Characterization of Anodizing Process on Aluminum Series 6 with Variable Voltage

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Abstract. Anodizing is one way of coating oxidation on aluminum, which is carried out by anodic oxidation at room temperature with the help of an electric current so that a chemical reaction occurs so that a layer is produced that can protect the metal from the effects of corrosion. The objective of this study was to determine the characterization of variations in electric voltage on surface hardness in the anodizing aluminum series 6. Specimens were made with dimensions of 50 mm × 25 mm with a thickness of 10 mm, then the cleaning process was carried out with a solution of sodium carbonate (Na_2CO_3) with a concentration of 50 gr/100 ml of reverse osmosis (RO) water. The etching, desmut, and anodizing processes were continued with a solution concentration of 400 ml of sulfuric acid (H_2SO_4) and 600 ml of RO water. The electric voltage variables used were 20, 24, and 28-V with a current of 2 A and an immersion time of 10 min. The results of the highest hardness test on a variable voltage of 20-V with a Vickers hardness value of 59.117 VHN. This proves that anodizing treatment on the surface of aluminum material with a variable voltage of 20 V will produce a high Vickers hardness value. These results indicate that the toughness of the material on the anodized specimen is also affected by the magnitude of the electric voltage.

Keywords: Anodizing · Aluminum 6 series · Variable voltage · Vickers hardness

1 Introduction

Approximately 8% of the earth's surface is composed of aluminum metal, which ranks third among other metals [1]. Aluminum is a light metal with excellent wear and corrosion resistance as well as excellent electrical conductivity in the industrial world. In addition, aluminum is a metal with excellent mechanical properties and welding capabilities. In addition, aluminum has advantages such as low density, corrosion resistance, low cost, good electrical and thermal conductivity, and oxidation resistance [2, 3]. Ab Rahim

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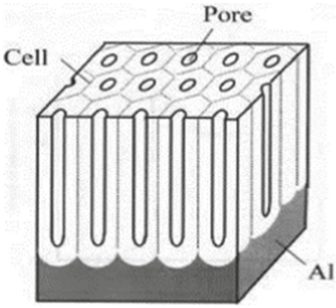

Fig. 1. Schematic display of the AAO membrane [11].

et al. (2015) and Kvande & Drabls (2014) report that aluminum is widely used in the industrial world to support the fabrication process and is widely applied by various material companies around the world [4, 5].

The anodizing process plays an important role in the manufacturing industry, including the machinery industry, the assembly of motor vehicles and bicycles, the aircraft industry, and the production of other goods. The purpose of anodizing is to oxidize the aluminum surface so that it is protected from the destructive influence of the environment that causes corrosion. Additionally, the anodizing process produces a more attractive, smoother, textured, and colored aluminum surface that is resistant to surface friction [6]. An anodizing process can be used to increase the hardness of aluminum. The anodizing process is the process of forming an oxide layer on metal by corroding a metal, particularly aluminum, with oxygen (O_2) taken from an electrolyte solution of sulfuric acid (H_2SO_4) that is used as a medium, thereby forming an oxide layer [7]. One benefit of the anodizing process is that it can make an oxide layer that is harder than the material of the workpiece.

In material engineering, the anodizing process is often applied to aluminum, so it is often referred to as aluminum anodizing, which is an electrochemical aluminum coating process by converting aluminum into aluminum oxide (Al_2O_3) on the surface of the material to be anodized [8]. Anodized aluminum displays an unusual porous structure. It is widely accepted that the anodizing film layer has a two-layer structure, namely a non-porous barrier oxide and a porous outer oxide. A thin barrier layer (10–100 nm) is found at the bottom of the pores at the metal interface [1–5]. Hu et al. (2015) say that the structure of porous anodic aluminum oxide (AAO) is made up of a grid of regular hexagonal cells with cylindrical pores with diameters from 25 nm to 0.3 m and depths greater than 100 m [9].

To reduce the time required to prepare the tubular membrane through the AAO orifice. This study improves the manufacturing process using one-step anodization. The AAO through-hole tubular membranes obtained were straight nanotubes with a thickness of 48 m and an average lower pore size of 109 nm [10] that shown in Fig. 1.

