

A simple surface treatment and characterization of AA 6061 aluminum alloy surface for adhesive bonding applications

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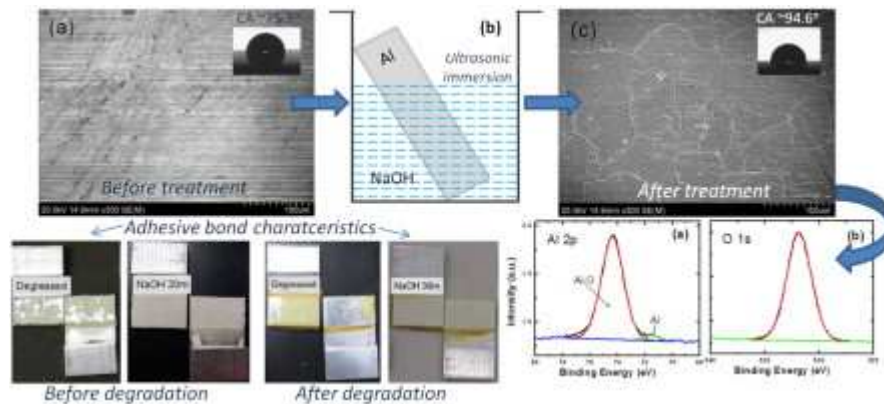
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Abstract

Structural adhesive bonding of aluminum is widely used in aircraft and automotive industries. It has been widely noted that surface preparation of aluminum surfaces prior to adhesive bonding plays a significant role in improving the strength of the adhesive bond. Surface cleanliness, surface roughness, surface wettability and surface chemistry are controlled primarily by proper surface treatment methods. In this study, we have employed a very simple technique influencing all these criteria by simply immersing aluminum substrates in a very dilute solution of sodium hydroxide (NaOH) and we have studied the effect of varying the treatment period on the adhesive bonding characteristics. A bi-component epoxy adhesive was used to join the treated surfaces and the bond strengths were evaluated via single lap shear (SLS) tests in pristine as well as degraded conditions. Surface morphology, chemistry, crystalline nature and wettability of the NaOH treated surfaces were characterized using various surface

analytical tools such as scanning electron microscopy and energy dispersive X-ray analysis (SEM/EDX), optical profilometry, infrared reflection absorption spectroscopy, X-ray photoelectron spectroscopy, X-ray diffraction and contact angle goniometry. Excellent adhesion characteristics with complete cohesive failure of the adhesive were encountered on the NaOH treated surfaces that are comparable to the benchmark treatments such as anodization, which involve use of strong acids and multiple steps of treatment procedures. The NaOH treatment reported in this work is a very simple method with the use of a very dilute solution with simple ultrasonication being sufficient to produce durable joints.

Graphical Abstract



Highlights

A very simple surface treatment method to achieve excellent and durable aluminum adhesive bonding. Our method involves simple immersion of aluminum in very dilute NaOH solution at room temperature with no involvement of strong acids or multiple procedures. Surface analysis via various surface characterization techniques showed morphological and chemical modifications favorable for obtaining highly durable bond strengths on the treated surface. Safe, economical, reproducible and simple method, easily applicable in industries.

1. Introduction

The adhesive bonding of aluminum structures is widely practiced in aircraft, automotive and marine industries due to many advantages over mechanical fastening or conventional methods such as welding, which include reduced corrosion and stress concentration, aesthetics and cost effectiveness [1], [2], [3], [4] and [5]. Adhesive

bonding offers capabilities such as large area bonding, bonding of dissimilar materials of varying thicknesses, prevention of galvanic corrosion while bonding dissimilar metals due to the insulating properties of adhesives, lighter weight than when joined with mechanical fasteners, and the use of less or no heat to create an adhesive joint eliminating any thermal distortion or residual stresses generally caused by heating [4]. However, the challenge facing industry is to find an effective, simple, safe and economical method of surface treatment leading to a good bond strength and long term durability. The most important criterion in surface preparation for adhesive bonding is that the surface must be very clean and free of organic contaminants. An initial cleaning via solvent degreasing is helpful to remove certain contaminants; however, it is also important to remove the mechanically weak thin layer of natural surface oxide and to replace it with a new uniform oxide layer in order to achieve better strength [2], [6] and [7]. The pretreatment of aluminum surfaces for adhesive joining generally comprises a surface modification by removal of the native oxide layer, altering either the chemistry of the surface or its topography. The mechanical removal of the native oxide layer via sand blasting or grit blasting is commonly employed in adhesive bonding applications. Formation of a stable oxide by anodization using phosphoric acid, sulfuric acid, chromic acid, boric acid, etc., is another standard method that is widely used to enhance the adhesive bond characteristics and improve corrosion resistance [5]. Surface wettability has been used as an indicative property by the adhesive bonding community to characterize the surface by means of water contact angle measurements. A completely wetting surface also indicates increased surface energy and the cleanliness of the surface. An overview of the surface free energy concept has been provided by Gallant and Savard [8] in the context of adhesive bonding. Another criterion that plays an important role in achieving good adhesive bond strength is that the surface must exhibit a maximum surface area in order to be able to mechanically interlock the adhesive, which is achieved by surface roughening techniques. In this work, we have utilized a simple method to remove the weak native oxide layer as well as to create a rough surface in one process by immersing the aluminum substrates in an ultrasonic bath of sodium hydroxide solution. We have investigated the adhesive bond strength on those surfaces as well as their durability under conditions of extreme humidity and temperature.

