

# Evaluation of Low Voltage Anodizing Process on the Aluminum Foil in Sulphuric Acid Solution

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## Keywords:

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Low voltage  
Anodized aluminum foil  
Microstructure  
Wettability

## ABSTRACT

*In this study, the anodizing process has been carried out on aluminum foil (0.20 mm thickness) by using a low voltage method. The aim of this work is to observe affect of low voltage anodizing in the surface nature of aluminum foil. The voltage was 5, 6, 7, 8, 12 and 15 V, and the duration of anodizing time was 30, 40, 50, and 60 min. The anodizing process was conducted by using 2 M aqueous sulfuric acid as an anodizing solution. The structural analysis of the oxide layer were performed by using 3D laser measurement optical microstructure. The maximum tensile load of anodized foil conduct by 5kN universal testing machine. The effect of low voltage anodizing on anodized aluminum foil microstructure, surface temperature, surface roughness, thickness, wettability, maximum tensile load, and current profile during anodizing were observed. The results obtained show that even at low voltages, the treatment has influenced the nature of the aluminum foil in terms of surface microstructure, oxide layer profile, maximum tensile load, current profile, surface temperature, wettability, and roughness.*

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## 1. INTRODUCTION

Metal anodizing have been used for decades in an effort to protect metals from corrosion attack and improve surface wear resistance [1-5]. Anodizing, allows to produce a thick artificial oxide layer, improvement in corrosion resistance, wear resistance and better surface mechanical properties. Anodizing is widely used to produce a protective conversion layer against wear in aluminum alloys. Anodizing also produces a porous layer that is important in industry, as it serves as the basis for surface finishing, painting, adhesion, electroplating, and certain decorative

functions [4] The anodizing process is an electrochemical process in which the metal surface undergoes physical and chemical changes through an oxidation mechanism by forming an anodic oxide layer that is able to inhibit the corrosion process. In the aluminum anodizing, oxidation occurs on the surface to form an  $Al_2O_3$  layer which is accompanied by a decrease in the aluminum content in the substrate. The layer formed has an effective adhesive bond with the substrate [6].

The anodizing is carried out by immersing the components in an electrolyte solution and connecting the aluminum to be anodized to the

anode of an electrical power supply. The power source that is widely used in the anodizing process is the DC type because it has advantages in controlling the anodizing parameters. After the power supply is turned on, the anions from the electrolyte will go to the anode and form an oxide layer. Characteristics and properties of the formed layer is highly dependent on the control of the anodizing process [7]. The structural characteristics of the aluminum oxide or alumina layer of concern are pore diameter, pore shape, distance between pores and thickness of the layer [8]. Meanwhile, the anodizing parameters that affect the structural characteristics of the coating include the type of electrolyte, concentration, temperature, duration and magnitude of the working stress [9].

There are two types of anodic aluminum oxide, Starting from the metal surface, the first is a thin, non-porous, hard and wear-resistant, electrically insulating layer, also called barrier-layer. The second is a thick oxide layer that is porous, and is usually fabricated to prevent corrosion, for which goal the pores are sealed by overgrowth of aluminum oxide at the pore mouths by keeping the surface in boiling hot water [3]. The porous structure appears during the anodizing process. Besides aluminum, several other metals that are often treated with anodizing are Ti, Zr, Mg, Nb, Si, Sn, W and Zn [3,10,11,12]. The most common form of aluminum oxide is amorphous aluminum oxide with a thickness of 2-3 nm formed when aluminum is exposed to air at room temperature [13].

Aluminum foil as an object of study in the anodizing process has been widely used [14,15,16]. Zhang et al 2015, conducted a study to fabricate superhydrophobic surfaces on aluminum foil via high-field electrochemical anodizing in oxalic acid aqueous solution [13]. The same thing was done by Song et al 2013, on anodized aluminum foil to produce a hydrophilic and hydrophobic surface layer using sodium chloride aqueous solution [17]. Likewise, Kikuchi et al 2014, carried out the anodizing process using aluminum foil using squaric acid to see the effect of the electrolyte solution and the anodizing time to the size of the oxide layer cells and the type of aluminum oxide phase formed [18] Another study was carried out by Stępniewski et al 2011, to develop nanoporous structures on aluminum foil using first anodizing method to see the effect of the anodizing process on the formation of the aluminum anodizing oxide

