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Effect of surface pre-treatment and temperature on the adhesive strength of hybrid aluminum joints

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Abstract

The strength of adhesively bonded hybrid joints is affected by various factors which include type of assembly, type of adherend, operating temperature, surface pre-treatment and others. In this study, the effects of pre-treatment and operating temperature on bonding strength are investigated experimentally. The experiments are carried out under mechanical abrasion, FPL and P2 etching conditions at different test temperatures of –20 °C, 0 °C, 20 °C, 50 °C. Three type rivet arrangements are tested in order to see an effect of rivet reinforcement. Test results showed that FPL and P2 etching improved the strength of the joint according to mechanical abrasion. The strength of the joints did not change significantly depending on temperature and humidity for mechanical abrasion, but the acid etched joints are affected by temperature and humidity, especially P2 etched joints presented lower strength at 50 °C and 95 % Rh. The reinforcement of the adhesive joint with rivets did not remarkably affect the joint strength.

Key words: adhesive joints, hybrid joints, surface treatments, temperature effects

1. Introduction

Adhesive bonding is one of the most commonly used joining techniques in advanced structures (automotive, nautical, aerospace, aircraft application, etc.). This is due to several advantages over other methods including welding, riveting and bolting. Compared with these methods, adhesive bonding can provide the following distinct advantages: more uniform stress concentration, load distribution over wider areas, lighter structures, the ability to join different materials, improved fatigue performance, high stiffness and no heat effects on adherends. However, adhesive bonding has several disadvantages as well. Adhesives are sensitive to environmental changes and their performance may degrade over time as they are subjected to varying moisture and temperature conditions [1, 2]. An important aspect in the use of adhesive joints in structural applications is the ability to predict their performance during the design stage. It is extremely important to include environmental factors, such as moisture and temperature in any predictions, as they may significantly decrease the joint strength over time [3]. Therefore, the combination of adhesive bonding with other joining techniques may be a viable solution for a design that must address a specific combination of the constraints mentioned above [4]. For example, if there is a failure due to environmental factors, rivets can protect structural integrity.

The two major factors influencing the strength of adhesive joints are temperature and moisture. The effect of temperature can be observed in the form of variations in the mechanical properties of an adhesive [5]. Moisture affects nearly all adhesive applications because water is pervasive in the atmosphere, readily absorbed and aggressive towards the displacement of physical bonding. Thus, the durability of adhesive joints in the presence of environmental moisture has become the main challenge for researchers in this area.

Kachlik and Klement [6] studied the properties of rivet-bonded joints using a polyurethane adhesive and blind rivets under the influence of aging. The results showed that in case of cyclic heating, the stiffness of joints decreased by 38 % and the peel strength had a decreasing trend upon exposition to selected environments, with degradation reaching 30 %.

Banea and Silva [7] investigated bulk specimen and adhesive joint tests with two different adhesive types

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(epoxy and polyurethane), under different temperatures. Results showed that the failure loads of both the bulk test and joint test specimens varied with temperature and needed to be considered in any design procedure.

Doyle and Pethrick [8] reported dielectric and mechanical analysis of aluminum-epoxy bonded adhesives joints exposed to de-ionized water, aqueous urea solution and salt water at 65 ℃. The results showed that in the case of the aqueous urea solution, passivation of the oxide by the urea reduced the rate of corrosion. The non-polar media aviation fuel and hydraulic fluid are able to plasticize the adhesive and there is a consequent reduction in the strength of the joint.

Keller et al. [9] investigated the fatigue response of adhesively-bonded pultruded GFRP double-lap joints under different environmental conditions. They concluded that the environment had a considerable effect on the fatigue behavior of the examined joints. They also concluded that increased temperature seems to shorten specimen fatigue life. An increase in temperature was found to provoke higher stiffness degradation, aggravated by the addition of humidity.

Proper preparation of an adherend surface is one of the most important factors in assuring adequate joint strength and durability of high-performance adhesive joints. The interface or interphase between the adhesive and the adherend is critical to stress transfer. The goal of a surface treatment is to form a strong and stable interface or interphase that is stronger and more durable than the adhesive being used, such that bond failure is cohesive within the adhesive, both initially and throughout the joint's service lifetime [10].

Harris and Beevers [11] used different types of grit blasting material and grit size to treat the mild steel and aluminum alloy substrates. The results showed that the initial dry strength was relatively independent on grit size in lap shear joints and showed 100 % interfacial failure for all cases. However, the butt joint showed increasing interface failure from 30 % to 70 % by area after immersion in de-ionized water at 60 ℃ for 12 weeks.

Knox and Cowling [12] applied the silanes (A187 and SiP) and the corrosion inhibitors (Albritec and Accomet-C) as surface pre-treatments on thick adherend lap shear joints, as well as strap joints aged in 100 % RH at 30 ℃ to obtain difference of surface pre-treatments. They concluded that the silane primers increased the durability performance of the joint more than the corrosion inhibitors. The reason for this result is that the application of primer on well prepared surfaces increases the stability of the adhesive and adherend interface against the diffusion of water.

