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Journal of Mechanical Engineering

An International Journal

Volume 11 No. 1

June 2014

ISSN 1823-5514

Interaction of Mixing Factors with Mechanical Properties of
PP/ENR Blend via Response Surface Methodology

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Journal of Mechanical Engineering (ISSN 1823-5514) is published by the Faculty of Mechanical Engineering (FKM) and UiTM Press, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia.

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Enhancement of Adhesive Bonding Strength: Surface Roughness and Wettability Characterisations

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ABSTRACT

In order to enhance the adhesive bonding, two different methods of surface treatments have been carried out on aluminum alloy 2024-T3 substrate i.e. alkaline etching and silane treatment. The topology and surface chemical analyses of treated alloy were investigated by AFM and XPS, respectively. The effect of surface treatments on the wettability and adhesive bonding strength were also studied. Three different formulations of polyurethane adhesive by varying the NCO: OH ratio that were prepared from the reaction of green polycaprolactone (PCL) polyol and diisocyanate. Silane treatment showed better wettability and adhesive bonding strength compared to alkaline etching surface treatment.

Keywords: *alkaline etching, silane treatment, polyurethane adhesive, wettability, adhesive strength*

Introduction

Metals have high energy surfaces to absorb oils and other contaminations from the atmosphere. Thus, it is impossible to obtain a quality adhesive bond without cleaning or abrading the metal surface.

In order to obtain interaction between the adhesive and the metal substrate, it is necessary to let the adhesive in wetting the substrate. Thus, surface preparation before applying the adhesive is one of the important factors in increasing the wettability characteristic. Some researchers have investigated the properties of adhesive bonding by immersing the aluminum in boiling water, followed by the application of silane. Rider and Arnott [1] have shown that the durability of aluminum bonding using this surface treatment has improved. Unfortunately, when it comes to the real industrial field, it would not be easy to immerse the large size of aluminum sheet in the boiling water especially for aircraft application. This limitation attracted other researchers to find an alternative method to improve the wettability of aluminum substrate surface. Underhill and Rider [2] reported that a porous hydrated oxide structure can be obtained on the aluminum alloy 2024 T3 and 7075 T6 when immersed in water temperatures as low as 40°C. The aluminum alloy 2024 T3 that has undergoes the pre-treatment with warm water 50°C has shown excellent performance on epoxy adhesive bonding [3].

Another surface treatment that has attracted among researchers is alkaline etching, by immersing aluminum substrates in a solution of sodium hydroxide (NaOH). Saleema et al. [4] has reported that NaOH treatment can be considered to improve the adhesive bonding. Furthermore, this treatment involves low temperatures.

Most of the literature works on surface treatment using epoxy adhesive, but only a few works using of polyurethane (PU) as an adhesive resin was studied. This inspired the authors to investigate the effect of surface treatments on the properties of PU adhesive/aluminum bonding. This research was focused on the surface treatments on the aluminum alloy 2024-T3 substrates with the intention of ensures the adhesive can be used in aircraft or automotive application. The substrates were treated using two types of surface treatment i.e. alkaline etching and silane treatment. The effect of surface treatments on the surface topology, wettability and adhesion between the substrate surface and PU adhesive are the main focuses of this study. We also focused on preparing the PU adhesives by re-acting the green polycaprolactone (PCL) polyol with diisocyanates with three different NCO: OH ratios (1.3, 1.5 and 1.7).

Materials and Methods

Materials

A PCL polyol based on palm kernel oil polyester (PPKO) with the hydroxyl value of 522 and molecular weight of 754 Daltons was prepared earlier by using ring opening polymerization method which described in our previous study [5]. 2, 4-diphenyl methylene diisocyanate (MDI) with NCO content 31% was obtained from Maskimi Polyols Sdn. Bhd., bis(4-isocyanatocyclohexyl) methane (H_{12} MDI) with NCO content 32%, sodium hydroxide (NaOH) and ethanol were purchased from Sigma Aldrich. γ -Glycidoxypolytrimethoxysilane A 187 (γ -GPS) was supplied by Momentive Performance Materials Pte. Ltd.

Alkaline Etching Treatment

Aluminum alloy AA2024-T3 was used as a substrate. The substrates were cut according to ASTM D1002-72 with a dimension of 25.4 mm x 102 mm x 1.6 mm. The substrate surface was grinded using an abrasive paper with grit 600. They were then wiped clean with tissues, soaked with acetone, to remove greases, dirt, etc. Then, the substrates were alkaline etched by immersion in 5% NaOH solution for 5 min at room temperature. Immediately after etching process, the substrates were rinsed using deionized water. The smut layer caused by the alkaline etching was then removed by immersion in 30% HNO_3 solution for 30 s and finally the substrates were thoroughly rinsed using deionised water and dried in an oven at 40°C for 30 minutes. Then, the PU adhesive was immediately applied on the substrates, to prevent the adsorption of airborne organic contaminants, which occurs rapidly and is known to cause a decrease in reactivity of the aluminum substrates [6].

