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Atmospheric pressure plasma oxidation of AA6061-T6 aluminum alloy surface for strong and durable adhesive bonding applications

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ABSTRACT

AA 6061-T6 aluminum alloy surface has been treated using atmospheric pressure helium–oxygen plasma at room temperature prior to bonding with a bi-component epoxy resin. The adhesive joint strengths were evaluated via single lap shear tests as prepared (pristine conditions) as well as following degradation by exposure to extreme temperature and humidity conditions (cataplasma conditions). Very high adhesion strength of 24 ± 1 MPa was achieved on surfaces after a very short exposure of the He/O_2 plasma of only 15 s under pristine conditions resulting in cohesive failure of the adhesive itself. Best results were obtained under cataplasma conditions with adhesion strength of 22.6 ± 1 MPa by introducing a very simple pretreatment with scotch brite® prior to plasma exposure. With many different surface treatment methods being predominantly tested and evaluated, the adhesive bonding community may highly benefit from the present work as the treatment method uses very simple, economical and safe procedures in obtaining results comparable to benchmark methods such as Forest Products Laboratory (FPL) etch, anodization and so on.

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1. Introduction

Many important fields such as aerospace, automotive and marine industries have shown great interest in adhesively bonded structures that are being extensively used by the respective industries [1]. Aluminum is one of the most commonly used materials in structural adhesive bonding due to its many advantages such as excellent mechanical properties, light-weight, abundance, and lower cost. Adhesive bonding of aluminum has several advantages over other joining techniques. These include large area bonding facilities, 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 [2,3]. Surface pretreatments for the adhesive bonding of aluminum have been developed over the years to give stronger and more durable bonds in service [3–6]. Methods such as sulfuric, phosphoric or chromic acid anodizing, conversion coatings and chromic–sulfuric acid etch (FPL etch) are commonly being used in practice to create a surface that can marry with the adhesive being used for bonding [3,4,6]. Recently, we have reported a method involving ultrasonic immersion of aluminum coupons in a dilute NaOH solution in obtaining strong and durable joints [5]. Other methods use mechanical approaches such as grit-blast or sand blasting to remove the mechanically weak native oxide layer. All these surface treatment methods aim at modifying the original surface by altering either the chemistry of the surface to enhance the chemical bond between the surface and the adhesive or to modify the surface topography by creating certain roughness which allows the adhesives to get mechanically interlocked in the rough patterns.

In the present work, we have used the atmospheric pressure helium–oxygen plasma process to oxidize the aluminum surfaces prior to bonding with a bi-component epoxy resin. We have investigated the adhesive bond strength on those surfaces under pristine conditions as well as the joint durability under conditions of extreme humidity and temperature via single lap shear adhesion tests.

2. Experimental

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 He/O_2 plasma exposure. The plasma oxidation of the acetone degreased aluminum surfaces was carried out using an Atomflo™ atmospheric pressure He/O_2 plasma (Surfx Technologies LLC, USA) at rf power of 150 W and flow rate of 30 L/min for He and 500 mL/min for O_2. The surface to plasma torch distance

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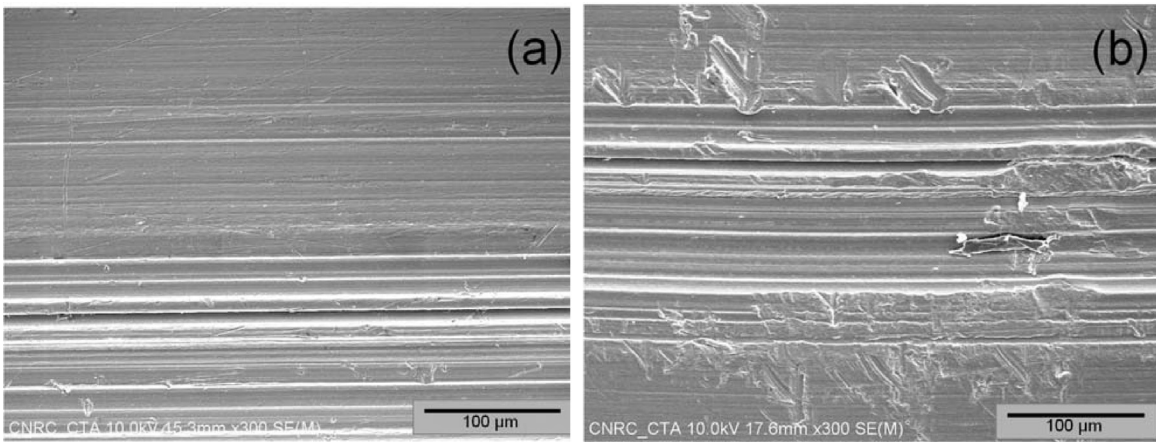