Bowditch [13] applied surface pre-treatment to aluminum alloy and tested it after exposure to water immersion. The 50 ℃/water environment did not cause any noticeable effect on surface treatment. However, the phosphoric acid anodizing process showed superior durability at 40 ℃. The failure occurred near the interface at low temperatures. At higher temperature cohesive failures occurred because of the degradation in the adhesive.

Miranda et al. [14] examined the influences of two pre-treatments on the mechanical behavior of adhesive bonded 2024-T3 aluminum alloy joints, before and after aging by water immersion. Alkaline etching and acid pickling treatments were applied singly, or in combination, with phosphoric acid anodizing. The durability of the bonded joints was shown to depend on the thickness of the oxide film morphology. In high humidity environments the shear strength of single lap joints decreased.

Prolongo and Urena [15] investigated the effect of pre-treatment on the adhesive strength of epoxy-aluminum joints. A1050 and A2024 aluminum alloys were used to see effects of alloying elements. The sulphuric acid-ferric sulphate etches showed high joint strength according to dichromate-sulphuric acid etching, alkaline etching, or mechanical abrasion. The joints with Al-Cu-Mg alloy substrates (A2024) generally presented higher adhesive strength values than those with pure aluminum (A1050) adherends, due to the selective etching of some allowing elements and intermetallic compounds, which have different electrochemical potential.

Zuo et al. [16] studied a new pre-treatment (phosphoric/boric/sulphuric acids anodizing) for adhesive bonding of aluminum alloys. The results showed that through the process of phosphoric/boric/sulphuric acids anodizing, a thicker film with high porosity and large pores can be obtained. In films by boric/sulphuric acids anodizing and phosphoric acid anodizing under humid and hot environments, the phosphoric/boric/sulphuric acids anodic film showed higher bonding strength and durability.

Borsellino et al. [17] investigated effects of the substrate surface condition and the adhesive properties on single-lap aluminum joint. The results showed that roughness had a different effect on the wettability of each kind of resin. In spite of their wettability characteristics, the high viscosity resin joint is the most resistant while the polyester joints are poorer. This is due to the intrinsic strength of the resin joint adhesive, the effect of mixing with catalyst and the enhanced stability of aluminum oxide in alkaline environment.

Pirondi and Moroni [18] simulated the failure behavior of rivet-bonded and clinch-bonded hybrid joints using the FEA. The Gurson-Tvergaard-Needleman (GTN) model and ductile damage (DD) model were used to simulate damage and failure of the rivet and plates. The cohesive zone (CZ) model was employed for the adhesive layer. The authors concluded that different damage models, tuned with experiments per-

Table 1. Chemical composition of A1050 aluminum alloy (wt.%)

Fe	Si	Cu	Mn	Mg	Zn	Ti	Al
0.317	0.060	0.001	0.006	0.002	0.006	0.016	99.593

Table 2. Typical properties of aluminum A1050

Property	Value
Modulus of elasticity (MPa)	71000
Yield strength (MPa)	140
Tensile strength (MPa)	155
Poisson ratio	0.31
Density ($g\,cm^{-3}$)	2.71

Table 3. Typical dimensions and mechanical properties of rivet

$\varnothing d_1$ (mm)	$\varnothing d_2$ (mm)	L (mm)	Tensile load (N)	Shear load (N)
4.8	9.5	11	3070	2230

formed on simple joints (riveted, clinched or adhesively bonded), can be combined in a unique model to simulate effectively the failure behavior of hybrid joints.

Sadowski et al. [19] focused on the mechanical response analysis of the steel adhesive double lap joints reinforced by rivet. Riveted, adhesively bonded and hybrid joints were investigated both experimentally and numerically. The results showed that, although the adhesive joint was very strong, the application of an additional rivet leads to a very significant energy absorption by about 35 % in comparison to simple adhesive double lap joints.

Using the ABAQUS FEA program, Sadowski et al. [20] analyzed the damage and failure behavior of the hybrid joint reinforced by five rivets. Both the experimental and numerical results showed that the tensile strength of the hybrid joint was higher than the adhesive bonded joint or the joint with five rivets. Addition of the rivets to the adhesive bonded joint increases the energy absorption during the failure process in comparison to the riveted or the adhesive bonded joints.

As summarized above, the previous studies showed that there was not a clear relationship between hybrid structure and joint strength. The aim of this work is to clarify the influence of the surface pre-treatment and temperature on the joint strength of aluminum lap joints under static loading conditions.

2. Experimental study

2.1. Material

Test specimens of $40 \times 100\,mm^2$ are cut from a 3 mm thick sheet of A1050 aluminum alloy. The chemical composition of specimens is given in Table 1. Typical properties of aluminum A1050 are presented in Table 2.

