The preparation and characterization of a hydrophobic surface on an aluminum substrate

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Abstract. The development of a hydrophobic surface on an aluminum substrate as a self-cleaning surface has been carried out. In this research, a hydrophobic aluminum surface was fabricated using a combination of boiling and STA modification technique which is environmentally friendly. The specimens in circular shape with a diameter of 14 mm were cut from a 1 mm thick aluminum sheet and were then cleaned using ultrasonic cleaner. Afterward, the specimens were boiled for 10 minutes in water. Following this, the specimens were immersed in STA-ethanol-deionized water solution at a temperature of 60^{o} C for 5, 10, 15 and 20 hours. Surface morphology, roughness, and hardness of the hydrophobic aluminum surface were examined. The surface morphology analysis presents the existence of rough microstructure on the coated aluminum surface. While the roughness value increase with the increase of immersion times, the surface hardness value does not appear to change significantly for immersion time up to 15 hours and slightly decrease for the longer immersion time. The water contact angle of 133^{o} and rolling angle of 13^{o} was achieved which indicates the ultra-hydrophobicity of the aluminum surface has been obtained.

1. Introduction

Today, researchers have developed material surfaces that have hydrophobic properties. A hydrophobic nature occurs if the contact angle of the water is around 90°-180° so that the repulsion of water becomes high which will cause the impurities found on the surface to be adsorbed on the water and slip down [1]. Water-resistant surfaces are influenced by two factors, namely the chemical composition of a surface and the roughness factor. The chemical composition of the surface of a material affects the hydrophobic nature because the nature of the water is not symmetrical or polar, so the hydrophobic surface must be nonpolar. The symmetry of a material can be obtained from chemical reactions. Hydrophobic properties also utilize low surface energy so that it can reduce the value of wettability on the solid surface and produce a surface that is hydrophobic [2]. Apart from the chemical composition, the structure-morphology also influences when there is an interaction between water and the surface, which results in an action-reaction force between the two. Using the principle of the law of equilibrium, the rougher the surface or the lesser the surface of the water touches the surface, the more equilibrium water is. With this equilibrium in water, it causes water to remain spherical so that it will not wet the surface.
In everyday life, hydrophobic can be used in terms of coating materials such as glass, electrical insulators, clothing, and others. The coating on this material will prevent material from dirt and dust sticking to the material that will cause stain and corrosion. Hydrophobic properties can also slow the occurrence of freezing of water on the surface of the material (anti-icing), thus inhibiting adhesion of ice or snow [3]. In addition, this hydrophobic nature will be able to reduce the disposal of food products and toiletries as

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leftovers that are still attached to food or toilet containers (soap, shampoo, cosmetics, perfume, etc.) [4]. This happens because of the excess of hydrophobic surfaces that have self-cleaning and drags reduction properties [5,6, &7].

Several techniques for obtaining a hydrophobic surface on solid material have been developed. There are include layer by layer and self-assembly technique, sol-gel techniques, etching technique, electrospinning technique, electrodeposion technique, and chemical vapour deposition technique [8]. Other researchers developed a technique for making hydrophobic layers by anodizing [9] and adding stearic acid (STA). Mokhtari, et al. [10] stated that making hydrophobic surfaces on aluminum 6061 can be carried out by a one-step anodization method and modified by low surface energy materials. However, the processes mostly use chemical solutions that are less environmentally friendly and expensive.
The description above shows that previous research has succeeded in developing the hydrophobic surface of various metals and non-metals involving a variety of hazardous chemical solutions, technologies that are not environmentally friendly, high costs and equipment that is difficult to obtain. Therefore it is necessary to develop a procedure for making a hydrophobic coating which is quite easy and no special equipment is needed, so that this method can be carried out easily and cheaply and environmentally friendly by providing treatment on the surface of aluminum alloy.

2. Methodology

The Preparation of the Hydrophobic Layer
Aluminum plates with a thickness of 1 millimeter were polished using silicon carbide paper from #400 to #1500. Then the plates were cut into round shapes with a diameter of 14 mm by water jet machine and cleaned using an ultrasonic cleaner by immersing it in acetone solution (Figure 1a). Following this, the plates were soaked in distilled water for about 5 minutes. After cleaning, the plates were boiled in boiling water for about 5 minutes and cooled to room temperature. The plates were then immersed in a ethanol-water solution containing STA 2.6% mass for 5, 10, 15 and 20 hours at a temperature of 60°C. The aluminum plates were then dried at room temperature for approximately 3 hours (Figure 1b).