The AA2024-T3 aluminum anodizing process employs a 10% sulfuric acid solution with current densities ranging from 1.5 A per square decimeter to 3 A per square decimeter to 4.5 A per square decimeter and immersion times ranging from 30 to 60 min. This study aimed to characterize the effects of variations in electric voltage on the surface hardness of anodized aluminum series 6.

2 Related Works

The results of the study [12] on the aluminum alloy plate AA2024-T3 showed that the hardness value was influenced by current density and anodizing time with varying values. The larger current so, the shorter has the optimal anodizing time. The results obtained are that the longer anodizing time, the aluminum oxide layer will be higher [13].

The best anodized layer thickness of 89.6 m was obtained from the hard anodization study with a temperature of 9 °C, oxalic acid of 2 wt%, and anodization time of 60 min. The hard anodization study with a temperature of 5 °C, oxalic acid of 1 wt%, and anodization time of 30 min yielded the highest anodized layer hardness of 515 HV [15]. Another anodizing process is carried out with a variation of the current strength of 0.75, 1.5, 2, 25, and 3 A with variations in immersion time of 30, 40, 50, and 60 min in Fig. 2. Tests carried out include the microstructure and the thickness of the oxide layer. The test results show that the addition of strong currents in the anodizing process affects the thickness of the oxide layer [16, 17]. The oxide layer thickness, surface hardness, and wear resistance of 6061 aluminum are quality characteristics.

Experiments were carried out using orthogonal arrays L9 (3^3). The results showed that the parameters that had a significant effect were the type of solution, the duration of immersion time, and finally the magnitude of the voltage. Experimental observations by Zhang et al., (2021) in Fig. 3 assume that oxygen gas can play an important role in the anodizing process. With the formation of hydrogen gas at the cathode, the pressure in the closed container is gradually increased so that the oxygen bubbles cannot break the layer exposed to the anions. The growth of anodic oxide is sped up by the increase in the size of the oxygen bubbles in the channel.

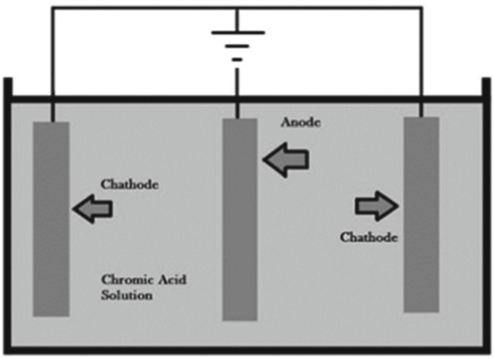

Fig. 2. Anodizing process [14]

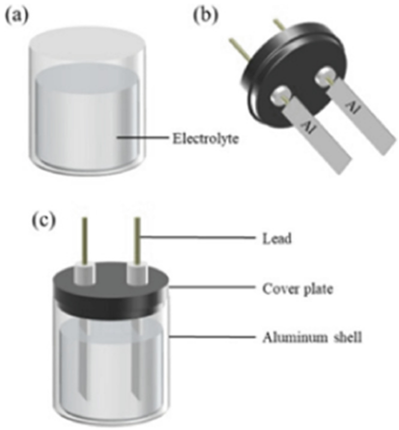

Fig. 3. Anodizing in a closed container: (a) aluminum shell, (b) cover plates and leads, (c) sealed container [18].

3 Methods

In this study, the aluminum anodizing process used 3 specimens with dimensions of 50 mm × 25 mm × 10 mm. The number of specimens of the material was tested using the Vickers hardness test with a variable voltage of 20, 24, and 28 V. To figure out the Vickers hardness value, each sample with a variable voltage was tested at three random points.