Silane Treatment

The substrates have undergone the same process of grinding and cleaning as the alkaline etching treatment. Then, the substrates were pre-treated in warm water (50°C) for 30 min, rinsed with distilled water and dried in ambient air. Aluminium was then dipped in 1% γ -GPS solution, prepared with a ratio of 1ml γ -GPS to 99 ml ethanol under continuous stirring for 30 min, for the duration of 5 min for the surface silanization. The silane films were dried in ambient air and then held at 110°C in the oven for 30 min.

Preparation of Polyurethane Adhesive

The PU adhesives were prepared in a clean, dry paper cup. Two types of isocyanate (MDI and H_{12} MDI with a ratio of 65:35) were added to the requisite amount of polyol resin. The ratio of NCO to OH was also varied from 1.3, 1.5 to 1.7; thus, the samples of PU adhesives were labelled PU1.3, PU1.5, and PU1.7, respectively. The mixture was mixed well using a mechanical stirrer at 1000

rpm for about 15 seconds and then applied immediately to the substrates using a brush. Then, the adhesive bonding was cured in an oven at a temperature of 120°C for one hour.

X-Ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy (XPS) is a sensitive spectroscopic technique that analyses the surface chemical elements on about 10 atoms of the outermost layer on the surface of the sample by recording the electron binding energy. This technique also gives information about the actual compounds on the surface of the sample.

The test was performed by using the spectrometer *Kratos / Shimadzu* model *Axis Ultra DLD*. The samples with a diameter of 5 mm were evacuated (1×10^{-9} torr) for 12 hours before analysing. In this experiment, there were two spectral scans have been carried out for each sample, i.e. wide and narrow scan spectrum. Wide scan spectrum shows the presence of the elements on the sample surface qualitatively. Almost all of the elements can be detected based on the energy binding value except the elements hydrogen and helium. Narrow scan spectrum enables the quantitative analysis and interpretation of the chemical state of an element. Based on spectral peak areas and sensitivity factors, the presence of elements in the percent of the sample surface can be determined. Chemical state of the element was then determined by performing a matching curve and deconvolution peak by using the software, *Vision Manager*.

Atomic Force Microscopy (AFM)

Atomic force microscopy (AFM) was used to scan and analyse the surface of the substrate samples before and after treatment for the analysis of surface roughness. AFM operation occurs when the sharp cantilever tip interacts with the sample surface by giving a signal to the forces between the molecules of the sample surface and the cantilever tip.

In this study, the topography images in 3D, including the physical characteristics of the surface were generated by using the tools NTEGRA Prima NT-MDT model. The roughness profile such as average roughness (R_a), root mean square roughness (R_q), mean spacing (S_m) and peak density (D) were determined and recorded. The average roughness is described as equation (1) [7]:

$$R_a = \frac{1}{L} \int_0^L |Z(x)| dx \quad (1)$$

where $Z(x)$ is the function that describes the surface profile analysed in terms of height (Z) and position (x) of the sample over the evaluation length L (Figure 1).

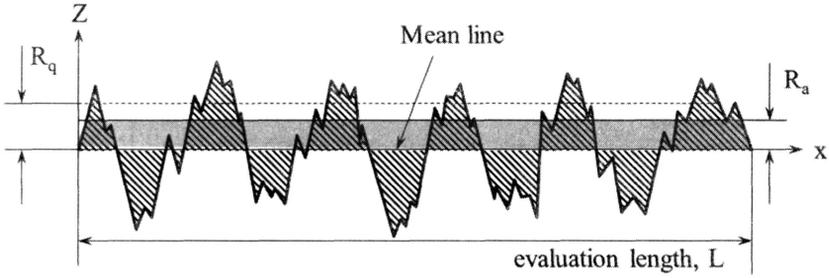


Figure 1: Profile of a surface (Z) that represents the average roughness R_a and R_q based on the mean line [7]

The root mean square (RMS) is a statistical measure used in different fields. The R_q is a function that takes the square of the measures. It is similar to the average roughness, with the only difference being the mean squared absolute values of surface roughness profile. The function R_q is defined as [8] equation (2):

$$R_q = \sqrt{\frac{1}{L} \int_0^L |Z^2(x)| dx} \quad (2)$$

The S_m is the average spacing between peaks in the length of the evaluation. In this case the peak is defined as the highest point, along the profile, between line crossings over the midline and returning below the midline. The spacing between peaks is the horizontal distance between the points where two peaks cross above the midline [8]. Three individual spaces in a surface profile are shown in Figure 2. The mean spacing is the average of the three individual spaces on the evaluation length. Thus the S_m is defined as the average of spacing individual (S_i) in equation (3):

$$S_m = \frac{1}{L} \sum_{i=1}^L S_i \quad (3)$$

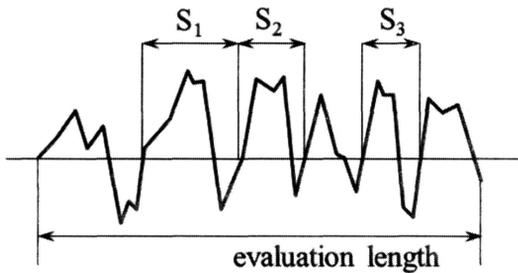


Figure 2: Spacing between peaks

Meanwhile, D represents the density of peaks, i.e., the number of peaks per unit area.