The blind rivets and epoxy adhesives are used to join aluminum sheets. The body of rivet is made of aluminum alloy and the mandrel is made of zinc plated steel. The geometry of the rivet is shown in Fig. 1. Typical dimensions and mechanical properties of rivet are presented in Table 3.

Loctite Hysol 9466 adhesive is applied on the adherend surface. This is a two component epoxy adhesive which cures at room temperature after mixing. It provides excellent bond strengths for a wide variety of plastics and metals. The physical properties of adhesive are presented in Table 4.

2.2. Surface treatment

Three different surface treatment methods were applied prior to pure adhesive and hybrid bonding of the lap shear test specimens. For the first treatment, all of the specimens were abraded with the same mesh size sandpaper (P220C) and cleaned with Loctite 7063 cleaner. For the second treatment, the specimens were immersed for approximately 12–15 min in prepared optimized FPL solution at 66–71 °C after the abrasion and cleaning. For the third treatment, abraded and cleaned specimens were immersed for approximately 10–12 min in prepared P2 solution at 60–65 °C. After immersion step for each treatment, all samples were rinsed in deionized water for 1–3 min and dried in an

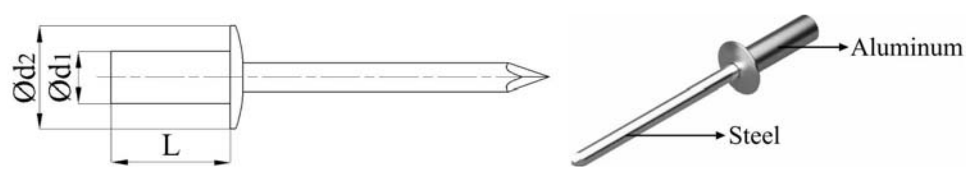

Fig. 1. Blind rivet geometry.

T a b l e 4. Typical properties of Hysol 9466 [21]

Property	Value
Bulk modulus (ASTM D882) (GPa)	1.718
Elongation (ASTM D882) (%)	3
Tensile strength (ASTM D882) (MPa)	32
Average shear strength (ASTM D1002-94) (MPa)	26 (aluminum)
Glass transition temperature (ASTM E1640-99) (℃)	62

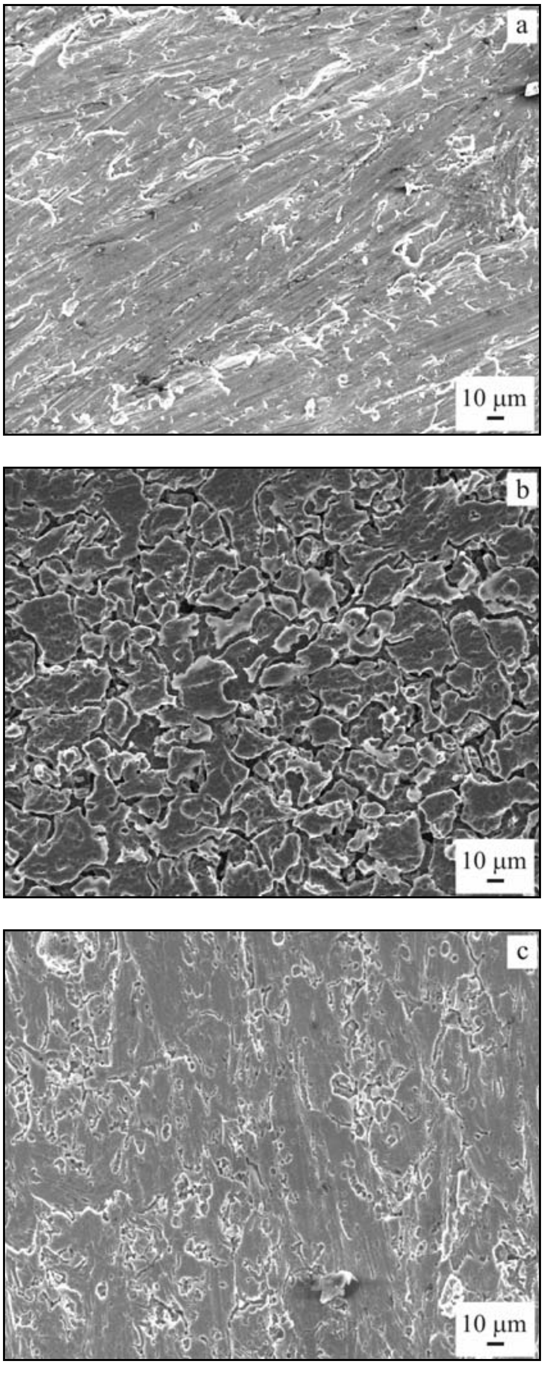

Fig. 2. Scanning electron micrographs of substrates after surface treatment: a) abrasion, b) FPL etching, c) P2 etching.