3.1 Anodizing Process on Aluminum 6xxx Series

The stages of the anodizing process in Fig. 4 use the 6xxx series aluminum material. Series 6 aluminum alloys are aluminum alloys with magnesium and silicon as the main alloying elements and the Mg2Si phase as the strengthening phase. Series 6 is a heat-treated, toughenable aluminum alloy. This alloy has the advantages of moderate strength, high corrosion resistance, no tendency to stress corrosion cracking, good welding performance, unchanged corrosion performance of the welding zone, good formability, and process performance [19].

The anodizing process includes: (1) Cleaning, which involves removing any dirt that has stuck to the aluminum surface with a pure detergent solution of sodium carbonate (Na_2CO_3) at a concentration of 50 g/l. (2) Etching is the process of getting rid of the oxide layer on the surface of the aluminum that cleaning or rinsing can't get rid of. With a 100 g/l concentration of caustic soda (NaOH), this process is also used to get a more even and smoother surface on the workpiece. (3) Desmut is a process that functions as a cleaning of black spots caused by the etching process. The solution used is a mixture of phosphoric acid (H_3PO_4) 75% plus sulfuric acid (H_2SO_4) 15% and nitric acid (HNO) 10%. (4) In the rinsing step of anodizing, RO water is used to remove any chemicals that are stuck to the aluminum surface after the cleaning, etching, and desmut steps. (5) Anodizing, also called "anodic oxidation," is a coating process that uses electricity to turn

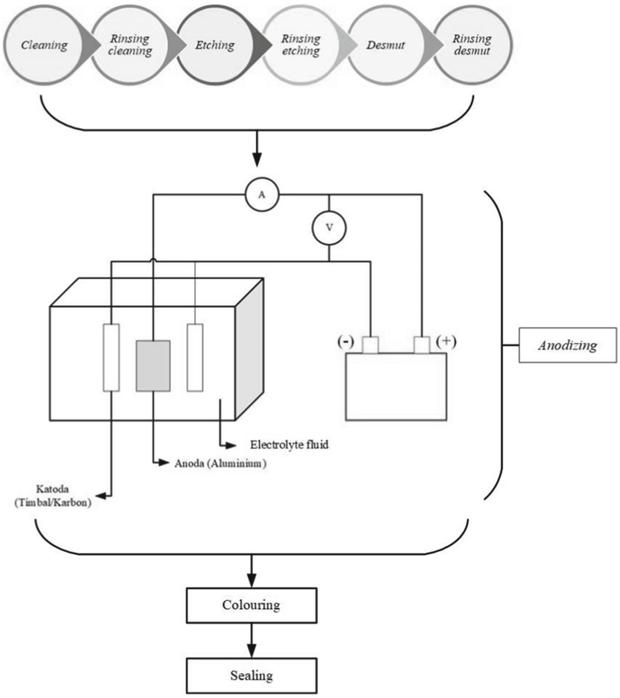

Fig. 4. Scheme of anodizing process on aluminum 6xxx series.

aluminum into aluminum oxide. The solution used is sulfuric acid with a concentration of 400 ml/l.

The current used is 3 A, for 10 min with a variable voltage of 20, 24, and 28-V. The metal or workpiece is mounted on the anode (+) and as a cathode (−) can use a sheet of tin or aluminum and carbon. (6) The coloring and sealing process is the final stage of the anodizing process. This staining gives color to the pores of the oxide layer that forms after the anodizing process, while sealing closes the pores of the oxide layer or keeps the color of the oxide layer from fading after the coloring process.

3.2 Vickers Hardness Test

Using the Vickers hardness tester, the mechanical properties of the anodizing material are examined. The objective of the surface hardness test on anodized aluminum is to determine the surface hardness value. This test employs the macro-Vickers hardness method with a 30 kg load (Fig. 5).

$$VHN = \frac{P}{A}$$

$$VHN = \frac{P}{\frac{d^2}{2\sin\frac{136^\circ}{2}}}$$

Fig. 5. Vickers hardness equipment.