The Testing of hydrophobic properties
The hydrophobic layer formed on the aluminum plate was examined including roughness, hardness, water contact angles, and the morphology of the surface. Water contact angles were measured with deionized water droplet at room temperature using a high magnification DSLR Nikon camera. The rolling angle measurement was conducted as follows. A water droplet was placed on the structured aluminum being treated, and the measuring plane of the sample was tilted until the droplet started to slide. The photograph of the rolling water droplet was taken with a digital camera. An electron microscope was used to examine the morphological structure of the plate surface.

3. Results and Discussion

The morphological structure of the aluminum surface being treated has been investigated using a scanning electron microscope (SEM) as shown in Figure 1. The figure depicts the surface morphology of aluminum plates before and after different treatment steps. Figure 1.a is the SEM image of the aluminum surface after sandpaper grinding then cleaning. It shows that the surface possesses irregular structure, some smoothie scratches, which is in the same way with the sandpaper grinding direction are observed. After the aluminum was immersed in boiled water, the surface is rougher than that of sand ground, and some pores were clearly found (Figure 1b). The observed pores were associated with the dissolution of partial boehmite which was generated from the interaction between aluminum and boiled water, into the water [11]. Figure 1.c shows the microstructure of the immersed aluminum surface in STA solution for 5 hours. It can be seen that STA has deposited into the surface in a not uniform small grain and flake structures. Different height level of the deposit was also observed. Along with the duration of immersion time, more STA deposits into the aluminum surface. At microscale the surface covered with a protrusion in flower-like structure about 4-20 μm in diameter, with the relatively smooth area remaining in between (Figure 1d). Figure 1 (e) shows higher magnification SEM images of single flower-like structure which consist of irregular deformed sheet morphology in 0.2 mm thick, randomly oriented to the surface. This structure behaves as pillar-like structure [12].

