treatment prior to bonding. The process started by wiping the bonding surface with isopropanol to dissolve any oils and generally clean it. Next, the surfaces were Scotch-Brite scrubbed with deionized water and Ajax in order to generate a water break-free surface. The coupons were dried using paper towels.

The P2 paste etchant was applied to each coupon so as to completely cover the surface for 20 minutes until being washed off. The coupons were then dried for 10 minutes in a 160 °F oven. Between this step and bonding, all coupons were stored in a drawer within a climate-controlled laboratory.

Following the duration of outlife time, an adhesive would be made up of 100 parts A, 17 parts B, and a small amount of 5 mil glass beads. Before applying the epoxy to the coupons, a line was drawn across the width of the coupons ¾” away from the end. Epoxy was then applied to pairs of coupons along the surfaces, except the marked-off square. In order to assure a consistent bondline, multiple alligator clips were evenly spaced to hold the coupons together for the duration of adhesive curing. These assemblies were left overnight to cure and then post-cured in a 200 °F oven for one hour.

The final wedge test assembly was formed by driving a 1” wedge fully into the coupons at the end that was left unbonded (Figure 2). After an hour, to assure that the crack growth had slowed to a marginal rate, the initial crack length was measured at 4x magnification.

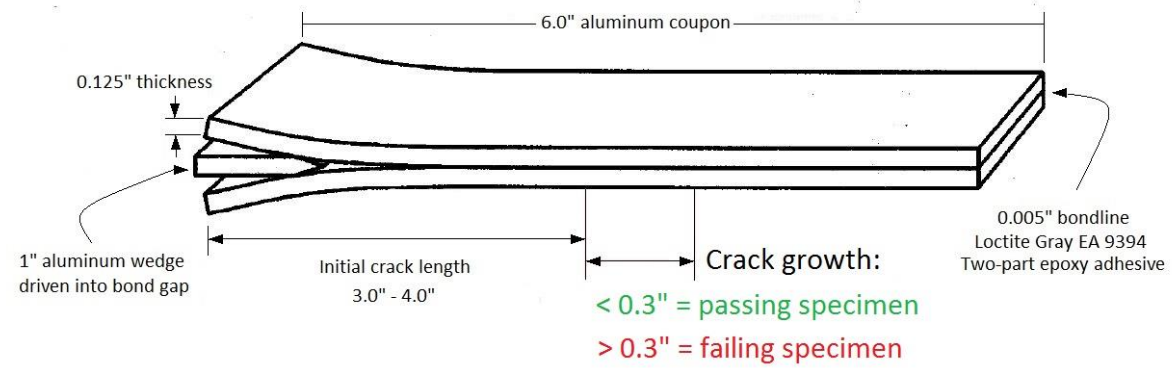

Figure 2: Diagram of a typical wedge test assembly with an aluminum wedge introducing a stress concentration and a crack from one end.

Each wedge test assembly was then sealed in a desiccator jar on a tray above the potassium sulfate solution for one hour and held at a constant 95% RH and 122 ^{o}F in a low-temp oven. Crack growth was then measured 30 minutes after humidity exposure. If the crack grew less than 0.30” from the initial measurement, the specimen passed the test. The final step of the testing consisted of breaking apart each wedge test assembly and noting the primary failure mode type – adhesive or cohesive.

3.3 Evaluating Failure Modes

After a wedge test sample had been tested, it was split open and the failure mode was recorded. The bonds between coupons were observed to fail either adhesively (Figure 3a) or cohesively (Figure 3c), though there was most commonly a mix of both failure modes (Figure 3b) along the coupon surfaces. Adhesively failed bonds manifested as regions of bare aluminum where the adhesive-to-oxide bonds had sheared off, leaving an inverse image of smooth adhesive on the opposite coupon. Cohesively failed bonds manifested as corresponding regions of rough, porous adhesive on either coupon, where the adhesive-to-adhesive bonds had failed. In this study, a sample exhibiting cohesive failure over at least 50% of its surface was considered to have cohesive failure, otherwise it was considered to have adhesive failure.