structure arrangement [9]. Aluminum anodizing oxide structure is considered to be a proper and reliable template for the fabrication of optics, optoelectronic and electronic devices [19] and also template for the fabrication of nanomaterials [20]. Study conduct anodized aluminum foil was also carried out by Zhu et al 2015, using the anodized aluminum foil to make electrolytic capacitors. The dielectric film layer is formed with a gamma aluminum oxide structure by combining mechanical attrition and anodizing process using a boric acid solution [21]. In addition, the use of various types of electrolytes that are not common in anodizing in the produce of porous aluminum oxide has also been carried out [22].

Study related to the use of the anodizing process on aluminum foil has been carried out [13-18]. However, to obtain a high porous aluminum oxide growth rate, relatively high voltage is usually set in the aluminum anodizing, see e.g. the works of Stępniewski et al 2011., 30-60 V [9], Kikuchi et al 2014., 100-120 V [18], and Akiya et al 2015., 150-170 V [23]. The use of a voltage that does not match with the type of electrolyte can cause a number of problems and difficulties, for example, the oxide layer is prone to cracking and burning, there is need for a cooling system during the anodizing, and the process stability is an issue as current fluctuates and is difficult to control. However, in applications such as anodic aluminum oxide membranes for nano-porous growth templates, the formation of layers by anodizing is expected to form more microvoids compared to other types of defects. One way to reduce the number of unwanted defects is by thickening the AAO layer which can be achieved in several ways including a longer anodizing duration [9], higher voltage and a more concentrated solution [24].

In this study, the use of anodizing with a low voltage range was carried out to observe the formed oxide layer. Variations on the duration of anodizing were also carried out to see the effect. Based on the description above, this study aims to look effect of the use of low voltage in the anodizing process carried out on aluminum foil, especially on the surface microstructure, maximum tensile load, electric current conditions during the anodizing process, surface temperature profile of the sample after anodizing, surface wettability and surface roughness.

## 2. RESEARCH METHOD

This study used commercial aluminum foil (Al: 99.5%) with dimension 60 mm x 30 mm x 0.2 mm. Before anodizing process, aluminum foil is cleaned using water and soap and then followed by ethanol ultrasonic cleaning. The electrolyte solution used is 2 M sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). The anodizing process was carried out in an anodizing bath with size, 30 cm x 20 cm x 20 cm. An aluminum plate with size 60 mm x 30 mm x 5 mm was use as cathode. The voltage was setting of 5, 6, 7, 8 , 12 and 15 V using DC voltage and anodizing duration was 30, 40, 50 and 60 min.

The combinations of voltage and duration for microstructure test was 5V (30 min, 40 min, 50 min and 60 min), and 60 min (5V, 6V, 12V and 15V), for tensile load test: 6V and 8V (30 min, 40 min, 50 min and 60 min), surface temperature test: 5V (30 min, 40 min, 50 min and 60 min), wettability test: 5V (30 min, 40 min, 50 min and 60 min), roughness test: 5V (30 min, 40 min, 50 min and 60 min) and current profile: 30 min (5V, 6V, 7V and 8V). Sequential selection of voltage aims to see whether there is a linear relationship.

The distance between the aluminum foil and the cathode is set at 30 mm, see Figure 1.