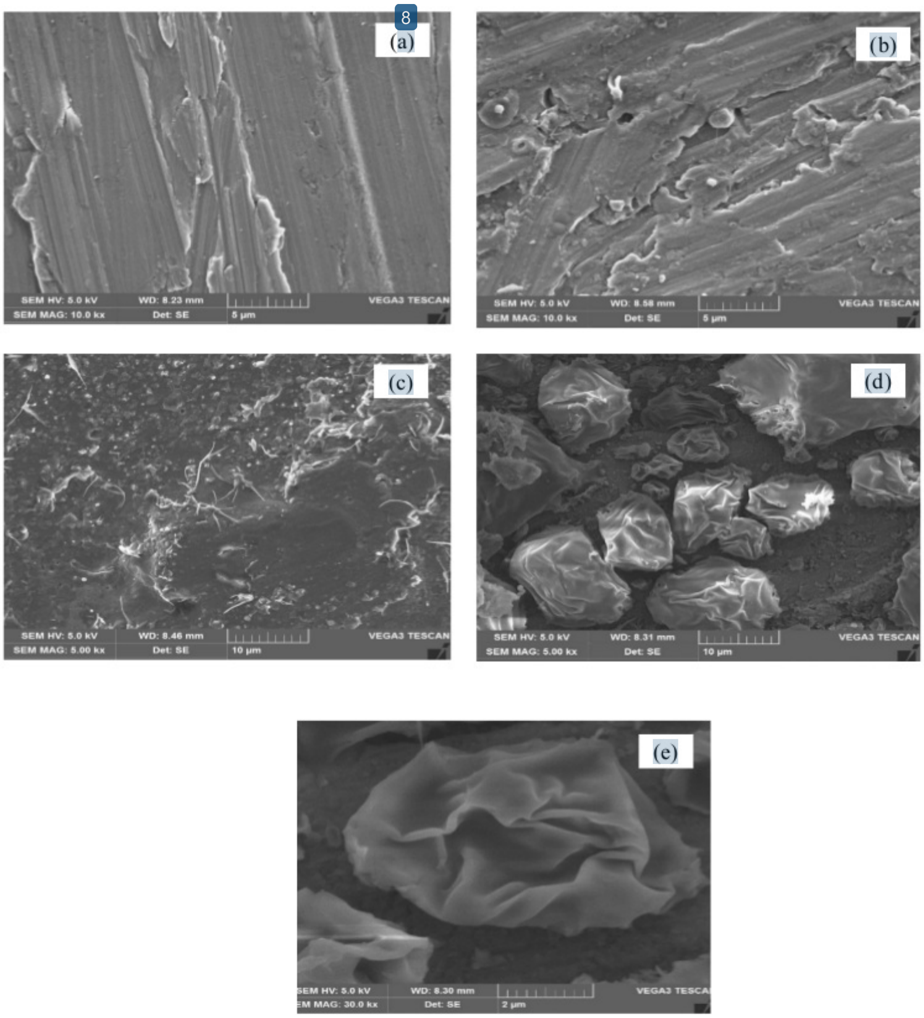

Figure 1. SEM images of the aluminum surfaces, (a) sandpaper grinded, (b), boiled, (c) immersed for 5 hours, (d) immersed for 20 hours. (e) image of the flower- like structure.

To investigate the effect of immersion time of STA treatment on the aluminum surface roughness, a series of experiments were conducted. The alteration surface roughness value of the aluminum after treatments is depicted in Figure 2. The treatment significantly changes the roughness values. After being boiled in the water treatment, the surface roughness (Ra) increased slightly from 0.609 to 0.825 μm. The existence of porosity due to the dissolution of boehmite into the water may be associated with this, as can be seen in Figure 1b. In general, the surface roughness value of the aluminum also increases with the increase of immersion time. The surface roughness from the immersion time of 5 to 10 hours increases doubly to 3.538 μm. It may suggest that the flower-like structure has formed. With the increase of immersion time, the flower-like structure appears more clearly. With respect to this the surface roughness increase moderately. The surface roughness test was in line with the morphological examination as depicted by the SEM images. Vickers hardness testing carried out to evaluate the hardness alteration during the treatment processes using 100 grf load. In general, the hardness values remain stable in the range of 51.5-53.4 HVN. Due to the low treatment temperature, the microstructure of the aluminum may not change.

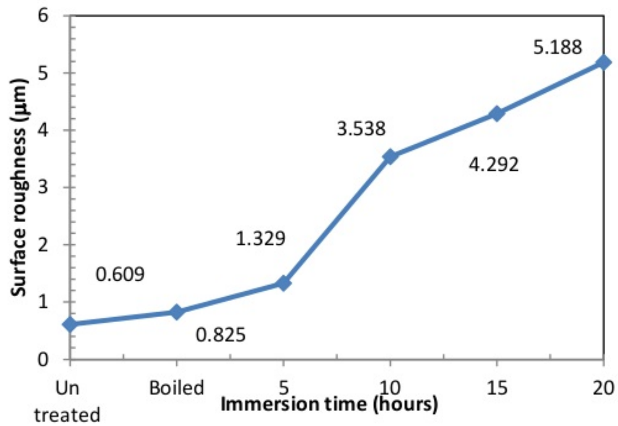

Figure 2. The relation between immersion time and surface roughness of the aluminum specimens

Aluminum is well known as hydrophilic material with a native oxidize layer in the surface. The surface wettability of the aluminum being treated has been studied by static contact angle measurement. The shapes of the water droplet on aluminum surfaces being treated were shown in Figure 3. Figure 3a and b show that aluminum surfaces are hydrophilic. The surface wettability had been converted from hydrophilic to hydrophobic by immersion in the STA solution (Figure 3.c and d) indicated by a quasi-spherical shape of the water droplet.