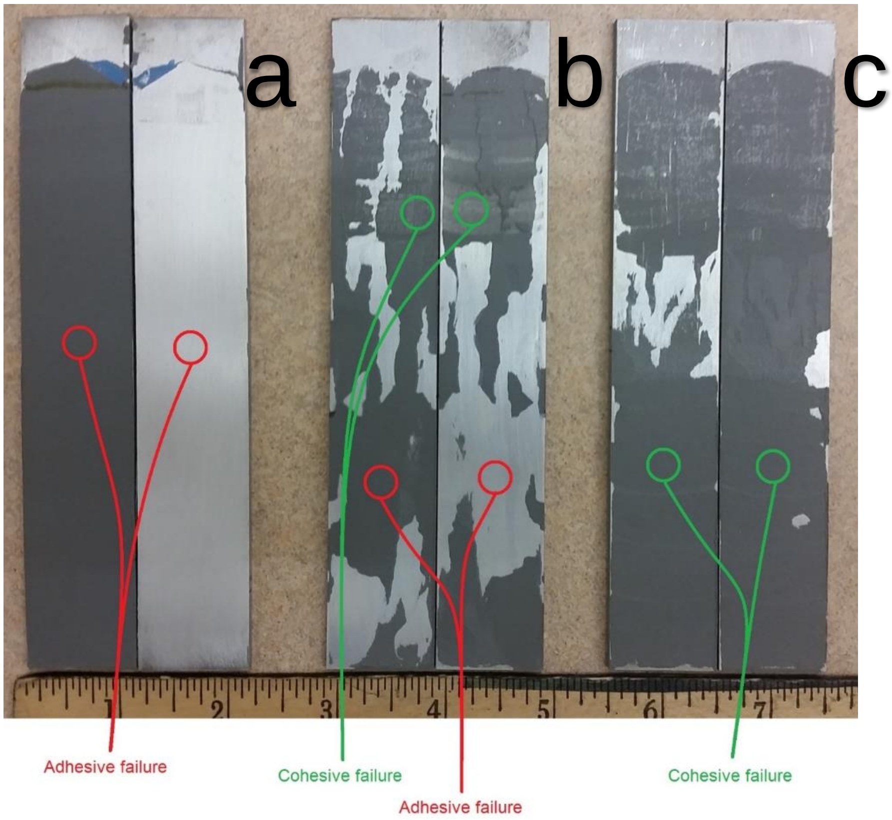

Figure 3: (a) Non-etched control 2024 sample exhibiting complete adhesive failure. (b) 24-hour outlife 2024 sample exhibiting primarily adhesive failure and spots of cohesive failure. (c) 6-hour outlife 2024 sample with nearly complete cohesive failure.

4. Results

4.1 Comparing P2-Etched and Non-Etched 7075

A control group consisting of two non-etched samples was tested. These specimens were prepared exactly the same as the etched specimens except that no etch was applied after scrubbing or before adhesive bonding. Both failed the wedge test with total failure. In a total failure specimen, the crack that forms initially due to the wedge propagates through the entire sample by the end of the test, leaving two debonded coupons. The etched 7075 samples fared significantly better than the non-etched samples. Only 2 out of the initial 21 etched 7075 specimens exhibited total failure.

In addition to crack growth, the primary failure mode of each 7075 specimen was evaluated. Both non-etched specimens exhibited mostly adhesive failure. Figure 4 shows an exceptional non-etched sample in which the part failed 100% adhesively. This specimen demonstrates the most undesirable result of an adhesively-bonded part.

Figure 4: Non-etched 7075 coupons after wedge testing show that epoxy only remains adhered to one face instead of being split between both faces.