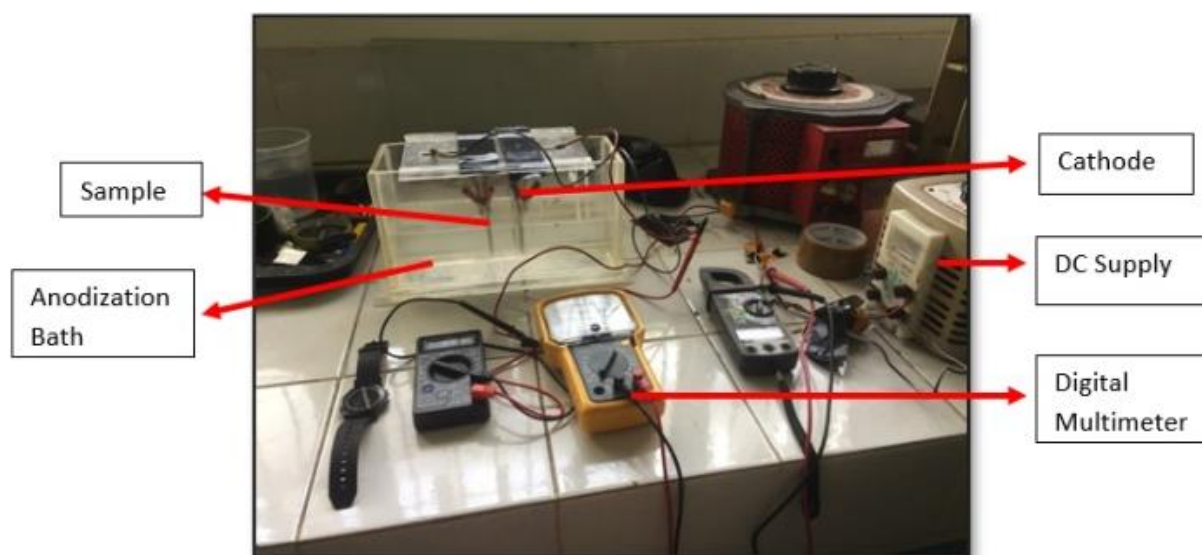


Fig.1. Anodizing experiment set up.

During the anodizing process, changes in current and voltage are measured using a digital multimeter. After the anodizing, the samples were removed from the anodizing bath and then washed gently with de-ionized water and then dried using an air dryer.

The anodized samples then observed using Olympus 3D laser optical microscope with profilometer feature to see the microstructure, surface texture and surface roughness. Tensile test on samples after anodizing was carried out using a 5 kN Shimadzu to measure the maximum tensile load after anodizing. The tensile test sample has dimension 50 mm x 30 mm. A hole with a diameter 10 mm was made in the center of the sample so that the sample can break off in the middle, see Figure 2.

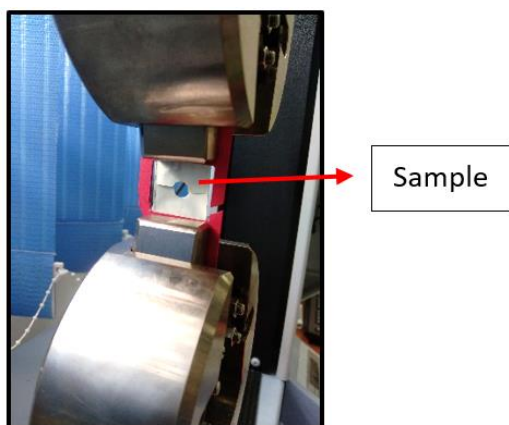
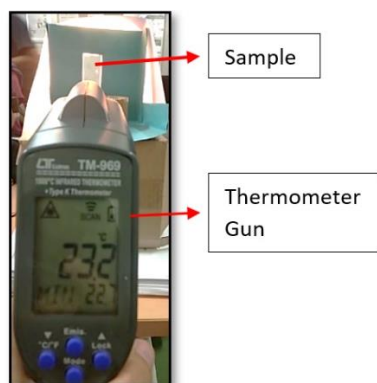


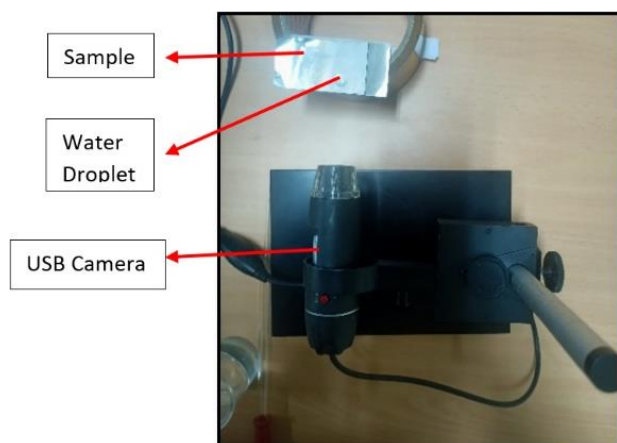
Fig. 2. Tensile load measurement.

In order to measure the surface temperature of the anodized sample, a heating lamp was used, the sample is placed in front of the heating lamp with a distance of 40 mm. the heating lamp temperature was 40°C The temperature measurement on the back surface of the sample was carried out using a thermometer gun and measured for 5 min, see Figure 3.