2. Material and methods

Sodium hydroxide solution of a very dilute concentration of 0.1 M was prepared by dissolving NaOH pellets in de-ionized water. Single lap shear (SLS) test coupons of AA 6061 aluminum alloy of dimensions 38 mm × 25.4 mm × 3.2 mm were acetone wiped for degreasing prior to immersion in the NaOH solution. The degreased substrates were ultrasonicated in the 0.1 M NaOH solution at room temperature for varying times of immersion, namely, 5, 30 and 60 min. These treated coupons were further rinsed ultrasonically in de-ionized water twice for 5 min to stop the reaction of NaOH with aluminum and then dried for more than 16 h in an oven at 70 °C to remove any excess water. The test samples were assembled using a bi-component epoxy adhesive to evaluate the adhesive bond strength via single lap shear tests.

The treated surfaces were characterized for microstructural and chemical analyses using various surface analytical techniques. Hitachi SU-70 field emission scanning electron microscopy with energy dispersive X-ray spectroscopy (FESEM/EDX) was used to study the morphological modifications as well as to perform elemental analyses of the NaOH treated surfaces. The root mean square (rms) roughness of the resulting surfaces was measured using an AD phase shift optical profilometer. The X-ray diffraction (XRD) analyses of the prepared surfaces were carried out using a Bruker D8 Discover system to investigate the crystalline properties. Infrared reflection absorption spectroscopy (IRRAS) and X-ray photoelectron spectroscopy (XPS) were employed to characterize the surface chemistry of the resulting surfaces. The IRRAS spectrometer (Nicolet 6700 FT-IR) is equipped with a Mid-IR MCT-A N₂-cooled detector and a KBr beam splitter. The Smart SAGA (specular apertured grazing angle) accessory was used to analyze samples at an average incidence angle of 80° relative to the normal surface. The spectra were recorded from 4000 to 650 cm⁻¹ for 120 scans with a resolution of 4 cm⁻¹. The IR radiation was p-polarized, and a background spectrum taken from a clean gold-coated reference sample was subtracted from the resulting spectrum. The XPS (VG ESCALAB 220iXL) survey and high resolution core level spectra were collected by using an Al K α (1486.6 eV) X-ray source. The wetting characteristics of all the samples were determined using a contact angle goniometer (Krüss GmbH, Germany) via static water contact angle measurements on water drops of size ~5 μ l using the Laplace-Young method.

The mechanical tests were performed by adhesively joining the NaOH treated surfaces as well as acetone degreased surfaces using a 2-component epoxy adhesive with a bond area of 12.7 mm × 25.4 mm and a nominal bondline thickness of 250 μ m under

pristine and cataplasma conditions using a mechanical testing system (MTS). The cataplasma conditions imply an extreme humidity and temperature exposure as defined by the standard Jaguar JNS 30.03.35. In this process, the assembled SLS specimens are subjected to 100% relative humidity at a temperature of 70 °C for seven days. The specimens are then transferred to a freezer and left for 16 h at a temperature of –20 °C after which the specimens are brought to room temperature and left for 24 h prior to mechanical testing. The SLS specimens were assembled within 1 h following the completion of the treatment process in order to preserve the surface as treated and prevent further contamination from the lab environment which could possibly change the surface characteristics. The assembled surfaces were left for seven days at room temperature to completely cure the adhesive before performing the mechanical tests. The crosshead speed used in the SLS tests was 0.5 mm/min.

3. Results and discussion

A chemical reaction between NaOH and aluminum takes place during the ultrasonic immersion of the aluminum substrates in the NaOH solution. The reaction results in an etching process providing a microrough structure to the treated surfaces. The SEM images in [Fig. 1](#) reveal the microstructural evolution of the various surfaces treated with 0.1 M NaOH solution for various treatment times. After 5 min of treatment time ([Fig. 1\(b\)](#)), it can be noticed that the surface looks much cleaner and possibly free of any organic contaminants as compared to the black spots noticed on the surface that was only acetone wiped ([Fig. 1\(a\)](#)). These black spots seen on the acetone wiped surface may simply be traces of organic contaminants that have not been completely removed in the degreasing process. Further treatment with NaOH for increased times of 30 and 60 min results in surfaces composed of micro-sized crater-like rough features and in addition exposes the grain boundaries which provides another degree of surface roughness ([Fig. 1\(c and d\)](#)). These microstructural investigations show in the present case that a treatment time of 30 min was essential to create a microrough surface topography.