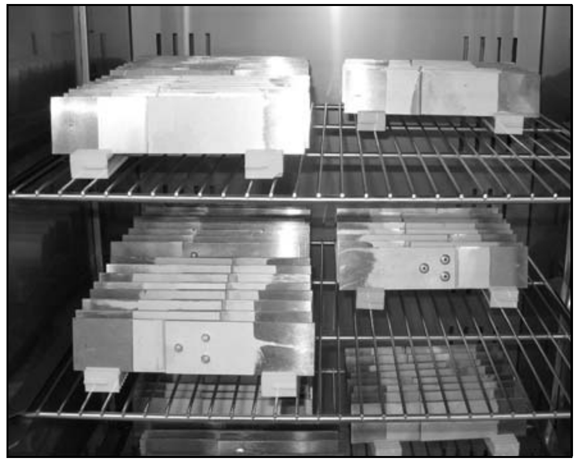

Fig. 3. The joints in climatic cabinet, at 50 ℃ and 95 % Rh.

oven at approximately 50 ℃. Both treatments, FPL and P2, were prepared according to ASTM D2651-01 [22]. The concentration of solution for each treatment is given in Table 5. Scanning electron micrographs of substrates after surface treatment are presented in Fig. 2. The different surface topographies were obtained by etching. These surfaces have more uniform roughness than those obtained through abrasion.

2.3. Temperature effect and aging

Temperature and humidity are two major factors that affect the strength of adhesive joints. In order to observe this effect, experiments were carried out at different temperatures and humidity levels. The joints were exposed to four temperatures (–20 ℃, 0 ℃, 20 ℃, 50 ℃) prior to testing. Deep freeze was used to achieve –20 ℃ and 0 ℃. According to the standard EN 2243-5 [23] addresses aging test for structural adhesives, the specimens were exposed to 50 ℃ and 95 % Rh in climatic cabinet for 30, 60, 90 days. The cabinet simulated real environmental conditions by controlling temperature and humidity with day and night cycles. The working temperature range of cabinet was –10 ℃/60 ℃ and the humidity set range was 10 %/95 % Rh. The joints in the climatic cabinet are presented in Fig. 3.

T a b l e 5. Composition of FPL and P2 etching solution [22]

Solution component	Concentration and condition	
	FPL etch	P2 etch
Sulphuric acid	287.9–310 g l^{-1}	27–36 % by weight
Sodium dichromate	28–67.3 g l^{-1}	–
Aluminum alloy-2024 bare	1.5 g l^{-1}, min	–
Ferric sulphate	–	135–165 g l^{-1}
Deionized water	until it receives	until it receives
Temperature	66–71 °C	60–65 °C
Immersion time	12–15 min	10–12 min

T a b l e 6. The summarization of experimental conditions

Joint type	Surface treatment	Test condition					
		–20 °C	0 °C	20 °C	50 °C ± 3 °C, 95–100 % Rh		
					30 days	60 days	90 days
Pure adhesive joint	Abraded	11.75*	10.85	11.91	12.17	10.56	12.42
	FPL etch	16.21	15.65	15.01	13.87	14.14	14.14
	P2 etch	16.96	15.73	15.55	12.13	11.15	12.50
Hybrid joint with 2 rivets	Abraded	12.38	12.11	13.05	11.26	10.88	12.92
	FPL etch	15.37	15.06	14.88	13.32	14.03	12.73
	P2 etch	15.21	15.68	15.17	13.15	12.18	12.85
Hybrid joint with 3 rivets	Abraded	13.33	12.24	12.72	10.61	–	–
	FPL etch	15.73	14.01	14.75	10.54	–	–
	P2 etch	14.88	15.00	13.5	10.83	–	–
Hybrid joint with 4 rivets	Abraded	13.77	13.39	12.95	11.04	–	–
	FPL etch	14.94	14.73	14.00	9.93	–	–
	P2 etch	15.33	14.47	13.40	11.51	–	–

*Mean values of test results for 4 specimens (MPa)

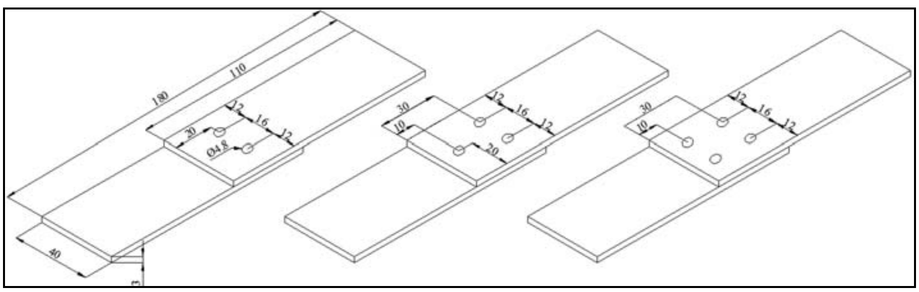

Fig. 4. The rivets layout.