Though the replications were limited, the results seemed to show that etching improves environmental durability and the presence of good adhesive-to-oxide bonds dramatically. Due to limited testing supplies, a control group was not repeated for 2024.

4.2 Crack Growth Measurements for 2024 Specimens

Due to material shortages and time constraints, 2024 specimens were only tested to outlife times of one week. Each outlife time tested consisted of three replications using the same methodology. The three crack growths of each outlife time were averaged and plotted with one standard deviation shown on either side (Figure 5). It is important to note that only two crack growth measurements could be made for the 1-hour and 24-hour outlife times. Two specimens exhibiting total failure during the wedge test could not be plotted or averaged with the rest of the group.

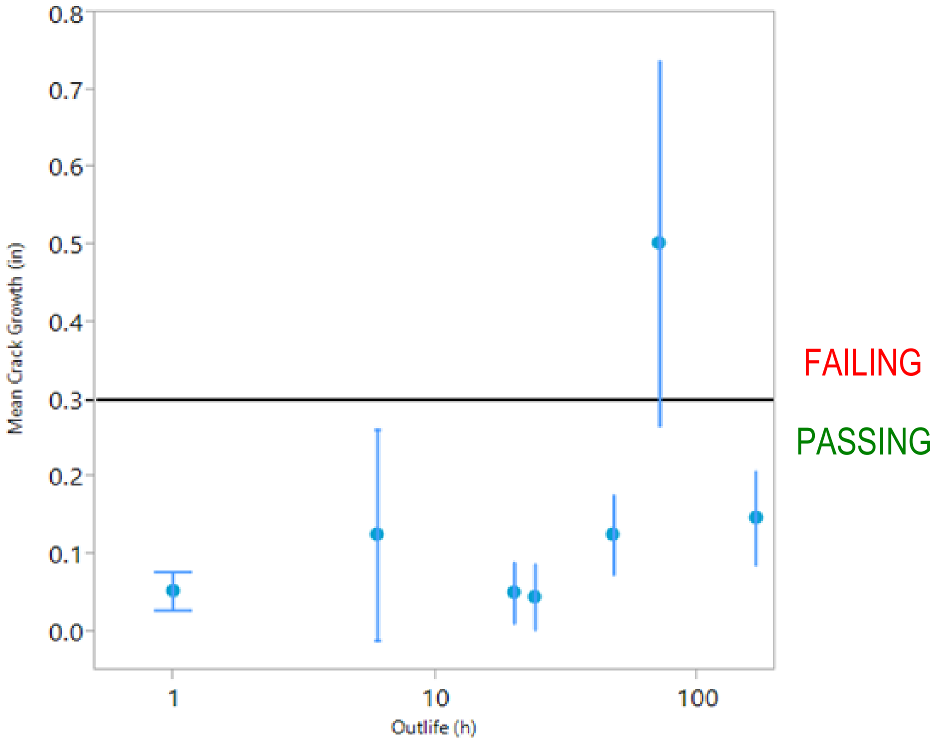

Figure 5: Graph of the crack growths at each outlife time for 2024 averaged with bars extending one standard deviation in either direction.

Even with two total failures, the 2024 samples had an 81% passing rate and a 48% cohesive failure rate overall. The three replications for one week, the longest outlife time tested, all passed the wedge test. On the other hand, the three 72-hour specimens failed or nearly failed the test. Perhaps these coupons were not stored effectively to prevent surface contamination between etching and bonding, or natural variation and few replications is to blame. Further testing of the same and higher outlife times with more replications is required to verify these results.

4.3 Crack Growth Measurements for 7075 Specimens

4.3.1 Initial 7075 Results

The initial round of 7075 testing included a total sample size of 21 specimens evenly distributed across seven outlife times from 1 hour to 4 weeks. Crack growth results were graphed with a line separating passing from failing samples (Figure 6). Only 19% of samples were considered passing, while only one (5%) of the samples failed cohesively. Two of the failing samples not shown in the graph exhibited total failure and the few that did pass were only accepted by a narrow margin.