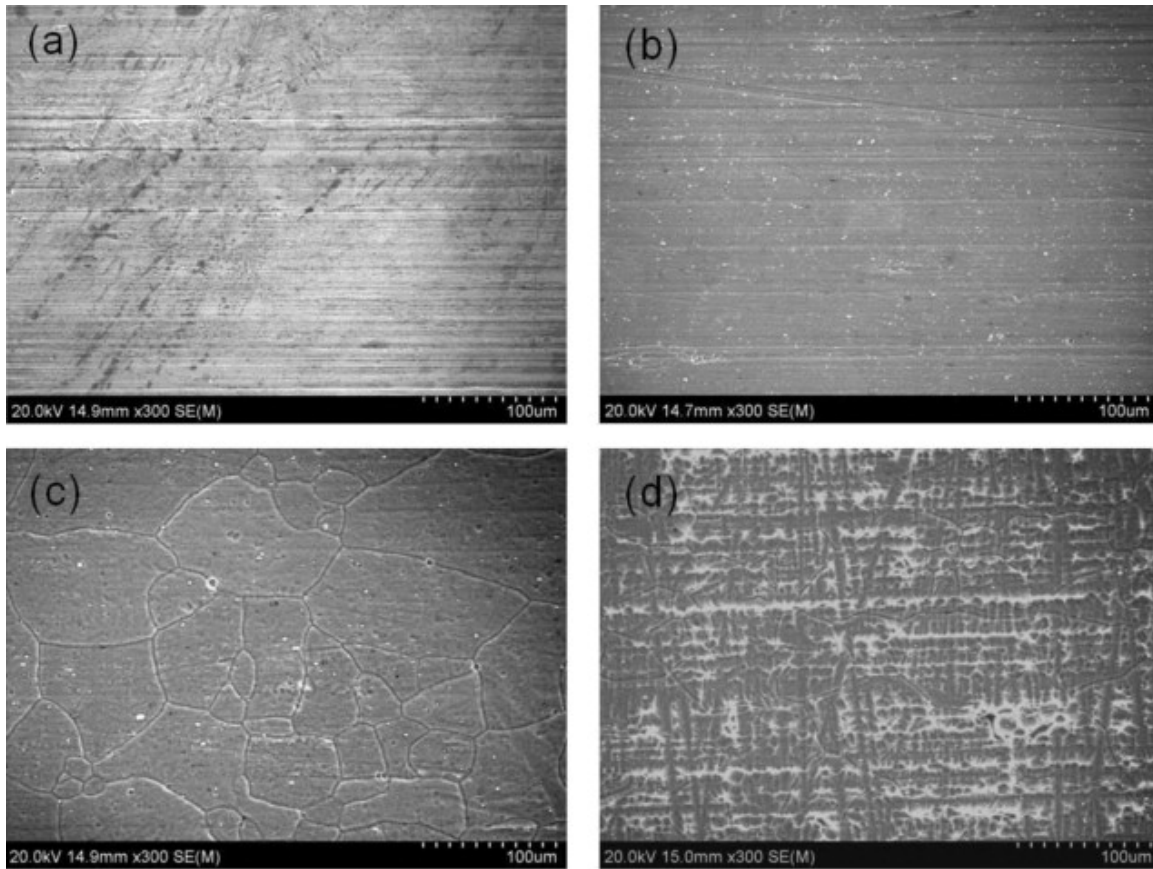


Fig. 1. SEM images of (a) acetone degreased AA 6061 aluminum alloy surfaces and those treated with 0.1 M NaOH solution for a period of (b) 5 min, (c) 30 min and (d) 60 min.

An optical profilometer was used to evaluate the roughness of the treated surfaces as a function of the NaOH treatment time (Fig. 2). After an initial 5 min of treatment, the rms roughness is found to decrease to $0.3 \pm 0.06 \mu\text{m}$ from $0.42 \pm 0.07 \mu\text{m}$ and then increase to $0.5 \pm 0.06 \mu\text{m}$ and $0.94 \pm 0.06 \mu\text{m}$ with further increase in treatment time to 30 and 60 min, respectively. The decrease in the rms roughness after 5 min is attributed to the removal of the surface contaminants during this short period of treatment, resulting in a clean surface as revealed by the SEM images in Fig. 1(a and b). Traces of black spots of surface contaminants along with extrusion lines observed on the acetone wiped surface (Fig. 1(a)) that were not removed during the wipe is considered to have contributed to a high roughness on the untreated surface. The 5 min treated surface, exhibiting a clean and much finer surface (Fig. 1(b)) resulting from the initial stages of the etching process, results in a decrease in the rms roughness. With a further increase

in treatment time, an accelerated etching reaction takes place in which the surface is roughened (Fig. 1(c and d)).

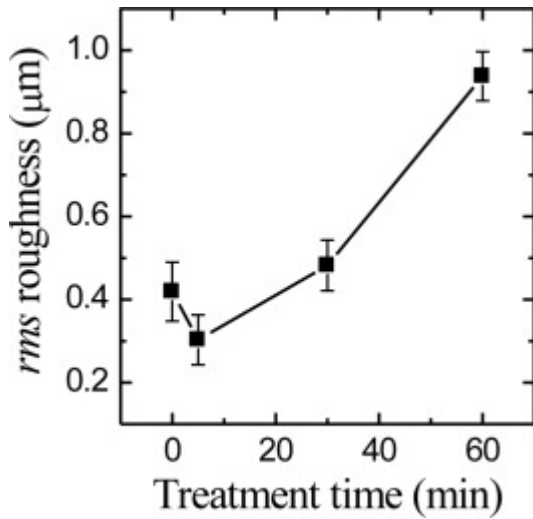


Fig. 2. Surface roughness of aluminum alloy surface as a function of the treatment time, treated with a 0.1 M NaOH solution.

As the reaction of NaOH with aluminum results in an etching process of the aluminum surface, it may be expected that the etching process may remove material from the surface. Therefore, thickness measurements after each treatment were performed using vernier calipers and were compared with the values measured before treatment. Fig. 3 shows a plot of substrate thickness measurements as a function of treatment time of the surfaces treated in 0.1 M NaOH solution. The measurements showed that there was no apparent change in the thickness of the surface following NaOH treatment indicating no apparent loss of material.

