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# Effect of surface pre-treatment on surface characteristics and adhesive bond strength of aluminium alloy

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ABSTRACT

In this work aluminium alloy surfaces have been subjected to three different methods of surface pre-treatments such as solvent degreasing, FPL (Forest Products Laboratory) etching and priming using an epoxy based primer. The treated surfaces were evaluated for surface energy, contact angle, surface topography, surface roughness and adhesive strength characteristics. The influence of surface pre-treatments on the variation of polar, dispersive and total surface energy of the surfaces is addressed. A wettability test was performed on the surfaces using an epoxy adhesive in order to assess the influence of the pre-treatment techniques on substrate/adhesive interaction. Theoretical work of adhesion values for the various pre-treated surfaces were calculated using the contact angle data and further tested experimentally by adhesive bond strength evaluation by tensile testing of a single lap aluminium-epoxy-aluminium assembly. The method of surface pre-treatment showed a profound effect on the surface topography and roughness by AFM. This study reveals that a combination of high surface energy and high surface roughness of the substrate along with good wettability of the adhesive contributed to the highest joint strength for the aluminium alloy through the FPL etching pre-treatment.



## 1. Introduction

Adhesively bonded aluminium joints have wide spread applications in aerospace, automotive and general engineering sectors because of their high strength-to-weight ratio, excellent corrosion resistance and improved manufacturability compared to those made by traditional welding techniques [1–4]. The durability of the adhesive bond strength and the long service life under demanding conditions necessitates the pre-treatment of the surface before adhesive bonding. Aluminium surfaces are usually covered with a weakly bound naturally formed surface oxide layer and adsorbed contamination, which needs to be removed to establish a durable bond between the metal and the adhesive. Various mechanical, chemical and electrochemical surface pre-treatment methods have been reported for aluminium substrates like liquid or vapour degreasing, abrading, grit-blasting, acid / alkaline etching, anodising etc. to name a few [5–12]. A full review on the surface pre-treatments for aluminium alloys has been reported elsewhere [13]. Alkaline etching removes the unstable aluminium oxide/hydroxide film and cleans the stubborn oils and greases off the bonding surfaces. Vapour degreasing consists of removing oils and other organic contaminants from the roughened surface using suitable solvents. Boiling water can act as a surface treating agent for aluminium alloy which results in durable adhesive strength with epoxy adhesive [14]. A combination of different pre-treatment techniques such as grit-blasting, acetone degreasing, alkali etching and phosphoric acid anodising, provides a better adhesive bonding property for aluminium alloys [15]. The most exploited chemical pre-treatment for aluminium adherents is based on chromic-sulphuric acid etching which generates a suitable oxide layer on the substrate surface and can produce strong and durable adhesive bonds [16].

The surface pre-treatment method adopted for the adherent would greatly influence the contact angle. An aluminium alloy surface after alkaline etching, dipping in warm water followed by treating with silane solution showed a decreased contact angle for water and a polyurethane adhesive on the surface [17]. A higher

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surface roughness of the adherent favoured the spreading of the adhesive drop on its surface. Studies conducted elsewhere have shown that surface roughness and chemical heterogeneities greatly influence the contact angle values of the adhesive formed on the adherent surface [18–21].

One of the factors deciding the durability of an adhesive joint is the extent of penetration of the adhesive into the pores of the surface film formed after the pre-treatment [22]. The penetration of the adhesive depends on many factors such as pore dimensions, contact angle between the adhesive and the substrate, viscosity of the adhesive and the viscosity–time characteristics at the temperature of applications [23]. In the case of adhesively bonded aluminium, there have been comparatively few studies on the influence of surface roughness on joint strength. It has been reported that a commonly used chromate pre-treatment improves the lap shear strength of bonded joints with optimal joint strength corresponding to a surface morphology consisting of etch pits of 1–5 μm in diameter [24]. Similar structures have been reported, where the etched and anodised aluminium surfaces were scanned using transmission electron microscopy (TEM) [25,26]. These workers have attributed much of the increase in joint strength of pre-treated aluminium to fine scale oxide structures.

The present study is focused on the effectiveness of various surface pre-treatments for producing strong adhesive bonds on aluminium interfaces which is evaluated using the single lap shear test with an epoxy adhesive. The influence of surface energy, contact angle, surface topography and surface roughness on the experimental shear strength properties of aluminium substrates was explored.