Contact Angle

Contact angles are closely related to wettability. A liquid (adhesive) will wet a solid (substrate) when its surface energy is lower than the solid surface energy. A smaller contact angle means that more of the fluid spreads over an area for a given fluid volume; hence the fluid has a high wettability on that surface. The surface tension of the liquid is then equal to the critical surface tension of the substrate. Large contact angles are associated with poor wettability.

The determination of contact angle on the substrate surfaces with different surface treatment processes were performed by using Ramehart contact angle Goniometer. Deionised water and PU adhesive resin were used as liquids to drop onto the surface of the substrates. For each material, five drops of water were placed along the surface to obtain the average of 10 readings for each sample.

Single Lap Shear Test

Specimens of single lap shear test were prepared according to ASTM D1002-72. The overlapped area was $12.7 \times 25.4 \text{ mm}^2$. The substrates were aligned and pressed with clips. A load of 1 kg was placed over the joint to squeeze out extra resin until the uniform thickness of 0.1 mm was obtained. Testing of bonded joints was carried out with Universal Testing Machine model Instron equipped with 35 kN load cell to measure lap shear strength of the Al joints. Ten samples of each case were tested in tensile load. The crosshead speed was kept at 0.5 mm/min. The shear stress was evaluated by the formula: $\tau = P/A$, where P is the tensile load, and A is the joint overlapped area.

Results and Discussions

Surface Chemical Analysis

Figure 3 shows a wide scan of XPS spectra for untreated, NaOH and silane treated AA2024-T3 substrates. Each spectrum indicates the presence of elements such as Al, Mg and Si. These elements are the basic components of AA2024-T3 aluminium alloy. In addition, O element is also found to be present at each sample substrates where it refers to an oxide or hydroxide layer. Peak of Cu element only can be observed on the NaOH treated spectrum. This may be correlated to the enrichment of Cu on the substrate surface during the etching process in the NaOH solution. In addition, the presence of the C element in both untreated and NaOH treated samples are believed from the atmosphere [9].

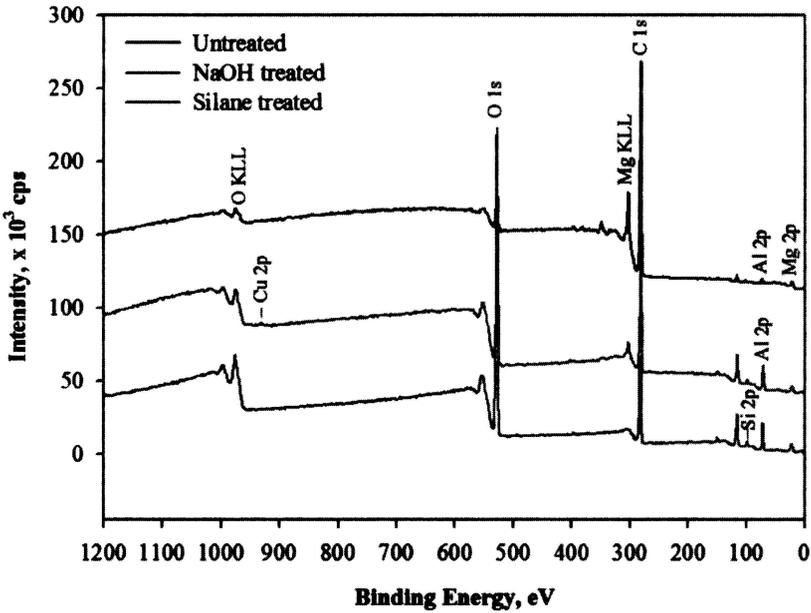


Figure 3: Wide scan of XPS spectra

Figure 4 and 5 show the narrow scan or high resolution core level spectra of Al 2p and O 1s for untreated and NaOH treated AA2024-T3 samples, respectively. Figure 4 (a) shows a narrow scan of Al 2p for untreated sample with two deconvoluted peaks with binding energy of 72.5 and 74 eV corresponding to the presence of metal elements of Al in the Al [10] and natural oxide (Al_2O_3) layer [11], respectively. Deconvolution peak for O 1s for the untreated sample (Figure 4 (b)) results in two peaks of O 1s. First peak of O 1s with binding energy of 531.5 eV indicates the presence of element O in the Al_2O_3 layers, while the second peak of O 1s at 533.1 eV indicates the presence of elements O in the layer of Al-OH or H_2O compounds [12].

Figure 5(a) shows a narrow scan of Al 2p for NaOH treated sample. The deconvolution of Al 2p peak resolves into two peaks of Al 2p. First peak of Al 2p that is at 74 eV refers to the presence of Al in the Al_2O_3 layer. The second peak of Al 2p (74.6 eV) indicates the presence of Al element in the compound of aluminium hydroxide ($\text{Al}(\text{OH})_3$) or H_2O [13]. The presence of both layers of Al_2O_3 and $\text{Al}(\text{OH})_3$ on the substrates shows that the reversible reaction between $\text{Al}(\text{OH})_3$ and Al_2O_3 with the presence of water or moisture from the environment has occurred. This phenomenon can be proved from the narrow scan of O 1s for NaOH treated sample (Figure 5 (b)).