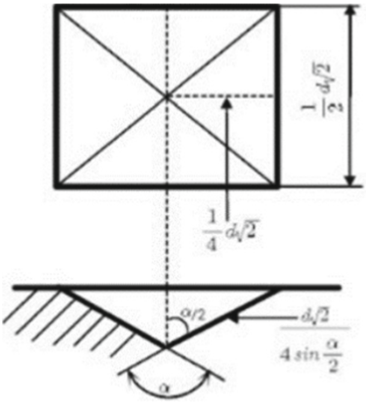

Fig. 6. Diamond pyramid indenter shape [2].

$$VHN = 1,854\frac{P}{d^2} \quad (1)$$

The Vickers method in Eq. (1) is carried out by pressing the test specimen with a diamond pyramid indenter in the form of a rectangular base and a large surface angle of 136° that shown in Fig. 6. The Vickers hardness number is found by dividing the test load by the trace's surface area.

4 Result and Discussions

At the stage of the formation of the oxide layer through the anodization process, several chemical reactions occur. At the anode (aluminum), it will be oxidized through the following reactions according to Eqs. (2) to (4):

$$2Al^{3+} + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \quad (2)$$

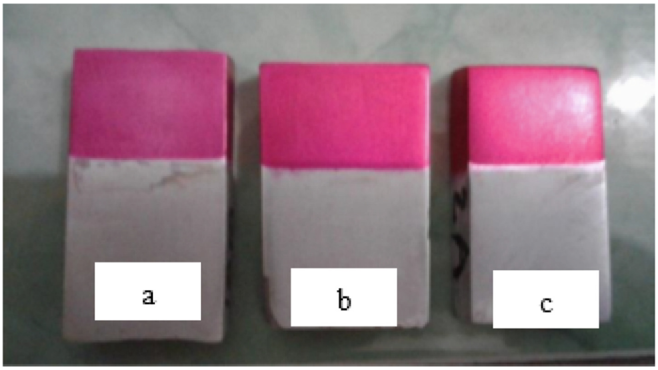

Fig. 7. Anodizing test specimens with a variable voltage (a) 20-V, (b) 24-V, (c) 28-V.

$$2Al^{3+} + 6OH^{-} \rightarrow Al_2O_3 + 3H_2O + 6e^{-} \quad (3)$$

$$2Al^{3+} + 3O^{2-} \rightarrow Al_2O_3 + 6e^{-} \quad (4)$$

Furthermore, the electrolysis of water will cause an electrolyte oxide reaction to occur and at the cathode will experience a hydrogen reduction reaction as in Eqs. (5) and (6) below:

$$H_2O \rightarrow OH^{-} + H^{+} \quad (5)$$

$$2H^{+} + 2e^{-} \rightarrow H_2 \quad (6)$$

In the end, the 6xxx series aluminum metal will undergo the electrochemical reaction in Eq. (7) (Fig. 7).

$$2Al^{3+} + 3H_2O \rightarrow Al_2O_3 + 6H^{+} + 6e^{-} \quad (7)$$

The anodized layer has a different structure from the naturally occurring oxide layer, where the layer has a porous hexagonal pillar structure that has unique characteristics that improve the mechanical properties of the aluminum surface [20]. In general, the oxide layer resulting from the anodizing process has hard characteristics comparable to sapphire, being insulative and resistant to load, transparent and free of debris.

Surface hardness testing aims to compare the value of the surface hardness of the raw material with the surface after the anodizing process. This test was carried out using the Vickers Hardness Number (VHN) method with a load of 30 kg. The results of these tests are then calculated to determine the level of hardness on the surface of the anodized aluminum 6xxx series with different variations in electrical voltage. Testing of three test points on each specimen with a distribution of one point on the specimen (middle), the average hardness value of the three points is taken in order to validate the surface hardness after the Vickers hardness test is carried out (Fig. 8, Table 2).

In Table 1, the Vickers hardness test was carried out on a series 6 raw material specimen, showing a hardness value of 54.58 VHN. While the comparison of the surface

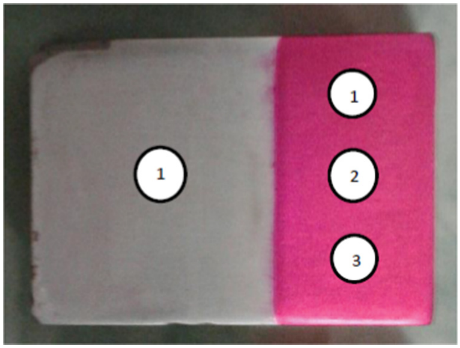

Fig. 8. Random distribution of anodizing specimens.