2.4. Joining process

Pure adhesive joints and hybrid joints were tested in this experimental study. Three types of rivet arrangements were tested due to limited test specimen area for hybrid joints. The arrangements of rivets are given in Fig. 4.

Single lap joints were bonded with Loctite Hysol 9466 epoxy adhesive. The adhesive was applied on one substrate for pure adhesive and hybrid joints after the surface treatment. The specimens were riveted immediately after applying the adhesive for hybrid joints. In order to completely cure the adhesive, specimens were left 7 days at room temperature. After this time,

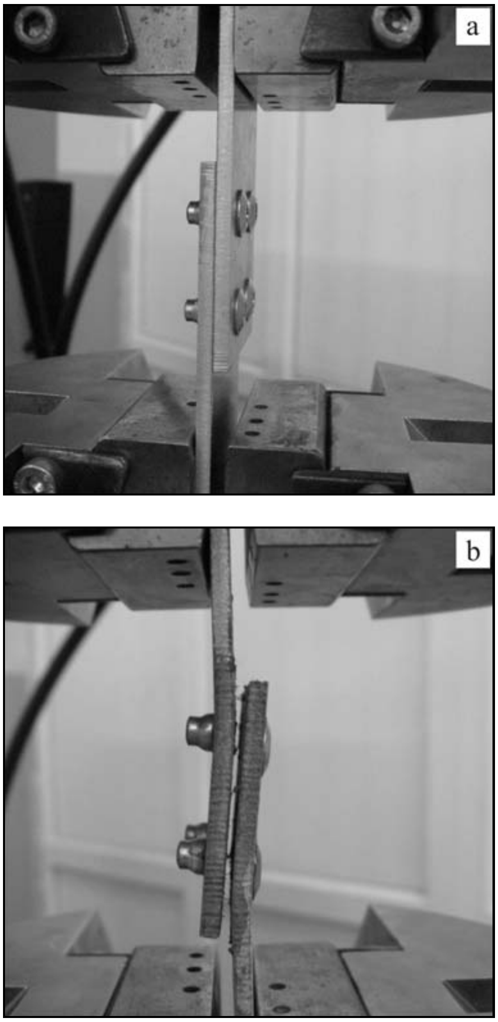

Fig. 5. Loaded specimens under tension loading: a) before failure, b) after failure.

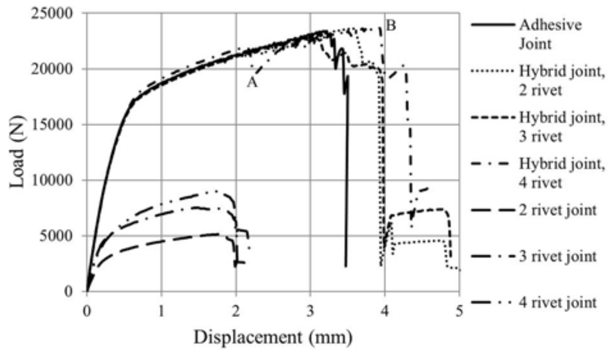

Fig. 6. Load-displacement curves for the riveted joints, adhesive joint and the hybrid joints at 20 ℃.

a part of pure adhesive and hybrid joints were left 30 days in deep freeze for –20 ℃ and 0 ℃, and the other part of the specimens were exposed to 50 ℃ and 95 % Rh in climatic cabinet for 30, 60, 90 days for aging tests. At the end of these periods, 4 specimens were tested for each condition. The experimental conditions are summarized in Table 6.

2.5. Tension tests

The specimens with both joined adhesive and rivets were loaded in tension using an Instron servo-hydraulic testing machine at a crosshead displacement rate of 2 mm min^{-1}. Figure 5 shows the experimental setup and loaded specimen.

3. Results

The experimental data were analyzed and compared with each other. Figure 6 shows the results of the tests at 20 ℃. All the hybrid specimens showed the similar behavior under all conditions. After the initial linear ramp, the specimens achieved the maximum load at a displacement of approximately 4 mm. After that point, a sudden decrease in load occurred due to failure of the adhesive. After releasing the adhesive load handling, the rivets continued to bear load up to final rupture.

The reinforcement of the adhesive joint with rivets did not remarkably affect the joint strength. Even the addition of rivets caused a decrease of the joint strength according to pure adhesive joint. Deterioration of the plane surface on bonding area with the addition of rivets and the notch effect caused by rivet holes could be the cause of the decrease in joint strength.