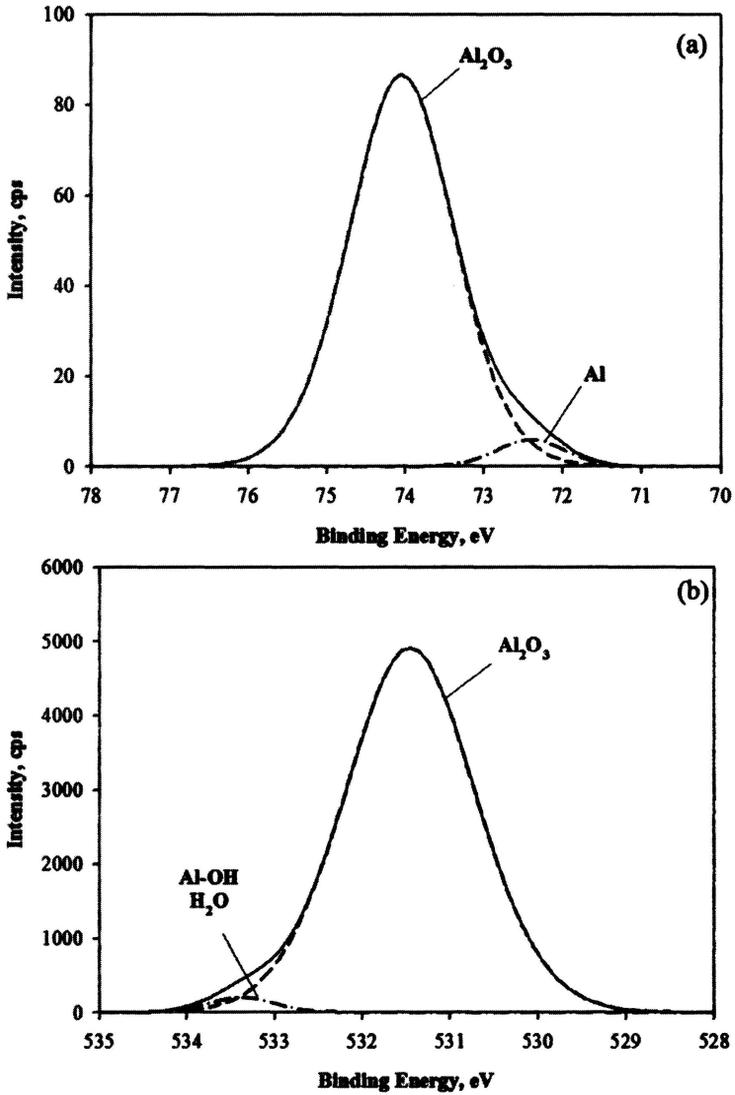


Figure 4: Narrow scan of XPS spectra for untreated (a) Al₂p, (b) O 1s

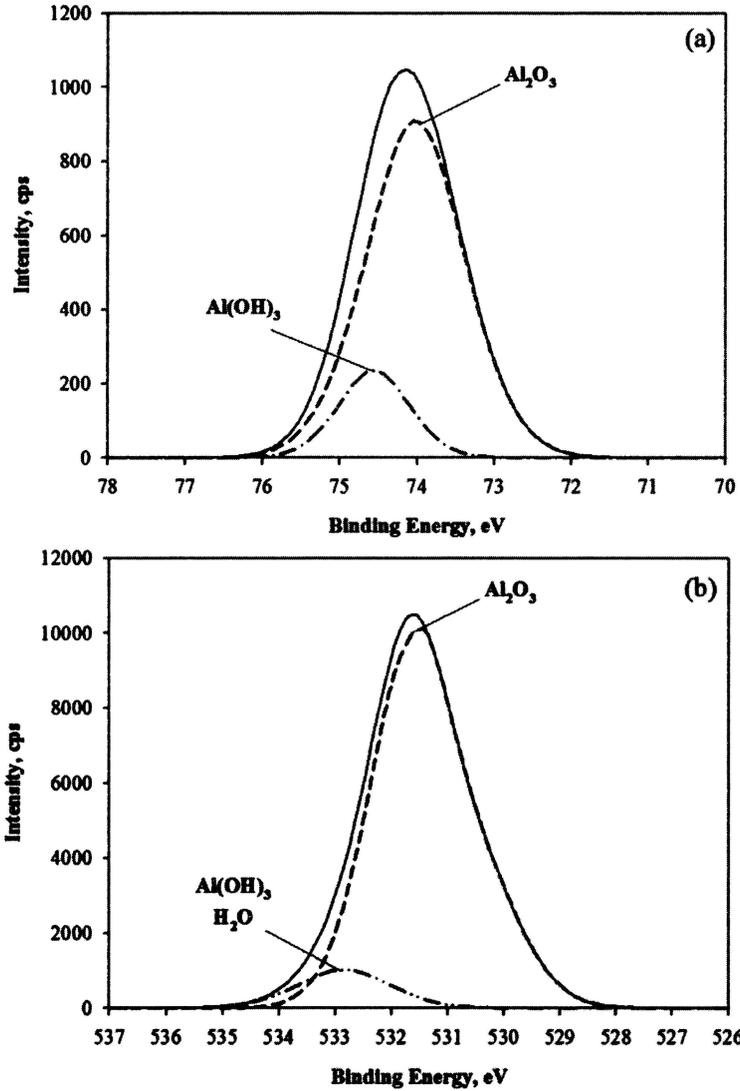
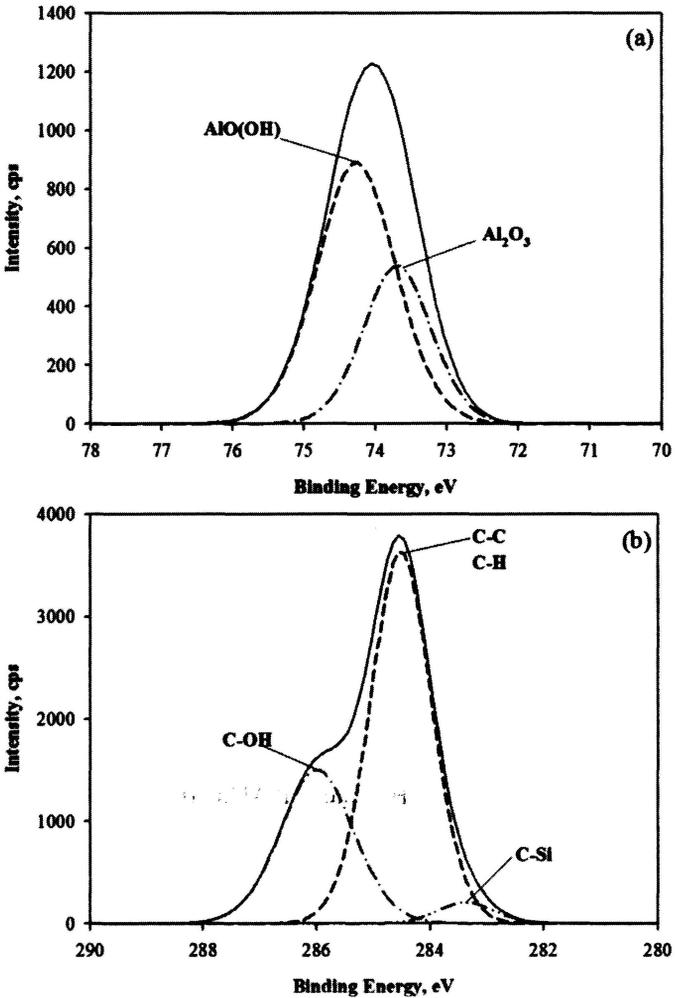


Figure 5: NaOH treated (a) Al 2p, (b) O 1s

The deconvoluted peaks of O 1s confirm the presence of O element in the of Al_2O_3 and $Al(OH)_3$ layers, which can be observed at 531.6 and 532.8 eV, respectively. Changes to the binding energy of O 1s for NaOH treated sample indicates surface chemistry of the substrate has changed [14].

Figure 6 exhibits the narrow scan or high-resolution core level spectra of Al 2p, C 1s, O 1s and Si 2p for the silane treated sample. Figure 6 (a) shows the deconvolution peak for Al 2p peak splits into two peaks which the first peak with the binding energy of 73.7 eV refers to the presence of Al elements in the Al_2O_3 layer. The second peak of Al 2p (74.2 eV) indicates the presence of Al element in the boehmite ($AlO(OH)$) compound [15]. These compounds are