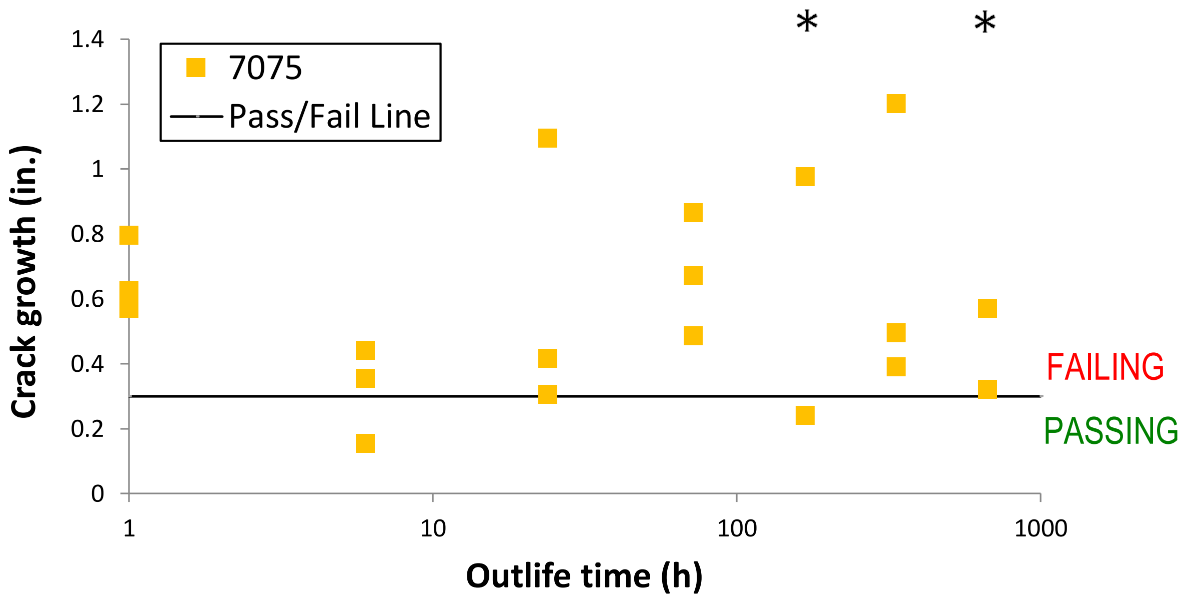

Figure 6: Graph of crack growth vs. outlife time of 7075 specimens showing mostly failed samples and high variation. *Non-passing samples that failed completely and for which exact crack growths could not be measured.

The poor environmental durability performance reflected by this data was attributed mostly to the manufacturing method used to craft the coupons. Shearing each 7075 coupon from a large sheet caused bowing across the coupon lengths which may have prevented the coupons from adhering flatly to one another and added stress in the bondline that caused cracks to propagate further. Hence, testing of the P2 etchant's capabilities were considered invalid and the results were discounted from the main analysis.

4.3.2 Water-Jet Cut 7075 Results

In an attempt to gather more reliable data, a new sheet of 7075 aluminum was obtained and instead water-jet cut into wedge test coupons to achieve a flatness similar to that of the 2024 coupons. Only eight specimens were tested, but results showed a higher passing rate (50% vs. 19%) and a higher cohesive failure rate (13% vs. 5%). This indicates that reducing curvature in the 7075 specimens improved their performance in the wedge test. However, the improved results were still much lower than those of the 2024 specimens (Figure 7). Since the same preparation was used on the water-jet cut 7075 samples and all 2024 samples, a difference in the alloys themselves seems to be responsible for the discrepancies in the wedge test results.