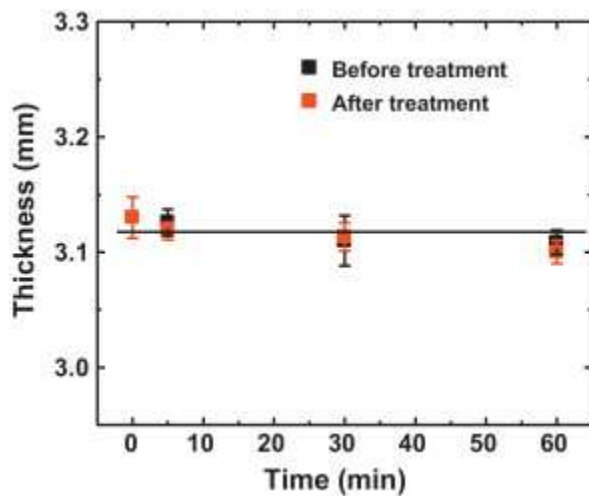


Fig. 3. Substrate thickness vs. treatment time of the substrates treated in 0.1 M NaOH solution.

The XRD analysis of both degreased and NaOH treated aluminum surfaces revealed the main peaks of aluminum at 2θ values of 38.48° , 44.74° and 65.11° assigned to Al (1 1 1), Al (2 0 0) and Al (2 2 0), respectively [9] as shown in Fig. 4. Fig. 4 compares the XRD patterns of the acetone wiped aluminum surface and the surface treated with 0.1 M NaOH for 30 min. No additional peaks signifying a crystalline transformation on the NaOH treated surface was detected in the XRD pattern.

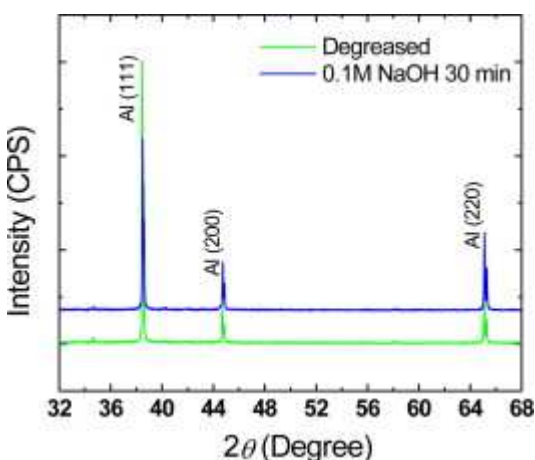


Fig. 4. XRD pattern of surface treated in 0.1 M NaOH for 30 min as compared to aluminum surface degreased by acetone wipe.

However, to understand the chemical nature of the final surface, further analyses were carried out using IRRAS (Fig. 5). The IRRAS spectra of all surfaces treated at various concentrations and times of treatment showed a considerable decrease in the intensity of the —OH band at $\sim 3500\text{ cm}^{-1}$ on the NaOH treated surfaces. Another interesting observation in the IR spectra of the NaOH treated surfaces as compared to the degreased aluminum surface is the appearance of a new intense peak at 944 cm^{-1} after treatment for 30 and 60 min. This peak has been assigned to the Al—O vibration arising from the Al_2O_3 layer on the surface which is in good agreement with previous reports [10] and [11]. NaOH treatment is generally used to remove the native oxide layer present on aluminum surfaces mostly prior to anodization processes [12], [13] and [14]. In the present case, the IR spectral investigations indicate that the NaOH treatment of aluminum surfaces results in the formation of a new stable form of oxide of the form

Al_2O_3 (944 cm^{-1}) on the surface following removal of the weak native oxide layer in addition to creating microrough surface features (Fig. 1). When an aluminum substrate is immersed in a solution of NaOH, an etching reaction produces a water soluble salt, namely, sodium aluminate and hydrogen gas as follows:

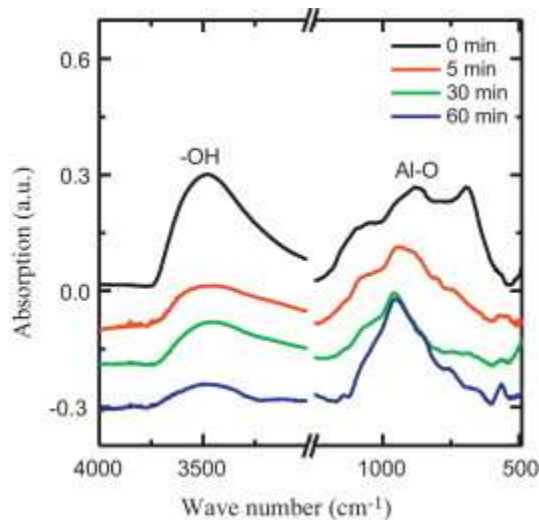
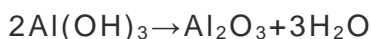
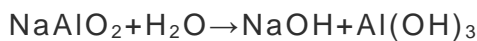


Fig. 5. IRRAS spectra of surfaces treated with 0.1 M NaOH for different treatment times as compared to the acetone degreased surface (0 min).