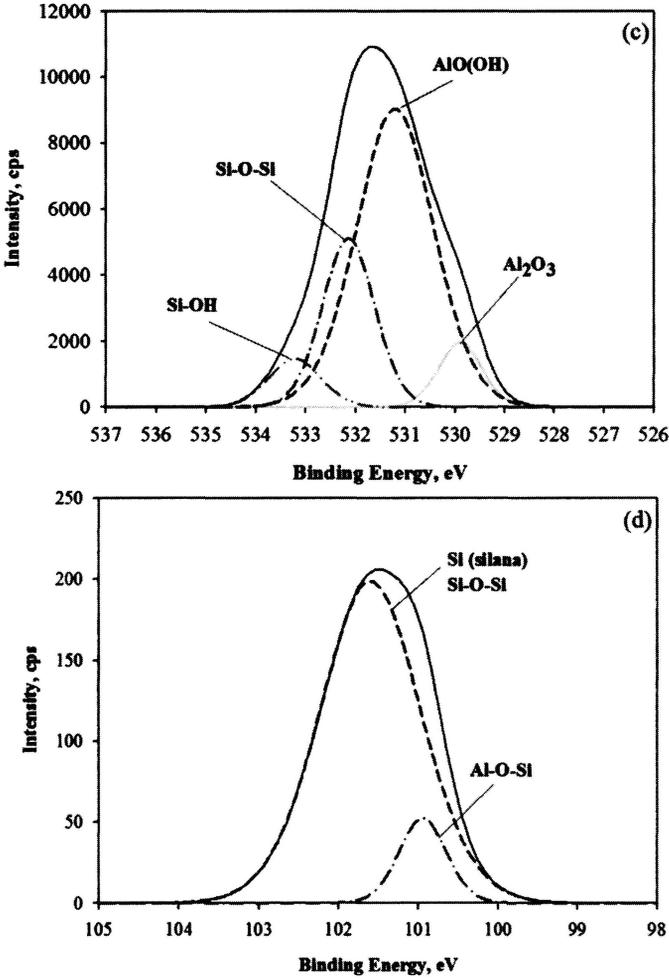


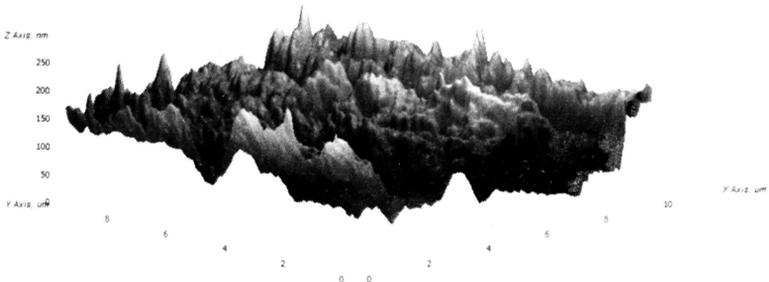
Figure 6: Silane treated (a)Al 2p, (b) C 1s, (c) O 1s, (d) Si 2p

formed during the pre-treatment process with warm water (50°C) before it is being treated with a silane solution [2]. Figure 6 (b) shows the narrow scan of core C 1s of silane treated substrate. The deconvolution of C 1s peak resolves into three C 1s peaks. Peak 1 (283.4 eV) refers to the element C in the silane C atoms bonded to the silicon atom (-C-Si-). Peak 2 (284.5 eV) indicates the presence of C element in the silane bonded with another C atom (C-C) or H atoms (C-H). Peak 3 with the binding energy of 286 eV corresponds to the presence of C elements in C-OH compounds that resulting from the environment.

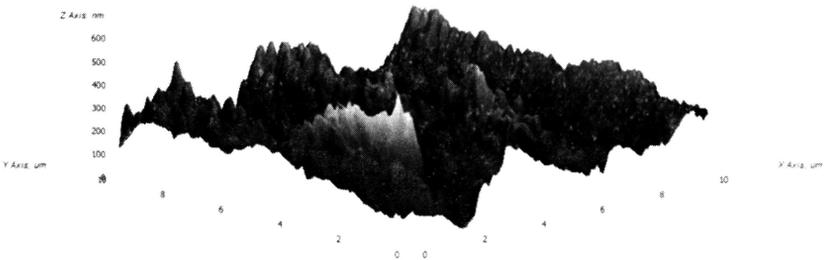
Narrow scan of core O 1s of silane-treated substrates are shown in Figure 6 (c). There are four O 1s peaks resulting from the deconvolution of O 1s peak. Peak 1 (529.9 eV), indicates the presence of O element in the Al_2O_3 layer, while the second peak (531.2 eV) confirms the presence of O elements in the layer $AlO(OH)$ [16]. Peak 3 (532.1 eV) refers to the presence of O element in the layer siloxane (Si-O-Si) [17]. Meanwhile, peak 4 which at 533.2 eV, is an indication of the presence of O elements in the Si-OH compound [18].

Figure 6 (d) shows the narrow scan of Si 2p core of silane-treated substrate. The deconvolution of Si 2p peak results into two Si 2p peaks. The first peak which at 100.9 eV confirms the presence of Si element in silane or siloxane whereas the second peak (101.6 eV), indicates the presence of Si element in the metallo-siloxane layer (Al-O-Si) [19].

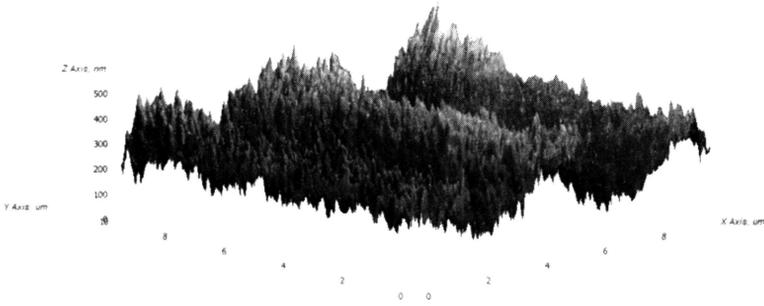
Topology and Surface Roughness Analysis



(a)



(b)



(c)

Figure 7: 3D texture image of substrate AA2024-T3 surface, (a) untreated, (b) NaOH treated dan (c) silane treated

Effect of different surface treatments on the topography or texture of AA2024-T3 substrate surface can be observed clearly in Figure 7 derived from testing of topography by using AFM. Three dimensional (3D) texture image of NaOH treated (Figure 7 (b)) and silane treated (Figure 7 (c)) AA2024-T3 substrates surface proved to be coarser than untreated substrate surface (Figure 7 (a)). The changes of surface roughness value can be seen in Table 1.