**Fig. 3.** Surface temperature measurement.

Wettability testing is carried out by dropping water on the surface of the sample and then measuring the contact angle between the surface of the sample and the water droplets, conducted by USB camera to capture contact between anodized surface and droplet water, see Figure 4.



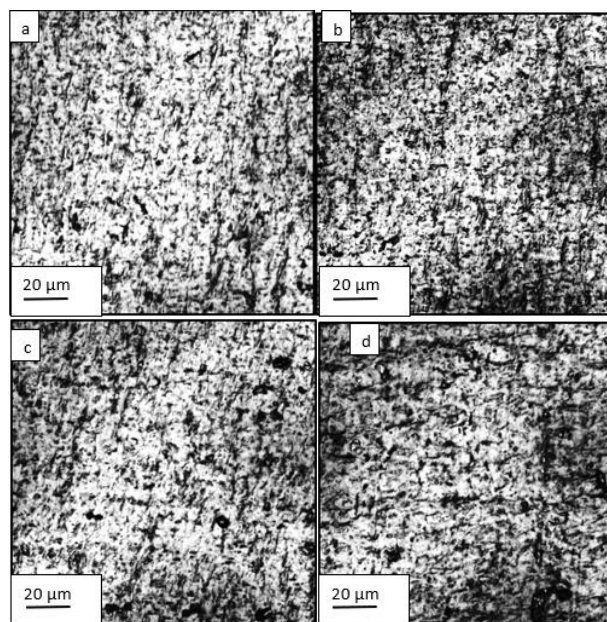
**Fig. 4.** Set up of wettability test.

### 3. RESULT AND DISCUSSION

Figure 5 shows the surface microstructure of aluminum foil after anodizing process at 5 V with variations in anodizing time duration. The layered structure has a number of voids, cavities, holes, dimples and microcracks scattered over the surface. The formed layer changes structurally after anodizing. This means that using a low voltage on the aluminum foil sample still forms the oxide structure which contain cavities, voids, holes and also microcrack.

Figure 5 also shows that the difference in anodizing duration causes differences in surface morphology. In Figure 5a with a duration of 30 minutes, the surface structure contains cavities and voids with a flatter surface contour with a few protrusions. The surface structure was slightly

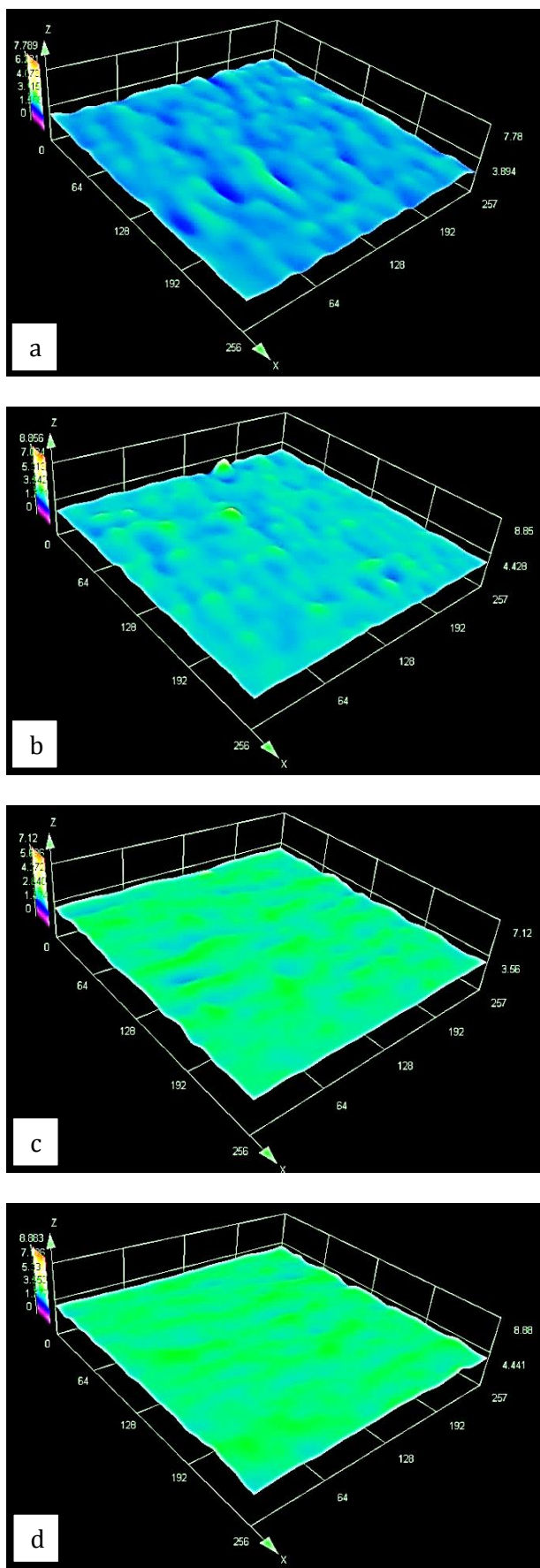
different at the anodizing duration of 40 min, 50 min and 60 min (figures 5b, 5c and 5d). The cavities, voids and microcrack are more easily visible, the previously flat surface becomes more protruding. The longer the duration of anodizing causes more cavities, voids and protrusions.