The sodium aluminate further hydrolyzes in the continuing reaction to produce aluminum hydroxide liberating NaOH to the solution as follows:



The aluminum hydroxide deposited on the substrate surface converts to aluminum oxide after dehydration during the drying process. The IR spectral analyses showing no trace of adsorbed water peaks between 1600 and 1300 cm^{-1} and negligible OH peaks at about 3500 cm^{-1} confirm the above sequence of reactions and the formation of dehydrated alumina at 944 cm^{-1} . However, the newly formed oxide may not be crystalline in nature as we did not observe any XRD peaks signifying the presence of oxides on the NaOH treated surfaces (Fig. 4). The oxide formed in the process may, therefore, be amorphous.

The creation of rough microfeatures (Fig. 1) on the surface following the reaction confirms the etching process [15]. The aluminum dissolved in to the solution during

etching is re-deposited in the form of aluminum hydroxide precipitates which converts into a fresh layer of aluminum oxide following dehydration. This phenomenon confirms that there is no apparent loss of material as complemented by the thickness measurements showing no change in apparent thickness following treatments (Fig. 3). The surface eventually roughens since the areas of bare Al are more prone to the etching reaction than those on which the hydroxide precipitates.

EDX analyses were carried out to estimate the relative concentrations of the oxygen and aluminum following treatment with 0.1 M NaOH for various times (Fig. 6). The oxygen concentration on an untreated surface was only 1.1% by weight. The EDX analyses showed that the oxygen weight percent increased to 1.33 and 1.78 when the surfaces were treated with 0.1 M NaOH for a treatment period of 5 and 30 min, respectively. The oxygen concentration decreased to 1.39 wt% with a further increase in treatment time. This increase and decrease in oxygen weight percent from the EDX analyses may indicate that the NaOH treatment initially favors the formation of a new oxide layer on the surface for a treatment time of up to 30 min. Further increase in treatment time results in a partial removal of the newly formed oxide layer. However, due to the surface sensitivity, XPS analyses were carried out on surfaces treated with 0.1 M NaOH for 5, 30 and 60 min to further understand the surface chemical characteristics following treatment for various times.

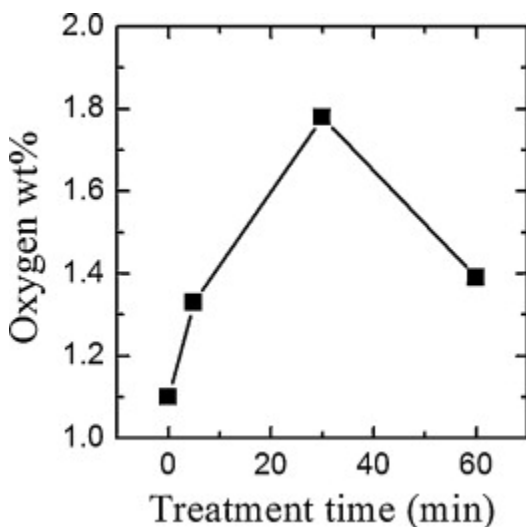


Fig. 6. Oxygen concentration by weight as estimated from EDX analyses.

XPS analyses of the different samples confirmed the presence of the different elements, namely, Al, O, Mg, Cu, Si, and C as shown in the survey spectra of the surfaces treated with 0.1 M NaOH for 5, 30 and 60 min in Fig. 7. These elements are the basic components of the AA 6061 aluminum alloy. The survey spectra as well as the high resolution Al 2p and O 1s spectra of the three surfaces do not show noticeable differences in the Al and O content on the respective surfaces indicating that the chemical composition of the three surfaces remains nearly similar. However, to understand the nature of the oxide layer formed following treatment with NaOH, the high-resolution core level Al 2p and O 1s spectra of the surface treated with 0.1 M NaOH for a period of 30 min were analyzed (Fig. 8). The Al 2p and the O 1s spectra confirmed the formation of oxide of aluminum on the surface composed of 30 at% Al and 70 at% O atomic concentration on the surface. The XPS binding energy value of the O 1s peak was 531.8 eV [16]. The Al 2p peak was resolved into two peaks with binding energies of 74.36 and 71.52 eV [17] and [18] corresponding to oxygen bonded to aluminum and the metallic aluminum, respectively, with respective atomic concentrations of 96.6 at% and 3.4 at% indicating that the surface is predominantly composed of aluminum oxide within the XPS sampling depth. The presence of a small metallic component on the high resolution Al 2p XPS spectrum may also be indicative of the formation of micro-rough aluminum oxide surface due to the etching reaction sequence. Two possible causes for the presence of the metallic component could be that either there are small patches of metallic aluminum visible after etching process is terminated or the thickness of the oxide layer formed is within the XPS sampling depth.

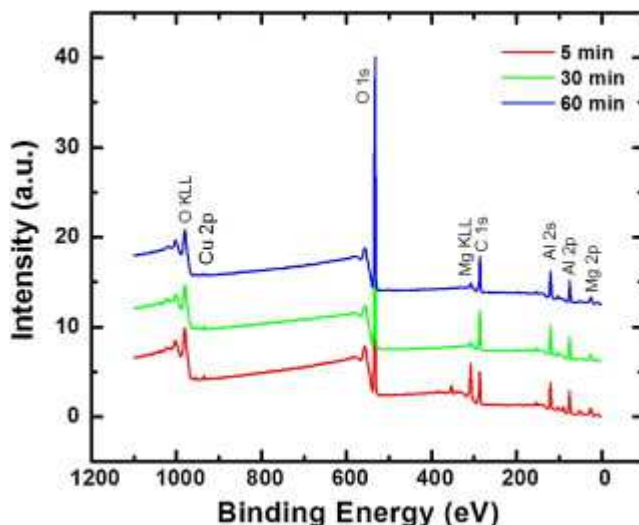


Fig. 7. Survey spectra of the AA 6061 aluminum alloy surfaces treated with 0.1 M NaOH for 5, 30 and 60 min.