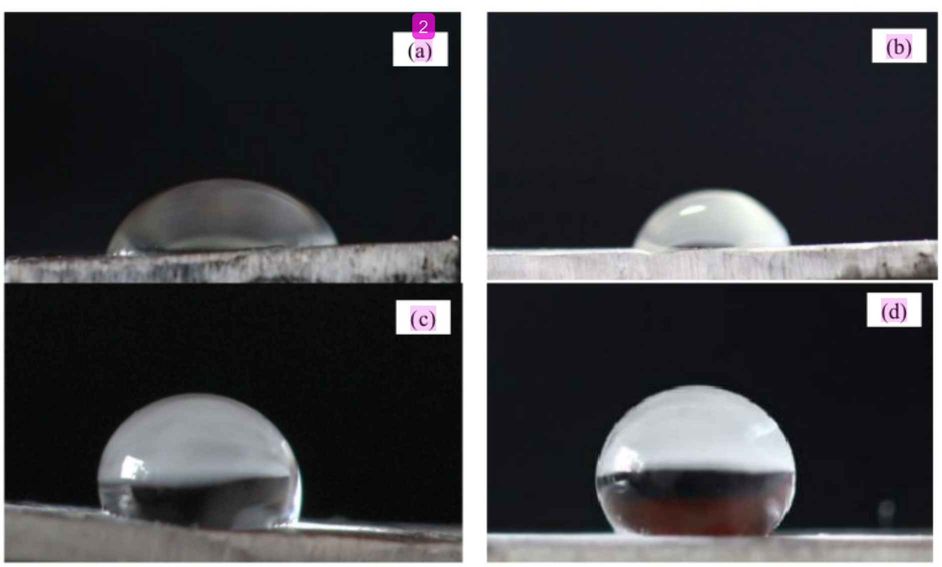

Figure 3. The shape of a water droplet on the aluminum (a) polished and cleaned, (b) boiled, (c) immersed for 5 hours, (d) immersed for 20 hours.

It can be found from Figure 4 that the contact angle of aluminum surfaces increase remarkably after each processing step. The water contact angle measurement resulted as polished and cleaned ultrasonically is measured of 57°. It is clearly categorized as a hydrophilic surface. This is comparable with the other researcher that found in the range of 45-82° [9, 13,& 11]. The contact angle slightly increases to 69° after the aluminum is boiled due to the existence of micropores on the surface in into the pores. This allows the boiled aluminum to unfollow the Wenzel's rule. In general, in accordance with Wenzel's equation, the surface roughness enhances the hydrophilic of the hydrophilic surface. Surprisingly, after the aluminum immersed into the STA solution for 5 and 10 hours, the water contact angle remarkably increases to 97° and 126° respectively indicating that the aluminum surfaces becomes hydrophobic.

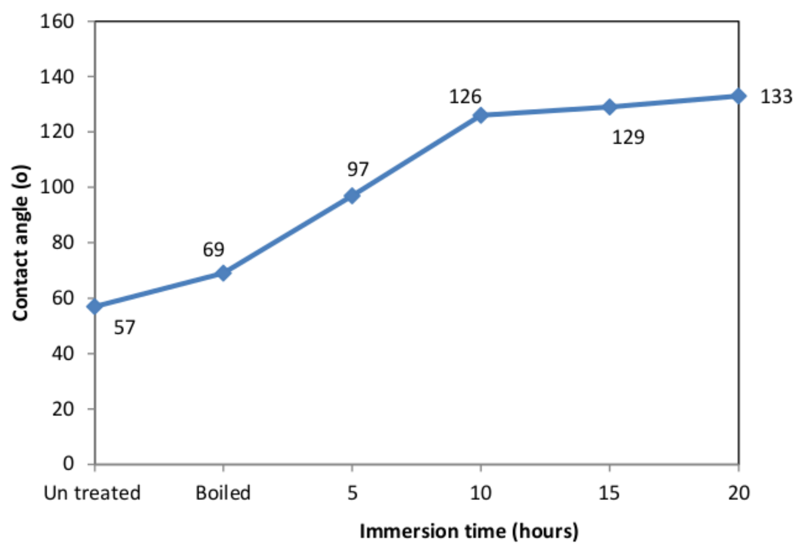

Figure 4 Relation of immersion time and water contact angle

The water contact angle then slightly increases to 129° and 133° with an increase of immersion time to 15 and 20 hours respectively. It is well understood that ultra-hydrophobic surfaces generally possess binary structures at both micrometer and nanometer scales [14]. In this research, the microscale flower-like structure and nanoscale morphological of irregular deformed sheet morphology constitute a hierarchical rough binary structure on the treated aluminum surface as depicted by Figure 1d and 2. As the roughness and pores reach an ideal diameter, the pores may easily act as a pillar to support droplet, and trap some air inside the pore. Water does not infiltrate into the pores of the surface and only interactions with micro/nano irregular structure. Therefore water is difficult to reach the bottom of the pores and to stick to the surface allowing the droplet to roll off quite easily. It has been found that the lowest rolling angles of 13° were obtained when the aluminum is immersed for 20 hours.

4. Conclusion

In summary, the ultra-hydrophobic aluminum surface has been successfully fabricated by immersion in an ethanol-water solution containing STA solution. Rough and porous structure in microscale and omit this pillar shapes were produced with the immersion time playing as an important role. The aluminum surface presents a high water contact angle of 133 and low rolling angle of 13 . This technique is expected to be a potential environmentally safe method to produce an ultra-hydrophobic surface.

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