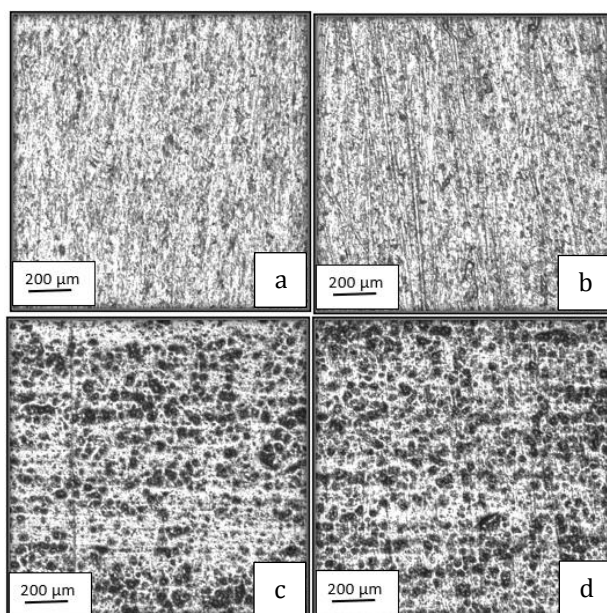
**Fig 5.** Photo of the microstructure of the surface of the anodizing aluminum of 5 V with anodizing duration: (a) 30 min, (b) 40 min, (c) 50 min, (d) 60 min.

Figure 6 shows changes in texture and layer thickness after anodizing at different durations at 5V voltages obtained using a 3D laser microscope. It is seen that the duration of anodizing exerts an influence on increasing the thickness of the aluminum oxide layer. The longer duration of anodizing causes the oxide layer to become thicker. However, the thickness of the layer does not look uniform causing an uneven texture of the oxide layer.

The layer thickness can be made thicker by increasing the anodizing current, as well as the concentration of the solution. The thickness of the layer also depends on the amount of the electric charge and the temperature of the electrolyte. Using a higher current density and keeping the duration of anodizing and electrolyte temperature constant will cause the oxide growth rate layer to increase significantly. Higher current density affects the speed of the anodizing process which is directly related to the growth and form of the oxide layer [22]. There is a linear dependence between pore size on anodizing voltage and thickness on anodizing time [14].



**Fig. 6.** Oxide layer profile for 5 V anodizing sample: a. 30 min; b. 40 min; c. 50 min and d. 60 min.



**Fig. 7.** Microstructure of the anodized aluminum surface for 60 minutes: (a) 5 volts, (b) 6 volts, (c) 12 volts and (d) 15 volts.