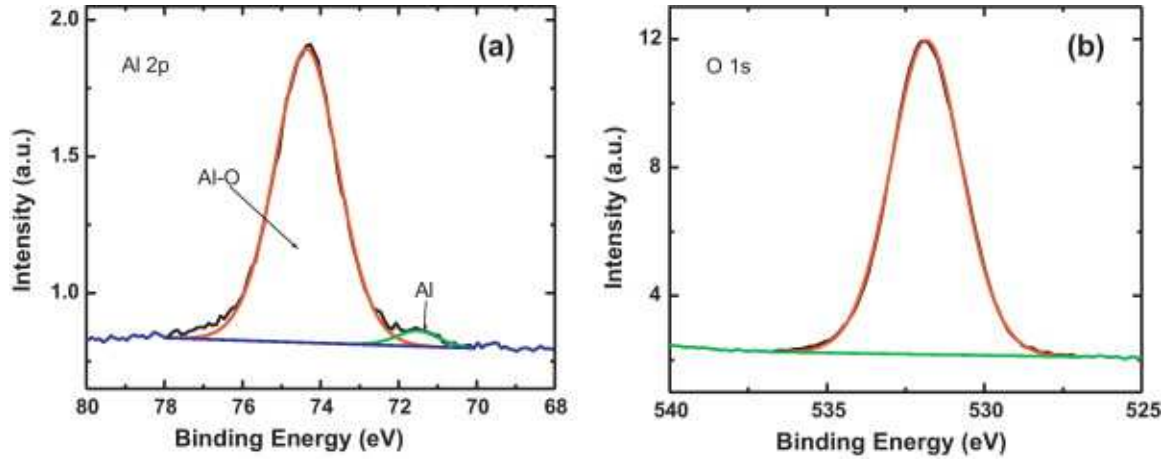


Fig. 8. High resolution core level spectrum of (a) Al 2p and (b) O 1s of the surfaces treated with 0.1 M NaOH for 30 min.

Since the wettability of a surface is considered as indicative of surface cleanliness as well as the suitability of a surface for adhesive bonding, the NaOH treated surfaces were characterized for wettability using water contact angle measurements. The wettability behavior of the surfaces treated in 0.1 M NaOH for different time periods is shown in Fig. 9. The measurements showed that the water contact angle increased to above 90° on the NaOH treated surfaces as compared to only $75.3 \pm 5^\circ$ on a degreased aluminum surface. Two basic models (namely Wenzel model [19] and Cassie–Baxter [20] model) are used to explain the contact angle behavior of water on a rough surface. According to Wenzel model true contact angles lower than 90° on a smooth surface provides an apparent contact angle lower than the true contact angle on the same surface when roughened and vice versa. Therefore, obtained contact angle of $75.3 \pm 5^\circ$ on the flat aluminum surface, clearly indicates that Wenzel model cannot explain the achievement of contact angle $>90^\circ$ on the NaOH treated surfaces. Another model, namely, the Cassie–Baxter model, however, states that roughening a surface can enhance the water contact angle value provided there is a sufficient amount of air entrapped in the irregularities of the rough surface rendering the surface a composite system composed of solid and air [20]. The Cassie–Baxter equation is written as:

$$\cos\theta_c = f_1 \cos\theta_1 + f_2 \cos\theta_2$$

where θ_c is the contact angle of the composite coating consisting of two components with contact angles θ_1 and θ_2 and corresponding area fractions f_1 and f_2 . In such a

composite system if f_1 is assumed to be the solid surface, which in our case is a combination of metallic aluminum and its oxide as revealed by the XPS analysis, and f_2 is assumed to be air where θ_2 is 180° and as $f_1 + f_2 = 1$, the above equation can be further modified as:

$$\cos\theta_c = f_1(\cos\theta_1 + 1) - 1$$

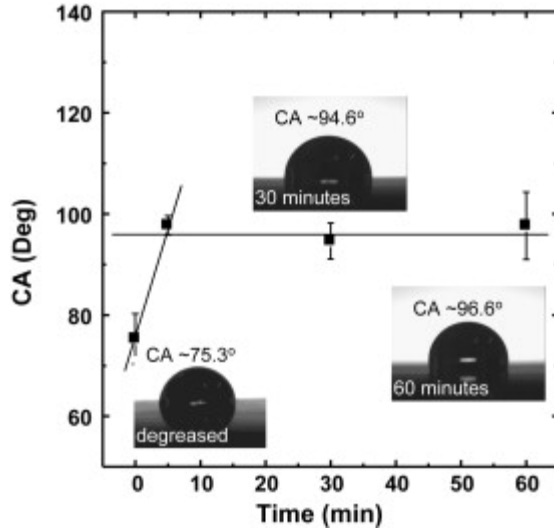


Fig. 9. Water contact angle as a function of time of treatment on surfaces treated with 0.1 M NaOH.