Table 1. Raw material hardness test results.

Variable	Test Distribution	∑D	Vickers Hardness Number (VHN)	∑ Vickers Hardness Number (VHN)
Raw material	Random	0,99	56,75	54,58
	Random	1,04	51,92	
	Random	1,01	55,07	

Table 2. The results of the hardness test after the anodizing process with a variable voltage

No	Variable	Test Distribution	∑D	Vickers Hardness Number (VHN)	∑ Vickers Hardness Number (VHN)
1	Anodizing with electrical voltage 20 V	Random	0,98	58,51	59,117
		Random	0,97	59,11	
		Random	0,97	59,73	
2	Anodizing with electrical voltage 24 V	Random	0,97	59,73	58,723
		Random	0,97	57,11	
		Random	0,99	57,33	
3	Anodizing with electrical voltage 28 V	Random	0,98	57,91	57,718
		Random	0,98	57,91	
		Random	0,99	57,33	

hardness of raw material and anodized aluminum with a voltage variation of 20, 24, and 28-V within 10 min has an average hardness of 59.117 VHN, 58.723 VHN, and 57.718 VHN, respectively, is shown in Fig. 9. The highest hardness value of 59.117 VHN at a variable voltage of 20 V indicates that the lowest voltage will increase the

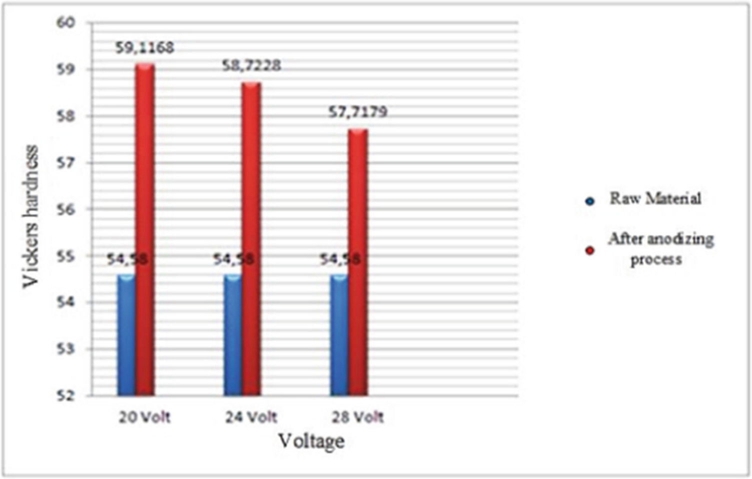

Fig. 9. Comparison of hardness values between raw material and anodizing with variable voltage.

surface hardness value of anodized aluminum. However, the use of a current density that is too high will affect the surface hardness of the anodized aluminum. Variations in sulfuric acid concentration and electrolyte temperature will also affect the surface hardness of aluminum metal in the anodizing process. The harder the temperature of the electrolyte, the harder the aluminum surface will become. And the increase in sulfuric acid concentration will also reduce the surface hardness of anodized aluminum. The ideal sulfuric acid concentration for anodizing is between 15% and 20%.

5 Conclusion

The variation of the electric voltage applied to the anodization process was also proven to have a large effect on the 20, 24, and 28-V voltage variables in 10 min. The hardness value on the surface of series 6 aluminum before the anodizing process is 54.58 VHN on the raw material. The hardness value of the material tends to decrease with the increase in the variable voltage. The same thing is shown in the graph of the effect of variations in electric voltage on the pore diameter. The graph shows a continuous decrease as the voltage increases. The highest hardness value was obtained with a 20-V voltage variation and was obtained at 59.117 VHN; a 24-V voltage variation was obtained at 58.723 VHN; and the lowest hardness value with a 28-V voltage variation was obtained at 57.718 VHN.

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