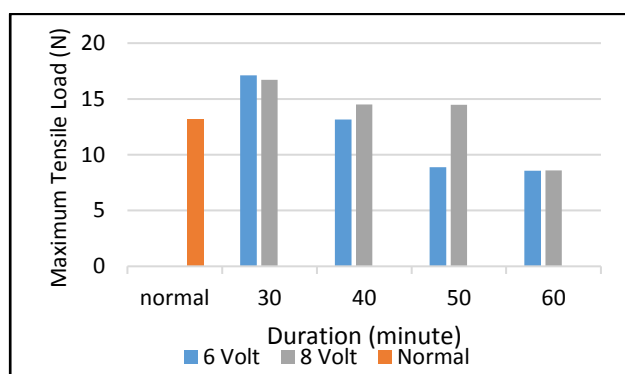
Figure 7 shows the results of the observation of the microstructure on the surface with variations in the anodizing voltage with an anodizing duration of 60 min. Similar with figure 5, figure 7 also shows the surface morphology consisting of voids, cavities, holes, dimples and microcracks (micro-morphology). The differences appear more pronounced with changes in voltage. At low voltages (5 V and 6 V) the sizes of void and holes are smaller and spread evenly. The increase in voltage causes the surface structure to be more dominated by voids and cavities which are larger and the density of voids decreases. This means that the use of low voltages can produce a layer structure similar to that commonly found at higher voltages, even on aluminum foil samples, specially with a longer anodizing duration.

The low anodizing voltage described by Pastore et al 2011, will cause the rate of growth of the oxide layer and formation of voids to be low. Pastore used a voltage of 6 V and found that the oxide layer formed has a higher high aspect ratio, thus forming an ultrathin membrane. Ultrathin layer can be formed in 60-120 seconds [24]. However, the amount of voltage used also depends on the type of electrolyte solution.

Liu et al 2006, revealed that the effect of voltage on anodizing produces the characteristics of the layer formed due to the destruction of the

natural oxide layer by high electrical resistance so that a new porous oxide layer is constructed [25]. The magnitude of the electric current during the anodizing process can indicate the speed of metal transformation into an oxide layer. The low anodizing current means the transformation speed is also low. As previously described, the rate of layer formation will increase with increasing anodizing voltage [26-27]. The consumption of aluminum into the oxide layer will be faster at higher voltage. However, the nature of natural oxide can be the reason for the current instability during the process of forming a new oxide layer by the anodizing process. In addition, according to Zhu et al 2015, in the conventional anodizing process, if there is a surge in the current, it will produce an oxide layer that is rich in defects that arise due to electrical instability [21]. According to Knörnschild et al 2015, there is a self-localizing mechanism that concentrates ionic currents in the spot-like area that occurs continuously and consumes the whole metal thickness under the spot for high current densities [28].

Tensile testing was carried out to validate the presence of an oxide layer on the surface. This layer will cause a change in the ability to withstand the maximum tensile load of the sample.



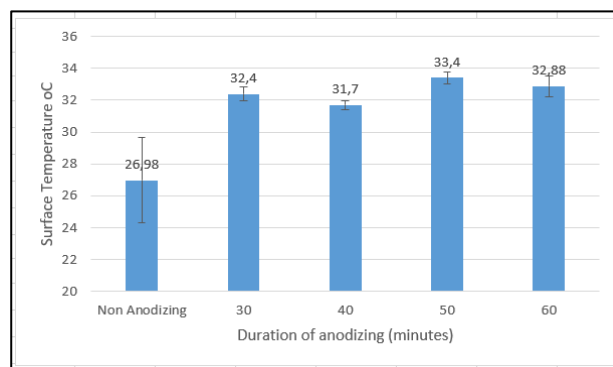
**Fig. 8.** The effect of duration of anodizing on the average maximum tensile load.

Figure 8 shows the maximum load comparison between samples without anodizing (normal) and samples with anodizing. Anodized sample have a maximum load value which is above the sample without anodizing see figure 8, especially at 8 volts except for anodizing time of 60 min. For samples of 6 V, duration 30 min and 40 min, the maximum load value is also above the samples

without anodizing. From Figure 8, it can also be seen that the longer the anodizing process makes the tendency for the maximum tensile load to decrease. However, a higher voltage has a better tensile load capability. The tendency of the sample's ability to withstand loads to decrease with increasing anodizing duration can be understood by relating the presence of the oxide layer as an anodizing product. This layer contains many pores and triggers cracks.

Samples with a better maximum tensile load could be caused by the layer structure not yet being a trigger for failure. Failure initiation can come from aluminum foil that has not transformed into aluminum oxide or it can also come from a barrier structure that has amorphous characteristics. In the anodized structure there are three different forms. First, the structure in the core which is still a metal structure, second is the barrier structure that covers the metal structure with amorphous characteristics and third is the outermost structure in the form of hydrated and amorphous.