According to Cassie–Baxter model, the water drops do not penetrate the rough irregularities, unlike in Wenzel model, rather they roll off the surface provided the fraction of solid (f_1) coming in to contact with the water drop is very small or negligible. We have previously reported such roll off behavior on certain surfaces engineered to mimic from lotus effect [21], [22], [23] and [24]. Again, this model also does not explain the contact angle behavior on our NaOH treated surfaces, since in our case the water drops remain stuck on the surface, although with a contact angle higher than 90° . However, recent studies on rose petals have shown that there exists a state of Cassie impregnating wetting regime in which the water droplets are expected to enter into the large grooves of the petal resulting in a highly adhesive behavior of water with the surface, but with higher water contact angle values in contrast to the so-called Lotus effect [25] and [26]. Our NaOH treated surfaces, therefore, may be categorized in the Cassie impregnating wetting regime as defined by the “rose petal effect” as the water drops while exhibiting higher a water contact angle greater than 90° remain stuck to the surface and do not dewet the surface on an inclined sample. We have previously reported such a Cassie

impregnating wetting regime and named it as a “sticky Cassie state” in the case of our ZnO nanotowers where the water drop remained stuck to the surface in spite of very high water contact angles [24]. The rose petal effect has also been widely observed by many other researchers recently [27], [28], [29] and [30]. Since the adhesion of water drops to the surface is higher as defined by the rose petal effect in spite of the higher water contact angle, it may be expected that these surface may exhibit high adhesive bond strength.

The water contact angles on all the NaOH treated surfaces, however, remain above 90° and similar irrespective of the increased treatment time and increased surface roughness (Fig. 2). Based on the XPS analysis, the surface composition remain nearly same on all the surfaces, however, in all cases XPS high resolution Al 2p spectra reveals the presence of metallic aluminum with a binding energy of ~ 71.5 eV in addition to an oxide peak of aluminum at 74.36 eV. Therefore, the surface may be considered as a composite surface composed of air, oxide of aluminum and metallic aluminum. The presence of metallic aluminum may lead to an increased surface energy as is the nature of metallic surfaces [24] and consequently, although the surface roughness increases, the contact angle is maintained constant.

Single lap shear (SLS) tests were performed on the surfaces treated with 0.1 M NaOH for various treatment times. The adhesion strength measured on the single lap shear specimens prepared by treating in 0.1 M NaOH for a period of 5, 30 and 60 min are shown in Fig. 10 and compared to the untreated specimen simply degreased in acetone. The degreased specimen presented a combination of visually interfacial and cohesive rupture providing shear strength of 14.5 ± 7 MPa under pristine conditions. However, the mode of failure encountered on the surfaces treated with 0.1 M NaOH was mostly cohesive with the bondline strength of 21.7 ± 3.2 MPa, 21.8 ± 0.5 MPa, and 21.4 ± 2.9 MPa for the specimens treated for 5, 30 and 60 min, respectively. In particular, the specimens treated with 0.1 M NaOH for a period of 30 min exhibited completely cohesive failure as compared to those treated for 5 and 60 min. These results indicate that a treatment time of at least 30 min was necessary to obtain completely cohesive failure. SEM investigations also support this observation as a rough microscale surface texture was obtained only on the surface treated for 30 min, with a rms roughness of 0.5 ± 0.06 μm as compared to those treated for 5 min with a rms roughness of 0.3 ± 0.06 μm . Therefore, an optimum surface roughness value of

$0.5 \pm 0.06 \mu\text{m}$, obtained on surfaces treated for 30 min has been found to be necessary to achieve the best bond strength of the joints.

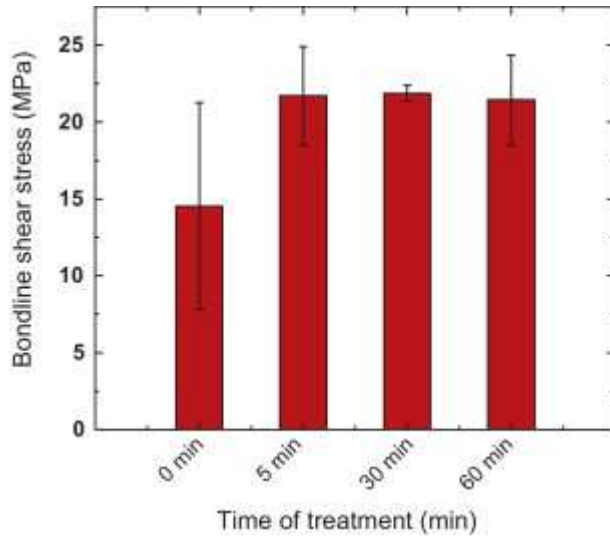


Fig. 10. Adhesion strength measured by single lap shear tests on surfaces treated with 0.1 M NaOH for different treatment times as compared to the acetone degreased surface (0 min) under pristine conditions.

The lap shear values obtained on the NaOH treated surfaces are comparable with those obtained by various anodizing processes which are generally considered as benchmark surface treatment methods in adhesive bonding [5]. Zhang et al., reported an adhesion strength of 23 MPa on phosphoric and chromic acid anodized aluminum surfaces and ~20 MPa on boric and sulfuric acid anodized surfaces [5]. Treatments such as anodization involve, however, the use of strong acids in the anodizing process and multiple steps such as pretreatments to remove the existing native oxide layer and post treatment to close the anodized pores. In our present case, the use of a very dilute base i.e. NaOH (0.1 M) in one single step was sufficient to obtain adhesion strengths comparable to those reported on anodized surfaces. Fig. 11 shows the images of the ruptured specimens treated with 0.1 M NaOH for 5, 30 and 60 min as compared to the surface that was only degreased with an acetone wipe.