Table 1: Surface roughness profile of AA2024-T3 substrate

Substrate	R_a (nm)	R_q (nm)	S_m (μm)	D
Untreated	36.48 ± 0.5	42.74 ± 0.5	0.57 ± 0.03	4.3 ± 0.1
NaOH treated	62.02 ± 0.2	81.23 ± 0.3	0.37 ± 0.01	6.5 ± 0.1
Silane treated	71.12 ± 0.4	84.29 ± 0.3	0.21 ± 0.01	11.2 ± 0.3

Table 1 shows the surface roughness profile of AA2024-T3 substrates with different surface treatments. The average surface roughness (R_a) value of untreated AA2024-T4 substrate is 36.38 nm. The R_a values for NaOH and silane treated AA2024-T3 substrate are 62.02 nm and 71.12 nm, respectively. In addition, the root mean square roughness (R_q) value also show a similar trend i.e. R_q (silane treated) > R_q (NaOH treated) > R_q (untreated). This micro to nano roughness is due to the mechanical abrasion process.

Table 1 also shows the value of S_m for all substrates of $0.57\mu\text{m}$ (untreated); $0.37\mu\text{m}$ (NaOH treated) and $0.21\mu\text{m}$ (silane treated). This indicates that the spacing between peak on the NaOH and silane treated surface are closer compared to untreated surface. Furthermore, the value of D shows the increment from 4.3 (untreated) to 6.5 (NaOH-treated) and 11.2 (silane treated). This indicates that the number of micro irregularity peak on the surface of the treated substrate is higher than the untreated surface. This means that the total interfacial contact area between the adhesive and NaOH and silane treated AA2024-T3 increased. This helps the wetting process of PU resin adhesive on the substrate surface.

Wettability

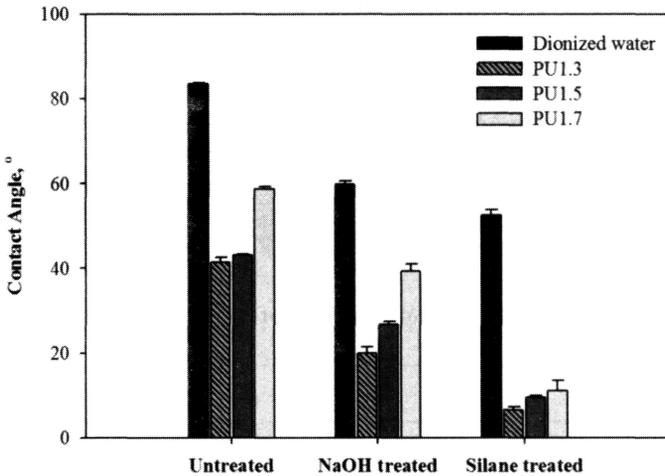


Figure 8: Contact angle of deionized water and PU adhesive resin on AA 2024-T3 substrates

Figure 8 shows the contact angle between liquid/resin drops and AA2024-T3 before and after surface treatments. From the figure, it is clear that the contact angle between deionized water and AA2024-T3 decreases after both of surface treatments. The contact angle between deionized water and silane treated AA2024-T3 shows the smaller contact angle, 52.5° compared to the one has undergone alkaline etching treatment ($\theta=59.8^\circ$). This indicates that the silane treatment gives better wettability to the substrate surface.

The similar behavior of the three PU adhesive resin/AA2024-T3 surface is the evident that the silane treated sample shows better wettability than the NaOH treated sample. Furthermore, high roughness favors the creation of

a well-defined drop spreading. This phenomenon might be due to chemical interaction between the resin and substrates [20]. The relationship between the contact angles and the surface roughness can be observed in Figure 9 where the contact angle is inversely proportional to the surface roughness. A complete wetting can be achieved by maximum contact between the substrate surface and the PU adhesive. This phenomenon contributes to enhance the adhesion between the substrate and the PU adhesive, hence affecting the strength and durability of adhesive bonds. Packham [21] and Bico [22] stated that the diffusion of the adhesive resin into the pores or voids on the substrate surface may contribute to the high strength.

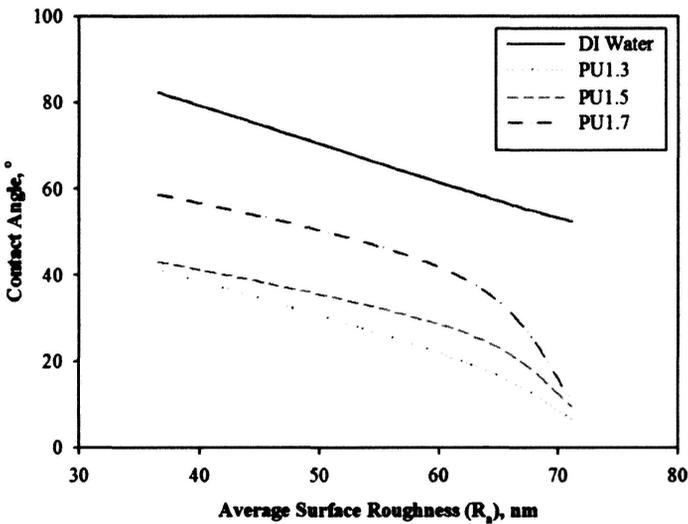


Figure 9: The effect of surface roughness of AA2024-T3 substrate on its wettability