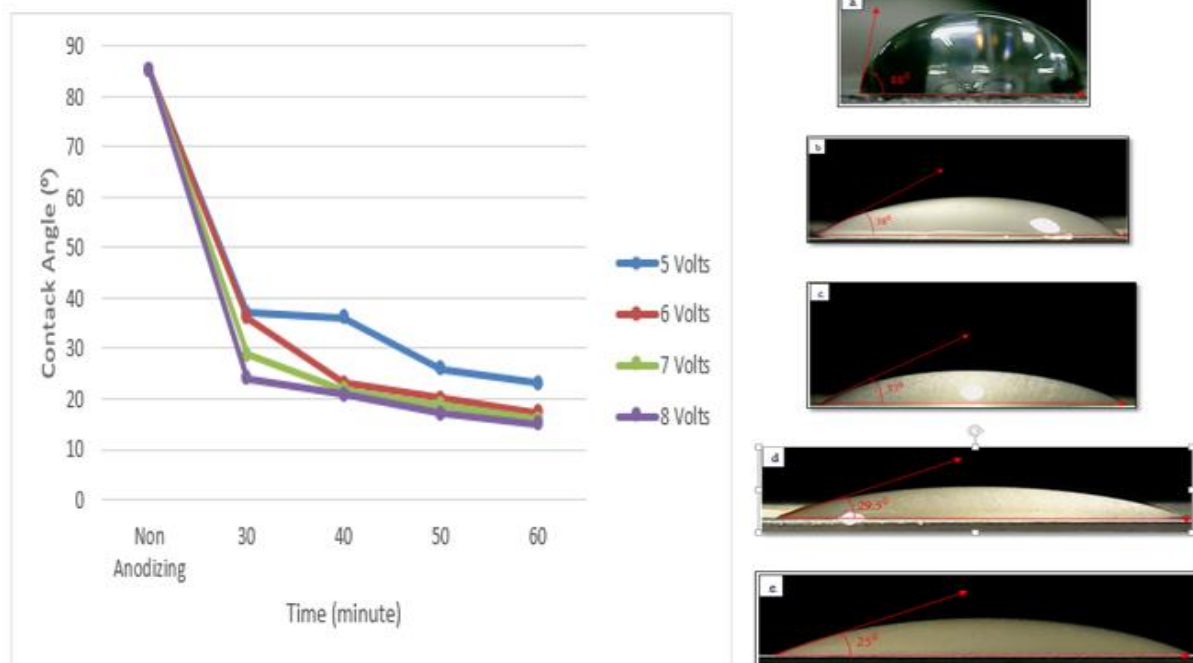
A similar narrative was uttered by Zhu et al 2015, that the anodized aluminum oxide layer is composed of an inner layer with high crystallinity, in which crystallites aggregated, large in size and rich in number. Meanwhile, on the outer layer with low crystallinity, in which crystallites dispersed, small in size and poor in number [21]. This phase transformation produces shrinkage, a number of voids, cavities and slit are distributed across the whole anodized oxide film. According to Erdogan et al, to increase the strength of the oxide layer significantly in the anodizing process, it is necessary conduct the etching cycle on the first anodizing treatment [19].



**Fig. 9.** Surface temperature average of anodized (5 V) and non anodized sample.

Figure 9 shows surface temperature of anodized (5 V) and non anodized sample, The surface temperature of the sample without anodizing has an average temperature of 26.9 °C meanwhile the surface temperature of the anodized sample is in the range of 30.9 °C to 33.9 °C. The difference in

surface temperature indicates that the amount of heat energy dissipation is also different, this means that there has been a change in the composition of the aluminum foil due to the anodizing process which indicates the formation of an oxide layer on the surface.

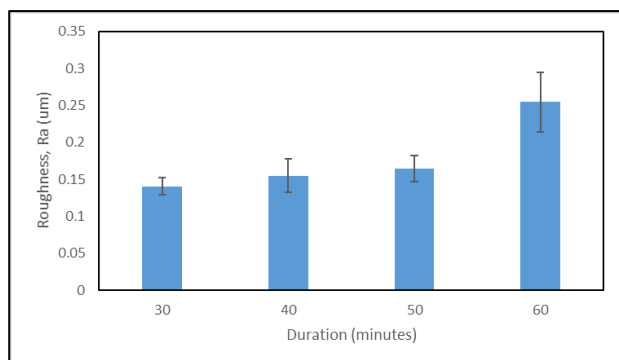


**Fig. 10.** Contact angle on the surface oxide layer with different voltage and duration; b. side view image of water droplets on the surface; a without anodizing, b-e anodizing at 5 V with different durations.