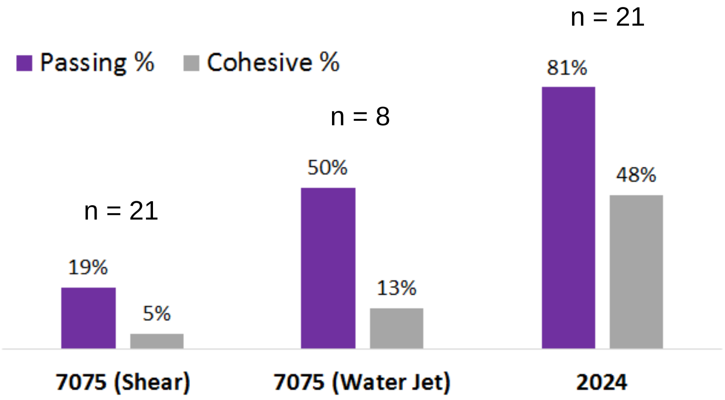

Figure 7: Bar chart showing that non-sheared 2024 samples performed far better than both sets of 7075 samples and that water-jet cut 7075 samples performed better than sheared 7075 samples.

4.4 Comparing 2024 and 7075 Results

As seen in Figure 8, the 2024 specimens exhibited much greater environmental durability than 7075 specimens overall and based on their crack growths at comparable outlife times. Even with an improved manufacturing method for 7075, the 2024 still showed more favorable results.

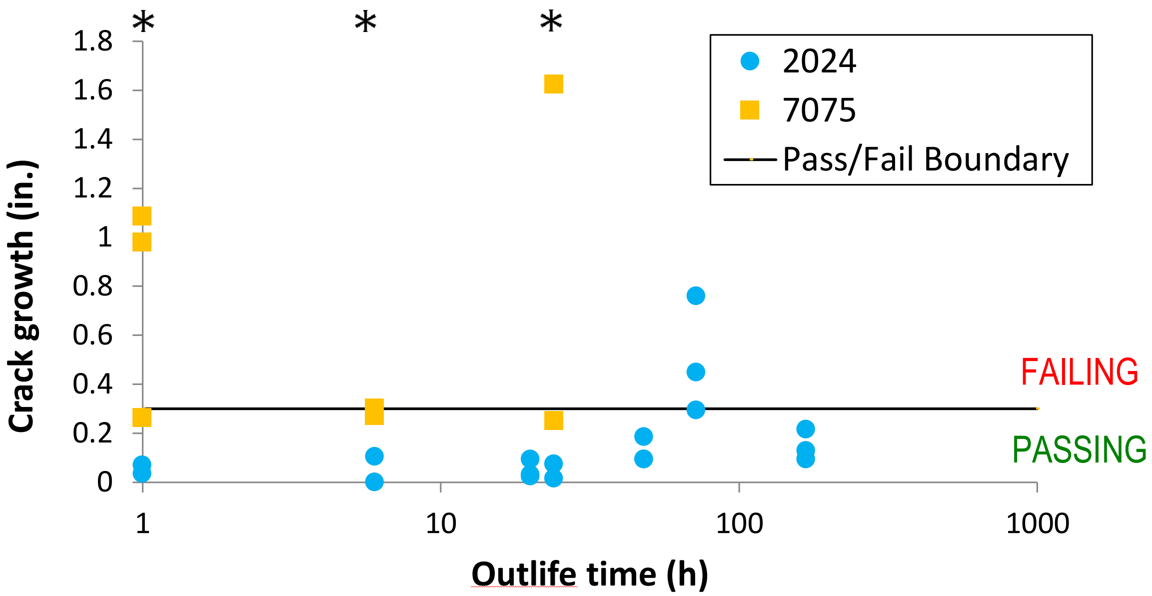

Figure 8: Graph of crack growth vs. outlife time of both 2024 and water-jet cut 7075, with 2024 exhibiting better environmental durability and more consistent results. *Total failure samples.