The test specimens were exposed to a little bending stress after the application of shear load during the tension test. The damage (crack) began at the end portion of the joint and the crack continued along the surface unimpeded until the plane surface which was caused by the rivets due to the bending. The progression of damage was interrupted by the rivets. So that, the reduction of joint strength at *A* point, which started with damage, increased to some extent again at point *B* (Fig. 6). This effect of the rivets on the joint can be seen as an advantage to extend ultimate damage time and displacement at the end of the final damage. This situation had been observed especially in the joints which were reinforced with 4 rivets. In addition, the joints were divided into two parts after the failure at the pure adhesive joint, structural integrity was impaired. With reinforcement of joint using rivets, the rivets continued to bear load after failure of the adhesive. For this reason the structural integrity remained intact after failure. This outcome can be considered to be an advantage in terms of safety.

We applied mechanical abrasion, FPL and P2 etching on the substrates to remove either weak adhering or contaminated on the surfaces, so that, the freshly

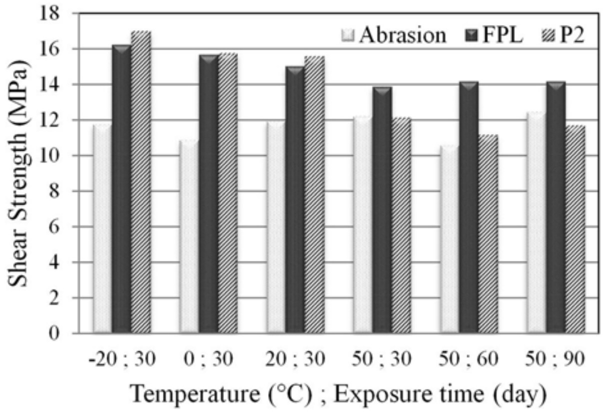

Fig. 7. Shear strength of adhesive joint as a function of temperature and applied surface treatment.

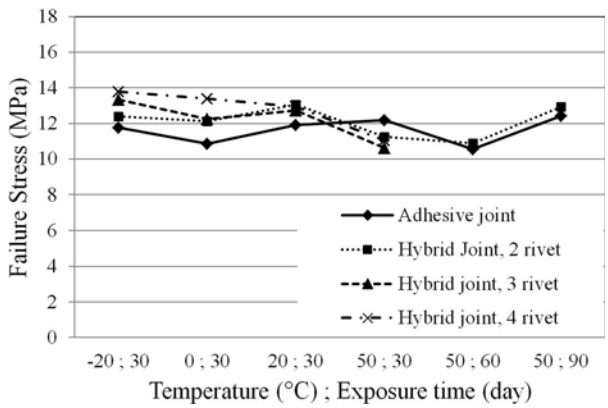

Fig. 8. Failure stress of abraded adhesive joint and hybrid joints as a function of temperature.

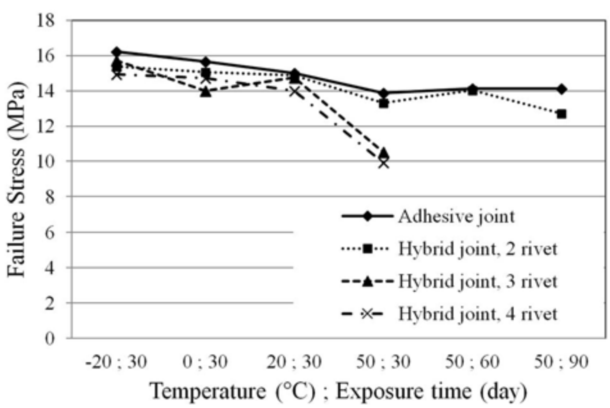

Fig. 9. Failure stress of FPL etched adhesive joint and hybrid joints as a function of temperature.

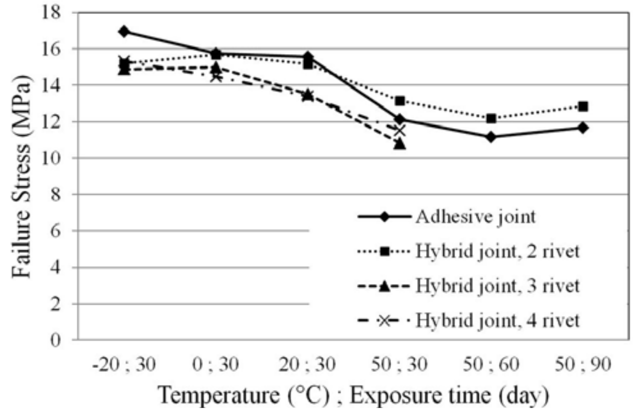

Fig. 10. Failure stress of P2 etched adhesive joint and hybrid joints as a function of temperature.