Fig. 1. SEM images of AA6061-T6 aluminum alloys (a) as degreased and (b) following 15 s of He/O_2 plasma exposure).

was maintained at ~3 mm under varying plasma exposure time of 15, 45 and 75 s. Certain coupons were pre-treated with scotch brite® (SB) SB7447B (3 M) prior to plasma exposure in view of obtaining improved durability of the joints following exposure to extreme temperature and humidity conditions. The SB pretreated surfaces were thoroughly rinsed with methyl-ethyl-ketone (MEK) to evaporate any water used during SB rubbing and then dried for 1 h at room temperature prior to exposure to plasma. 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. Infrared reflection absorption spectroscopy (IRRAS, Nicolet 6700 FT-IR) equipped with a Mid-IR MCT-B N_2-cooled detector and a KBr beam splitter was employed to characterize the surface chemistry of the resulting surfaces. 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 500 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 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 μl 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 μm under pristine and cataplasma conditions using a materials 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

Fig. 1 shows the SEM images of the AA6061-T6 aluminum alloy surfaces before and after exposure to helium–oxygen plasma for 15 s. An exposure to plasma for 15 s has not altered the surface topography as it is evident from the respective SEM images. The obvious texture seen on both surfaces before and after plasma exposure originates from the as-received substrate nature in its extruded form. The EDX analysis of the two surfaces showed a minutely higher oxygen concentration of 3.58% by weight on the plasma treated surface as compared to 3 wt.% of oxygen on the untreated surface.

In order to understand the chemical nature of the treated surfaces, IRRAS analyses were further performed. The IRRAS spectra are shown in Fig. 2 for the surfaces treated for different plasma exposure times as compared to the untreated surface. The —OH stretching absorption bands centered in the wavenumber range of 3600–3000 cm^{-1} arising from aluminum hydroxide formed on the plasma oxidized surface was observed in the full range IR spectra [5–8]. The peaks appearing around 1650 and 1500 cm^{-1} may be assigned to the bending mode of molecular water coordinated to octahedral and tetrahedral aluminum ion sites on alumina [9,10]. Studies also complement the fact that these peaks may originate from adsorbed water on an aluminum oxide surface [8,11]. The peak appearing at ~1147 cm^{-1} may be attributable to the

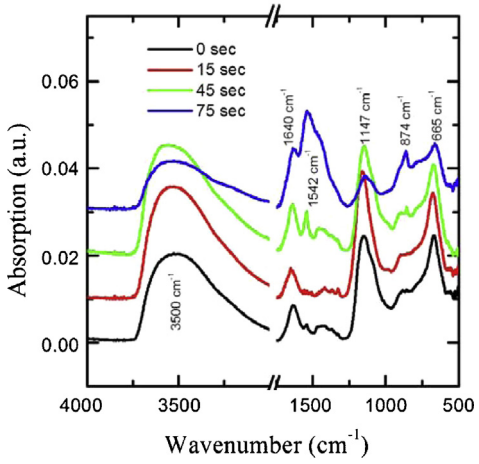

Fig. 2. IRRAS spectra of the plasma oxidized AA6061 aluminum alloy surfaces with different exposure times of He/O_2 plasma. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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