The difference in passing rates between the two alloys was also reflected in the failure modes, with 2024 having a higher percentage of the desired cohesive failure. However, the rate of cohesive failure for 2024 (48%) did not match the rate of passing wedge specimens (81%), indicating that preparation methods could be further improved. All of the cohesively failing specimens had crack growths of less than 0.30”, while the adhesively failing counterparts had much higher, more variable crack growths (Figure 9).

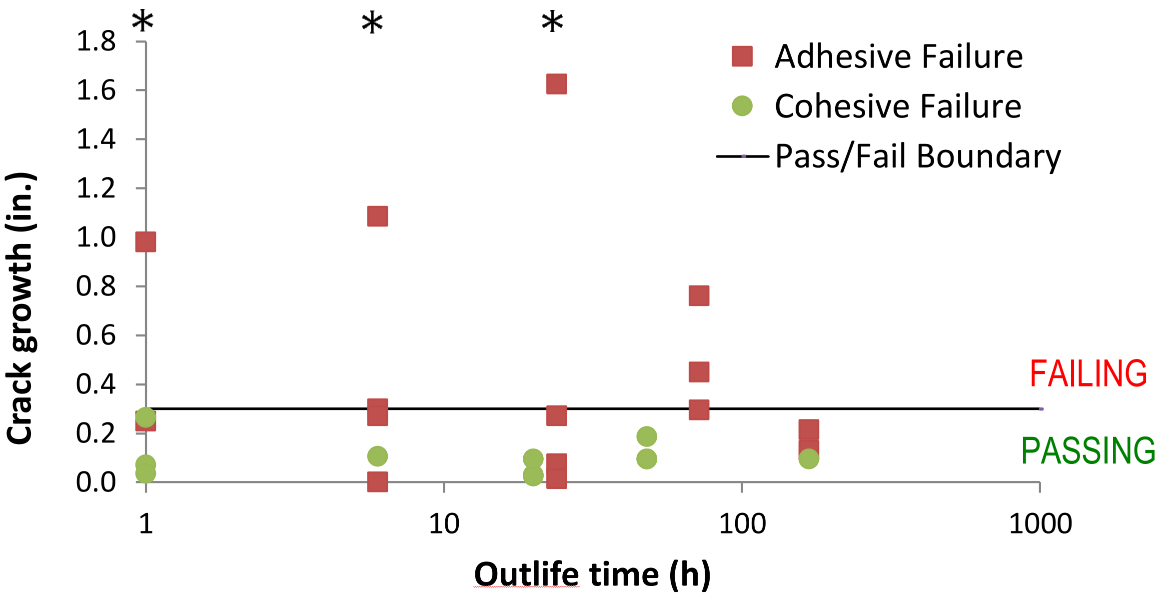

Figure 9: Graph of crack growth vs. outlife time, colored to indicate failure mode. *Total failure samples.

One possible reason for the incongruity may come from the chemical makeup of the alloys. The P2 etchant primarily contains ferric sulfate and sulfuric acid. The sulfuric acid attacks the entire aluminum surface and would, if not for the addition of ferric ions, simply dissolve the aluminum. The ferric sulfate helps form the ideal oxide layer by concentrating its attack on the copper to form pits and by slowing down the effect that the sulfuric acid has on the entirety of the aluminum surface [10]. Because the 7075 lacks the same high concentration of copper found in 2024, it is believed that the P2 etchant was not as potent with the former alloy.

Another possible cause of the discrepancies in results could be natural morphological differences in how the oxide layers of the two alloys form. To investigate such a phenomenon would require precise AFM imaging of both alloys before and after etching. Lastly, the fact that 7075 has a significantly higher yield strength (503 MPa vs. 324 MPa) might result in higher stress concentration during wedge testing, explaining the higher crack growths. The wedge test states that etch times and passing criteria may need to be adjusted on a case-by-case basis with different alloys. Based on a high adhesive failure rate observed, it may be the case that a proper wedge test for 7075 requires a longer etch time with the less aggressive paste etch to effectively prepare the surface oxide for bonding.