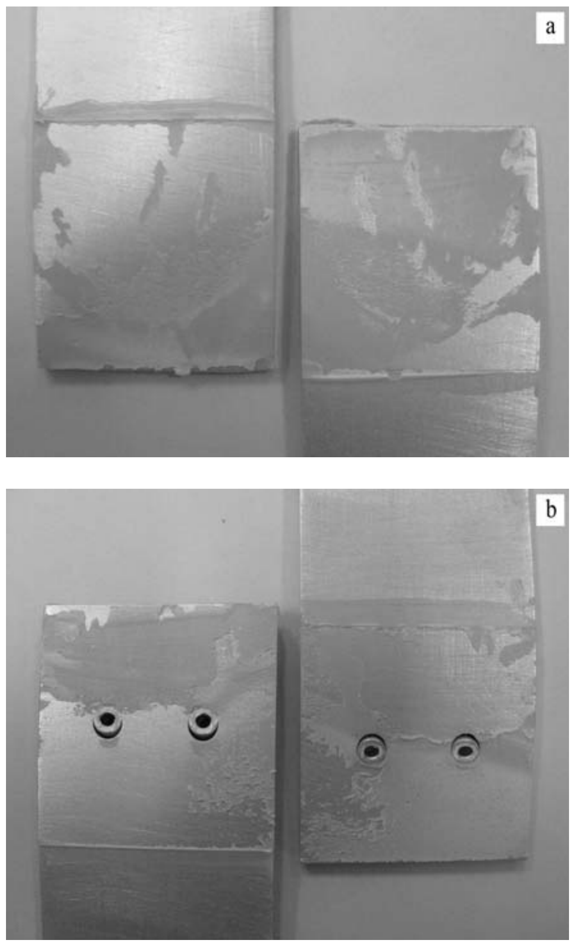

Fig. 11. Fracture surfaces: a) adhesive joint, b) hybrid joint.

oxidized layer on the substrate surface had been exposed directly to the adhesive. The results of shear strength for the adhesive joint as a function of temperature and applied surface treatment are presented in Fig. 7.

Comparing acid treatments and abrasion, acid treatments showed a higher porosity than abrasion (Fig. 2). In principle, a more porous oxide layer could