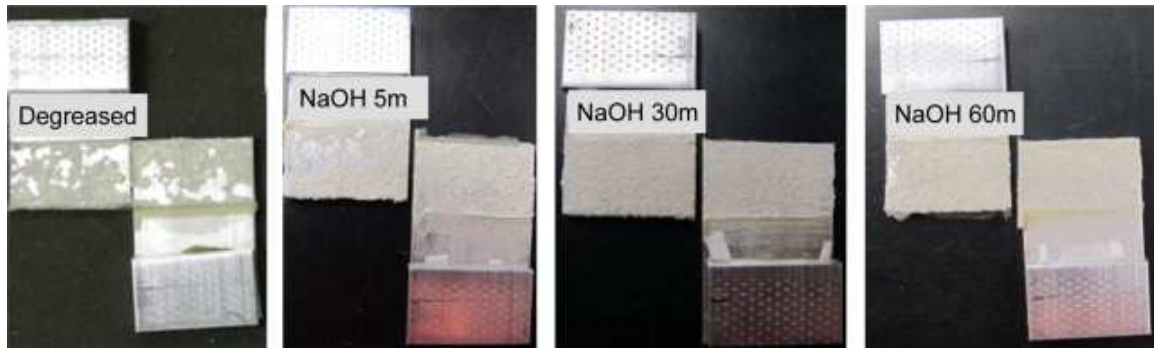


Fig. 11. Images of the ruptured specimens of AA 6061 aluminum alloys treated in 0.1 M NaOH for 5, 30, and 60 min as compared to acetone degreased aluminum surface under pristine conditions.

The surface treated for 30 min, which exhibited a complete cohesive failure in the SLS tests, was then tested for environmental durability by exposing adhesively joined SLS specimens to extreme conditions of temperature and humidity according to the Jaguar standard JNS 30.03.35. Fig. 12 shows the images of the ruptured specimen after the lap shear test. It is clear from the images that the surfaces failed cohesively in spite of their exposure to harsh environmental conditions such as heat and humidity. The lap shear strength obtained on the degraded specimens was 18.8 ± 3.4 MPa indicating a decrease of only 14% in adhesion strength of the joints as compared to that of the non-degraded specimens. A similar result has been previously reported by Zhang et al. on aluminum surfaces anodized using phosphoric acid [5]. However, in the present case, the use of a very dilute concentration of the acid-less NaOH solution is much simpler than the various anodization processes which involve acids such as sulfuric acid, phosphoric acid, chromic acid, etc. and pretreatment steps prior to anodization processes. Moreover, the performance of the joints in both pristine and degraded conditions is comparable to those reported on anodized surfaces [5]. The lap shear strength on the acetone degreased surface was also measured for comparison under degraded conditions and the lap shear strength obtained on these specimens was only 2.6 ± 0.5 MPa indicating a decrease of about 82% in the adhesion strength of the joints as compared to that of the non-degraded counterparts.

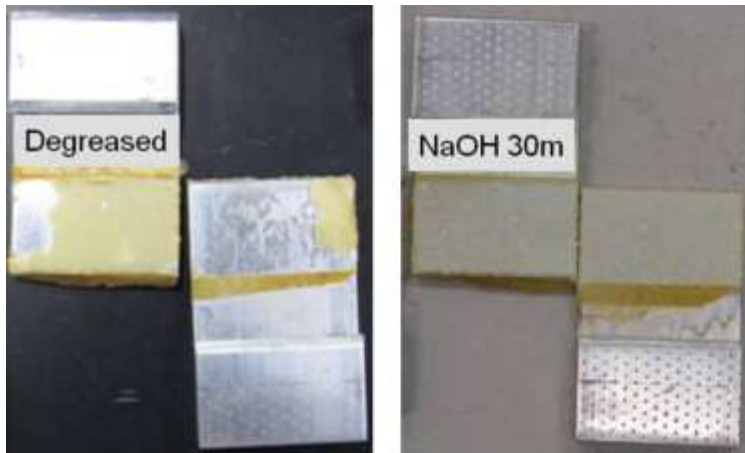


Fig. 12. Image of the ruptured specimen of AA 6061 aluminum alloy treated in 0.1 M NaOH for 30 min as compared to acetone degreased aluminum surface after degradation.

4. Conclusions

A simple and effective way of removing the weak native oxide layer from the AA 6061 alloy surface for adhesive bonding has been demonstrated by treating the surfaces in a very dilute NaOH solution. The morphological analyses by SEM as well as profilometry investigations reveal a rough microstructural evolution on the surface following treatment with NaOH, for which a treatment time at least 30 min was necessary. The chemical analysis of the surfaces by IRRAS, XPS and EDX techniques confirm the formation of alumina on the surface. The type of alumina formed on the surface is possibly amorphous as no peaks signifying the presence of oxides was observed in the XRD pattern. The single lap shear tests performed on the surfaces treated for different time periods show a complete cohesive failure of the specimen treated for 30 min. These surfaces also presented a complete cohesive failure following degradation under extreme humidity and temperature with a decrease in the adhesion strength as low as 20% as compared to SLS samples tested before degradation. These results demonstrate that the dilute NaOH treatment of aluminum alloy surfaces can be considered as a simple and effective means of surface treatment for adhesive bonding using epoxy adhesives as this treatment involves no harsh or expensive chemicals or high temperatures.

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