4.5 AFM Imaging

In order to further the study comparing P2 and FPL etchants with lap shear testing [13], scanning electron microscopy and atomic force microscopy were used to scan and image the surface of an aluminum sample that had previously been etched with P2 and another that had been etched by FPL (Figure 9). These scans were meant to characterize the oxide layers associated with durable, hydration resistant adhesive bonds and also to compare to the theoretical structures found in literature. SEM was unable to image at a high enough resolution to see any oxide structure, so only the AFM images were analyzed.

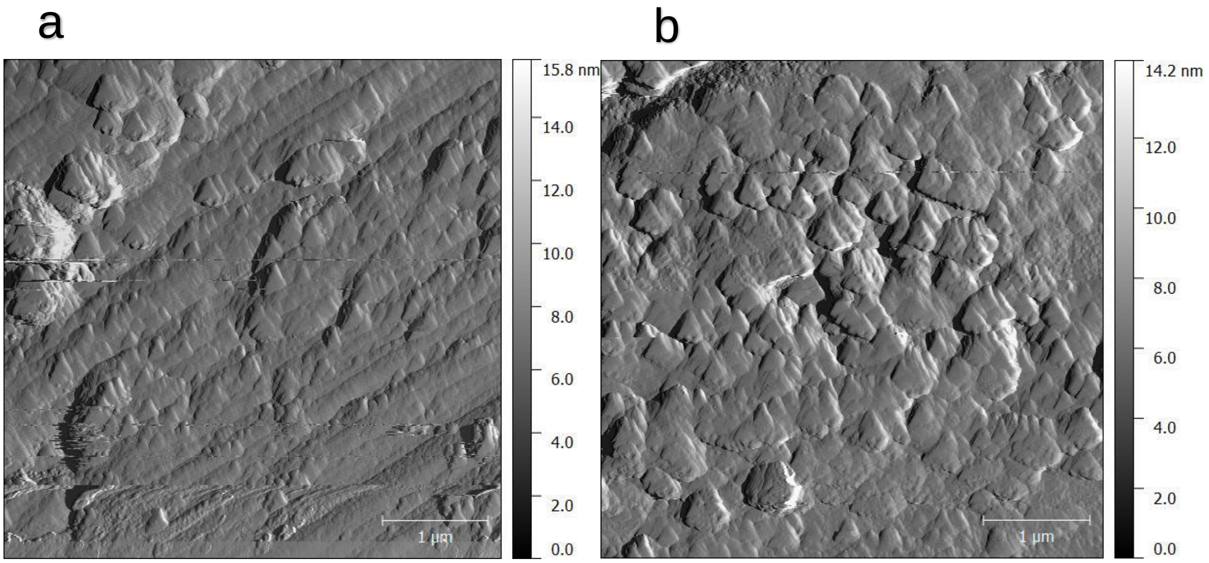

Figure 10: 4.5 x 4.5 µm AFM images of 2024 aluminum etched with (a) FPL two months prior to taking the image and (b) P2 two hours prior to taking the image.

According to the drawing in Wegman’s Surface Preparation Techniques for Adhesive Bonding, the oxide layer was expected to consist of a relatively flat surface with raised spikes regularly throughout the surface (Figure 1) [10]. The drawing was based, in part, on stereo STEM images. The rounded spikes in the drawing measured about 40 nm in height and 5 nm in diameter. Because the AFM images taken were not of high enough magnification, it was not clear whether these structures existed as they did in the drawing. However, the images did reveal a similar pattern between the two differently-etched surfaces when 4.5 µm square areas were imaged. Lines of elevated triangular regions protruded across the surfaces measuring 13-16 nm above the valleys. Additional imaging would be needed to view the nanometer-scale surface and to draw any conclusions.