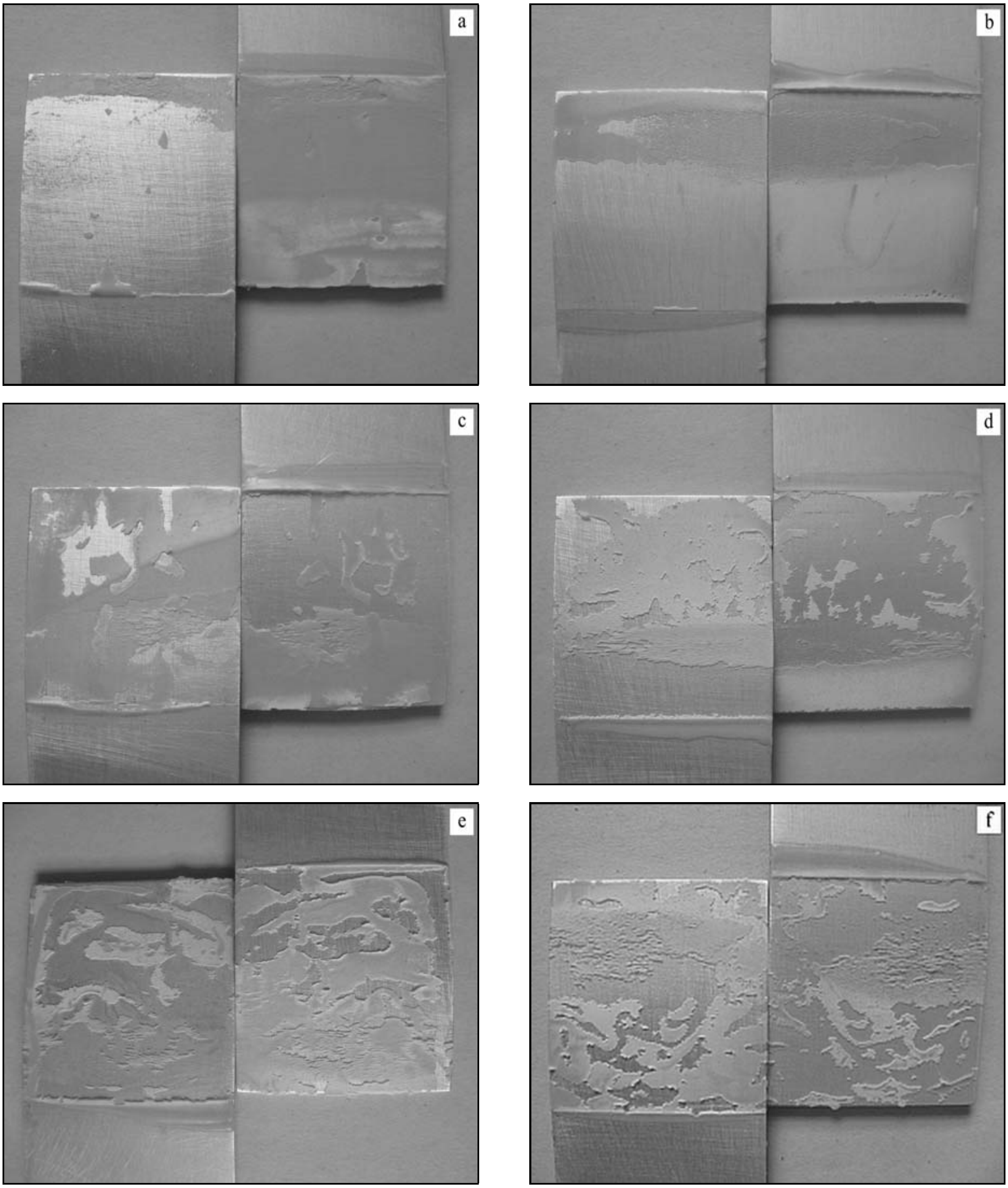

Fig. 12. Fracture surfaces of adhesive joints: a) –20 °C, b) 0 °C, c) 20 °C, d) 50 °C 30 days, e) 50 °C 60 days, f) 50 °C 90 days.

improve the contact area and provide some degree of mechanical interlocking with the adhesive. This outcome could provide a higher adhesive strength. Figure 7 shows the results of this experiment. In many cases, FPL and P2 etching improved the strength of the joint. The results showed that for the mechanical abrasion, the strength of the joints did not change due to temperature and humidity, but the acid etched joints were affected with temperature and humidity, especially P2 etched joints showed lower strength at 50 °C and 95 % Rh.

According to Fig. 7, under the 50 °C temperature, P2 and FPL provided similar results for all adhesive and hybrid joints. In most cases, the P2 treatment gave higher strength values. FPL treatment includes free hexavalent chromium which is very harmful to use, for this reason FPL can be replaced with P2 etching.

In general, the joints treated only with the abrasion method presented lower strength than the ones treated later with P2 and FPL etching. The homogeneous oxide layer, porous and well adhered, is necessary to improve the adhesive strength for the aluminum alloy.

Figures 8, 9 and 10 show the failure stress adhesive joint and hybrid joints as a function of temperature and surface treatment. The adhesive becomes more ductile with increasing temperature, for this reason the strength of the joints decreased and the failure displacement increased. When the temperature is low, this relationship becomes converse. Due to the adhesive becomes more brittle with decreasing temperature, the strength of the joints increased and the failure displacement decreased.

The fracture surfaces of the failed adhesive and hybrid joints after failure are presented in Fig. 11. Figure 11b presents the fracture surface of the hybrid joint after joint failure. The largest cohesive failure area is observed with the pure adhesive joint. The adhesive layer stuck to both surfaces. The adhesive layer at the hybrid joints is non-continuous due to joining force and rivet holes. So, the adhesive failure area is large. The body of the rivets is broken up into two parts due to shearing load because of low shear strength limit. Also, a little deformation occurred around the hole edge on the aluminum plates and all hybrid joints showed the same results. The fracture types for the hybrid joints and riveted joints are similar to each other (Fig. 11b). Meanwhile, the pure adhesive joints showed higher shear strength in comparison to the riveted joints.

The failure usually becomes cohesive failure which occurs in the adherend and adhesive interface. However, the fracture surfaces of the failure bond alter with temperature, and this alteration can be seen in Fig. 12. The failure surfaces at low temperature (Fig. 12a,b) show little adhesive deformation, indicating that the adhesive became less ductile. As the temperature increases (Fig. 12c–f), the failure surface of the adhesives shows more deformation which is a sign of higher ductility.

4. Conclusions

In this experimental study, the effect of pre--treatment and temperature on bonding strength of riveted, pure adhesive joint and hybrid joints is investigated. The following conclusions may be concluded:

1. The joints treated only with the abrasion method present lower strength than the ones treated with P2 and FPL etching. Comparing acid treatments and abrasion, acid treatments show a higher porosity than abrasion. FPL and P2 etching improved the strength of the joint.

2. The strength of the joints did not change significantly due to temperature and humidity for mechanical abrasion, but the acid etched joints were affected by temperature and humidity, especially P2 etched joints presented lower strength at 50 ℃ and 95 % Rh. Under the 50 ℃ temperature, P2 and FPL provided similar results for all adhesive and hybrid joints.

3. The reinforcement of the adhesive joint with rivets did not remarkably affect the joint strength. Even the addition of rivets caused a decrease of the joint strength according to pure adhesive joint. The rivet holes caused notch effect, so, the joint strength decreased. However, after the damage start, the progression of the damage was interrupted due to rivets.

4. Using the results of experimental studies, it can be concluded that hybrid joints are well suited to be used in automotive and aerospace industries as a joining technique. The total number of rivets may be reduced providing lighter construction and cost saving.

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