Lap Shear Strength

The shear strength of PU adhesive bonding is shown in Figure 10. It is also shown the effect of NCO: OH ratio on adhesive performance. As the NCO content increases, the adhesive performance improves. The trend of increasing lap shear strength for substrates after surface treatment is very clear. The observed trend correlates solely to the wettability of liquid or PU adhesive resin on the Al surface.

The contact angle between liquid/Al surfaces is inversely proportional to shear strength of adhesive bonding. When the contact angle is smaller, the wettability is better. This is because of the strength of solid/liquid and liquid/liquid interactions are strong. In the hydrophilic or lyophilic case, the drop

either follows the topography, which generates an efficient decreasing of the contact angle, or it spreads inside the solid texture, and then co-exist with the solid filled with liquid [22]. Thus, increases the shear strength as the PU adhesive is set or cured.

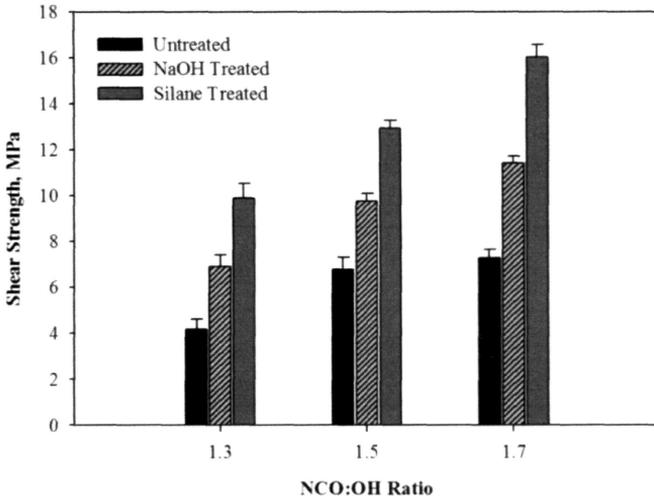


Figure 10: The effect of surface treatment on adhesive shear strength

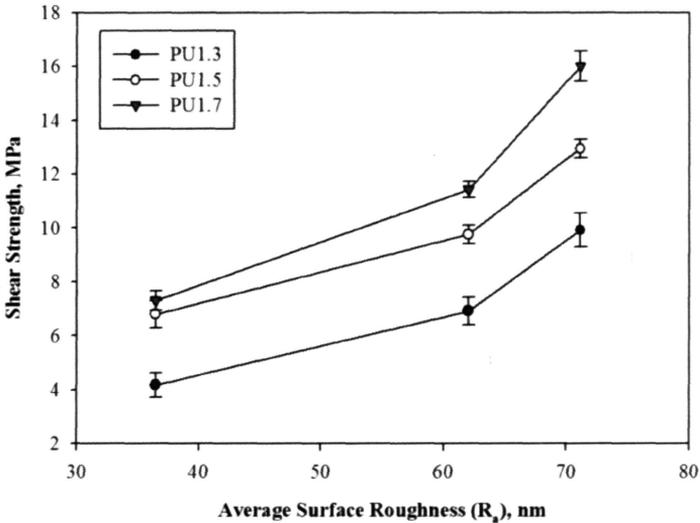


Figure 11: The effect of surface roughness on shear strength of adhesive bonding

The effect of surface roughness on shear strength of adhesive bonding is shown in Figure 11. The shear strength of adhesive bonding increases when the roughness increased. This is due to the mechanical interlocking theory of adhesion which states that good adhesion occurs only when an adhesive penetrates into the pores, holes and crevices and other irregularities of the adhered surface of the substrate, and locks mechanically to the substrate. Both surface treatments result in micro to nano-roughness (Table 1) on the substrate surface, which can improve bond strength and durability by providing mechanical interlocking. This is due to the surface area is increased by the surface roughness.

Conclusions

Different methods of surface treatment give different results of topography and surface roughness. The surface roughness also influenced the wettability of liquid/substrate surface. Adhesive bonding is found to display an interesting dependence on surface treatment. The wettability and surface roughness are main factors in contributing a good adhesion between adhesive resin and substrate. Silane treatment gives better wettability and adhesive bonding compared to alkaline etching surface treatment due to chemical attraction between siloxane film and the adhesive; and mechanical interlocking between adhesive and micropores on the substrate.

Acknowledgement

The authors would like to acknowledge Universiti Kebangsaan Malaysia and Ministry of Higher Education Malaysia (MOHE) for the financial support (FRGS/1/2012/SG07/UKM/01/1). Authors also thank Polymer Research Center (PORCE), Universiti Kebangsaan Malaysia for supplying the raw material used in this study.

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