5. Conclusions

The P2 etchant surface treatment increased the environmental durability of the bonds formed between the aluminum and the adhesive, compared to non-etched aluminum. While not ground-breaking, this reinforces why etchants are always used prior to adhesive bonding in every industry. The P2 etchant was effective on the 2024 alloy, producing mostly passing and cohesively failed wedge test samples with outlife times up to one week. More samples with longer outlife times would need to be tested in order to find a critical outlife time where environmental durability drops below the minimally-acceptable level. The 7075 alloy specimens exhibited longer, more variable crack growths and failed the wedge test more often than their 2024 alloy counterparts. The exact reason behind the difference is not known and requires further investigation. Lastly, cohesive failures were associated with samples passing the wedge test, which supports the literature that cohesive failure is indicative of better surface preparation, resulting in strong adhesive-to-oxide bonds that are resistant to corrosion and hydration.

6. Acknowledgements

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Appendix A: Initial Crack Length, Crack Growth, and Failure Mode for all Aluminum Wedge Test Samples

Sample	Aluminum	Outlife (h)	Initial Crack Length (in)	Crack growth (in)	Failure Mode
1*	2024	1	3.665	0.145	adhesive
2*	2024	1	3.05	0.735	adhesive
3*	2024	1	3.145	0	adhesive
4	2024	20	3.365	0.03	cohesive
5	2024	20	3.24	0.025	cohesive
6	2024	20	3.155	0.095	cohesive
7	2024	48	3.325	0.095	cohesive
8	2024	48	3.89	0.095	cohesive
9	2024	48	3.16	0.185	cohesive
10	7075	control	4.965	total failure	adhesive
11	7075	control	5.08	total failure	adhesive
12	7075	1	4.525	0.57	adhesive
13	7075	1	3.8	0.795	adhesive
14	7075	1	4.195	0.625	adhesive
15	7075	6	3.26	0.44	cohesive
16	7075	6	3.42	0.355	adhesive
17	7075	6	3.185	0.155	adhesive
18	7075	24	3.815	0.415	adhesive
19	7075	24	3.99	1.095	adhesive
20	7075	24	3.16	0.305	adhesive
21	7075	72	3.535	0.67	adhesive
22	7075	72	3.835	0.485	adhesive
23	7075	72	3.935	0.865	adhesive
24	7075	168	3.78	total failure	adhesive
25	7075	168	3.385	0.975	adhesive
26	7075	168	3.345	0.24	adhesive
27	7075	336	3.43	0.39	adhesive
28	7075	336	3.15	1.2	adhesive
29	7075	336	3.245	0.495	adhesive
30	7075	672	4.94	total failure	adhesive
31	7075	672	3.5	0.57	adhesive
32	7075	672	3.35	0.32	adhesive
33	7075	1	4.20	0.98	adhesive
34	7075	6	3.14	1.085	adhesive
35	7075	24	3.705	0.27	adhesive

*High ratio of glass beads invalidated results.

36	7075	1	4.175	0.25	adhesive
37	7075	1	2.975	0.265	cohesive
38	7075	6	4.815	total failure	adhesive
39	7075	6	3.73	0.30	adhesive
40	7075	24	4.26	1.63	adhesive

41	2024	1	2.96	0.07	cohesive
42	2024	1	2.225	total failure	adhesive
43	2024	1	3.15	0.035	cohesive
44	2024	6	3.48	0.27	adhesive
45	2024	6	4.045	0	adhesive
46	2024	6	2.935	0.105	cohesive
47	2024	24	3.31	0.075	adhesive
48	2024	24	3.13	0.015	adhesive
49	2024	24	total failure	total failure	adhesive
50	2024	72	3.375	0.76	adhesive
51	2024	72	3.555	0.295	adhesive
52	2024	72	3.985	0.45	adhesive
53	2024	168	3.52	0.095	cohesive
54	2024	168	3.365	0.215	adhesive
55	2024	168	3.55	0.13	adhesive