The properties of oxide layer are very different from that of aluminum, the aluminum oxide layer has higher specific heat capacity than aluminum, high temperature resistance, high hardness and low thermal conductivity. The duration of anodizing also causes a temperature difference surfaces with a tendency to higher temperatures for longer durations.

Measurement of the contact angle of the anodized samples was carried out to see changes in surface properties on wettability. Wettability can be thought of as a measure of the hydrophilic nature of a surface. A surface with excellent wettability means it has hydrophilic properties. The surface is categorized as hydrophilic if the contact angle is below 90°. From the results of the tests performed (figure 10), the maximum contact angle for anodized conditions was 38° (at 5 V, 30 min) while the minimum value was obtained at 15° (8 V, 60 min) see figure 10. Meanwhile, for samples without anodizing, the values obtained 86°. The

effect of anodizing voltage is seen at the contact angle where an increase in voltage causes a tendency to decrease the contact angle as well as the duration of anodizing which causes a decrease in contact angle with longer anodizing. Figure 10b shows a visual representation of the contact angle between the surface and the liquid at 5 V with different durations. The oxide layer formed on the surface of the aluminum foil shows better adhesion and better bonding properties. This property is formed due to a decrease in surface energy so that the interaction between the surface and the liquid becomes better. The decrease in surface energy is greatly influenced by the texture of the oxide layer which consists of pores, voids and small cavities. The use of low voltage in the aluminum foil anodizing process carried out in this study shows that a hydrophilic surface can be produced. The results obtained are in line with studies conducted by Song et al 2013, which even produced surfaces with superhydrophilic properties (17).



**Fig. 11.** Relation between anodizing duration at 5 V and surface roughness level.

Figure 11 shows the relationship between anodizing duration and roughness at 5 V. The high roughness can be caused by the morphology of the layer containing voids and pores. Longer anodizing duration produce greater number of micropores and voids and spread evenly on the surface. The presence of these micropores has an effect on the level of surface roughness and wettability. Surface roughness and surface energy are two important factors that are closely related in forming wettability properties. According to the Wenzel model, the surface contact angle of a rough surface will decrease if the surface roughness value results in a contact angle smaller than 90 degrees [29]. The level of surface roughness has correlated with adhesion force that the adhesion force decreased with increasing root-mean-square (RMS) roughness [30].

#### 4. CONCLUSION

Aluminum foil is anodized in low voltage using sulfuric acid as an anodizing solution to see its effect on the oxide layer formed. The Results can be summarized:

1. Observation of the anodized surface shows that at the same voltage, a longer anodizing duration indicates that cavities, voids and holes are more easily visible. The number of voids, cavities and holes appears to increase and the surface looks more wavy when compared to shorter anodizing durations. In addition, a longer anodizing duration leads to an increase in the thickness of the oxide layer. The results of microstructure observations also concluded that voltage has a more dominant effect than the duration of anodizing. The structure of the layers is significantly different. The size of voids, cavities and holes increases with

increasing anodizing voltage, this is found at 12 volts and 15 volts when compared to void, cavities and hole sizes at lower voltages (5 volts and 6 volts).

2. The anodized sample showed the highest tensile load value of 17.12 N at 6 volts (duration 30 minutes), an increase of 29.6% compared to sample without anodizing. The minimum tensile load obtained at 8.55 N at 6 volts (duration 60 minutes) decreased by 35% compared to non anodized.
3. The surface temperature of the sample after anodizing is higher (4.7°C-6.5°C) compared to non anodizing due to a decrease in heat dissipation after the sample is anodized.
4. The surface of anodized sample becomes more hydrophilic which is characterized by a decrease in the contact angle.
5. By increasing the duration of anodization increases the roughness of the layer, the minimum roughness is 0.14 μm (duration 30 minutes) and the maximum roughness is 0.25 μm (duration 60 minutes).

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