## 2. Materials and methods

### 2.1. Materials

Aluminium alloy, B51 SWP was used as the substrate. The adhesive employed was an epoxy resin supplied by M/s. Huntsman India pvt. ltd., cured at 100 °C with o,o′ Bis(2-aminopropyl) polypropylene glycol purchased from M/s. Sigma Aldrich, India. BR127, purchased from M/s. Cytec Industries Inc.,USA was used for priming the aluminium substrates. Diidomethane, 98% and water (HPLC grade), used as the reference liquids for measuring the surface energy of the aluminium substrates, was obtained from M/s. Spectrochem, India. Trichloroethylene 99.9% from M/s. Nice, India was used as the solvent. The FPL etch solution was prepared as per the standard procedure given elsewhere [27].

### 2.2. Methods

#### 2.2.1. Substrate preparation

All the substrate surfaces were initially abraded using P100 grade emery paper and any emery dust was removed with a clean forced air supply. The substrate surfaces were subjected to three different types of pre-treatment as follows.

*2.2.1.1. Solvent degreasing (method* ***a****).* Solvent degreasing was conducted by wiping the surface using a lint free cotton cloth soaked with trichloroethylene solvent followed by drying with a hot air stream which did not exceed a temperature of 60 °C. This surface was designated as Al-SD.

*2.2.1.2. Method* ***a*** *followed by FPL etching (method* ***b****).* This involved immersion in an FPL etch solution for 15 min. at 60 °C, followed by rinsing in tap water and drying with a hot air stream at a temperature not exceeding 60 °C. The resulting surface was designated as Al-SDFPL.

*2.2.1.3. Method* ***b*** *followed by primer application (method* ***c****).* Aluminium substrates were primed immediately after method b**.** Substrates were dipped in primer BR127, kept in a vertical position for removal of excess primer at room temperature for 30 min. and further cured at 120 °C for 30 min. in a hot air oven. This surface was designated as Al-SDFPLP.

#### 2.2.2. Substrate surface characterisation

The substrate surface was analysed for surface energy, contact angle, surface topography, roughness and adhesive strength properties. The contact angles of the reference liquids and the dynamic contact angle of an epoxy adhesive on variously pre-treated aluminium substrates were analysed using a sessile drop technique using a video based optical contact angle measuring equipment OCA20 (M/s. Data Physics, Germany). The surface energy of the substrates, which is calculated as the sum of the polar and dispersive components were measured by the Owens Wendt Rabel Kaelble (OWRK) method [28,29]. The theoretical work of adhesion of the epoxy adhesive on the variously pre-treated substrates was determined using the Dupre-Young equation. Single lap shear strength of the bonded joints was determined according to ASTM D1002-72. The testing was carried out with a universal testing machine (UTM) Instron Model 5569, where, five samples were tested in each case. The surface topography of the various pre-treated aluminium substrates was measured using a 300R atomic force microscope (M/s. WILec, Alpha, Germany) in a non-contact mode at a scanning speed of 1 μm/s. for an analysing area of 100 μ × 100 μ. All the images had 256 data points with a scan rate of 1.0 line/s. Three individual scan areas were considered and the roughness values were averaged to calculate the surface roughness using WITec project plus software.

## 3. Results and discussion

The substrate surfaces after various pre-treatments were analysed for contact angle, surface energy, surface topography and adhesive strength properties.

### 3.1. Surface energy

The contact angles made by the reference liquids water and diiodomethane on an aluminium substrate surface after the three pre-treatment techniques are reported in Table 1.

The variation of the dispersive and polar components of surface free energy and the total surface energy of the various surfaces studied are given in Fig. 1a–c respectively.

Fig. 1a indicates that even though the surface energy contribution of the dispersive component is high in comparison to the polar component, the variation is minimal for the different pre-treated surfaces. Fig. 1b shows that the variation in the polar contribution is more prominent in determining the total surface energy. The contribution by the polar component is the highest for the Al-SDFPL surface due to the freshly formed aluminium oxide layer on the surface and lowest for the primed surface. Hence the

**Table 1**
Average contact angles in degrees shown by two reference liquids and the surface roughness average of variously pre-treated aluminium substrates.

| System | Contact angle (°) | | Surface roughness $R_a$ (nm) |
|---|---|---|---|
| | $H_2O$ | $CH_2I_2$ | |
| Al-SD | 76 | 63 | 530 |
| Al-SDFPL | 32 | 56 | 620 |
| Al-SDFPLP | 93 | 54 | 390 |