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ANODIZING CHARACTERISTICS OF AA1100 IN 20% SULFURIC–PHOSPHORIC ACID ELECTROLYTES UNDER AERATED AND NON-AERATED CONDITIONS AT VARIOUS TIMES

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ABSTRACT

Aluminum alloy AA1100 exhibits good corrosion resistance but suffers from low surface hardness and wear resistance, which can be improved through anodizing. However, limited studies have examined the combined influence of sulfuric–phosphoric acid electrolyte composition and aeration. This study investigates the effect of sulfuric–phosphoric acid electrolyte composition and aeration on oxide layer mass, thickness, and hardness during anodizing. Anodization was conducted using a total electrolyte concentration of 20% with varying sulfuric acid (15–20%) and phosphoric acid (5–0%) ratios at a current density of 3 A/dm², operating times of 15 and 30 minutes, and room temperature under aerated and non-aerated conditions. The results show that aeration significantly increased oxide layer formation, yielding an average oxide mass of 0.0173 g compared to 0.0106 g in non-aerated systems. The maximum oxide mass (0.0375 g) was obtained at 20% sulfuric acid with aeration for 30 minutes. The highest oxide layer thickness of 47.94 μm was achieved using a 19% sulfuric acid–1% phosphoric acid electrolyte under aerated conditions at 30 minutes. Meanwhile, the maximum surface hardness of 352.60 HVN was obtained at 15% sulfuric acid–5% phosphoric acid with aeration for 15 minutes. These findings demonstrate that electrolyte composition and aeration play a critical role in optimizing anodic oxide growth and mechanical properties of AA1100.

Keywords: *aluminum, anodizing, oxide layer, electrolyte solution, sulfuric acid, phosphoric acid.*

1. INTRODUCTION

Aluminum is a type of metal that has light properties, is easy to process into various shapes as desired, and has good corrosion resistance, making this type of metal easily found in everyday life, especially in the automotive industry and household industry [1] [2]. From the results of previous research [1] showed that this metal also has weaknesses, namely it is easily deformed, the color tends to be dull, easily oxidized, and has low hardness and wear resistance values. To overcome these properties, additional treatment can be carried out, namely anodization. The anodization process is an electrochemical surface coating process where the workpiece is used as an anode that will be coated with oxide. Although previous studies have mainly focused on the effect of sulfuric acid concentration during anodizing, the combined influence of mixed electrolyte systems (strong and weak acids) and aeration conditions on the formation and mechanical properties of anodic oxide layers on AA1100 aluminum has not been extensively investigated. In particular, the role of aeration in modifying electrolyte dynamics, oxygen availability, and oxide layer growth during anodizing using a sulfuric–phosphoric acid electrolyte system remains limited in the literature.

In the research conducted by [3], it shows the results of the thickness of the oxide layer at variations in sulfuric acid solution concentrations of 16%, 17%, 18%, 19%, and 20% respectively of 16.5 μm , 13.5 μm , 11 μm , 12.1 μm , 9.9 μm . The composition of the 16% sulfuric acid solution with a dipping time of 15 minutes shows the maximum growth of the oxide layer with the highest conductivity of the electrolyte solution. In the variation of the 17% sulfuric acid solution concentration, the maximum electrolyte conductivity is produced with faster growth of the oxide layer on the aluminum surface and a layer solvent level that is not too high so that it forms a thicker oxide layer. However, in the range of sulfuric acid solution concentration variation of 18%-20%, it shows a very high conductivity of the electrolyte solution, with a dipping time of 15 minutes increasing the speed of the parent metal feed and redissolving the outermost oxide layer, so that the oxide layer formed tends to be thin.

This study was conducted with aeration and non-aeration system treatments. In the aeration system treatment, oxygen gas was added to the electrolyte by providing fine air bubbles and letting them rise in the electrolyte solution. It is expected that after the treatment, the system will increase the hardness and thickness of the aluminum oxide. In the non-aeration system treatment, it was carried out without adding oxygen gas to the electrolyte, then a comparison of the thickness and hardness of the resulting aluminum oxide was carried out.

Based on several studies that have been conducted previously, it is necessary to conduct the latest research with the addition of strong and weak acid solutions and the addition of aeration and non-aeration system treatments to determine the characteristics of aluminum metal 1100 anodized with 20% sulfuric and phosphoric acids and aeration and non-aeration systems at different time intervals. It is expected that this research will produce new information regarding the effect of anodizing treatment on aluminum metal on the mechanical properties of aluminum metal.

Aluminum 1100 is an alloy metal that has light properties, high corrosion resistance, good conductivity, high thermal conductivity, and wear resistance. Aluminum alloy 1100 has a minimum Al compound content of 99.9% of the total compounds contained in Al-1100 and the general hardness of aluminum alloy 1100 is 35-55 HVN [4].

Anodization is an electrolysis process carried out to produce an oxide layer (Al_2O_3) with a thickness higher than the naturally formed oxide layer. Thickening of the oxide layer (Al_2O_3) can protect the metal by increasing the properties of the metal, thereby increasing resistance to wear and corrosion. The working principle of anodization is that the anode and cathode are dipped in a certain pH and given a current flow, the anode is connected to the positive pole and the cathode to the negative pole. The anode will be oxidized into ion form so that the metal can bond with oxygen to form an oxide layer (Al_2O_3) [4].

In the anodization process, a thin oxide layer is formed that is well connected to the base metal. The following is the reaction equation for the formation of an aluminum oxide layer:



The oxide layer that is formed has a positive effect on resistance to friction and absorption of dyes, creating the unique color of aluminum. This oxide layer is useful for protecting the surface of aluminum metal from damage caused by its reaction with compounds in the surrounding environment. During the anodizing process, two types of oxide layers are formed, namely the base layer and the pore layer. The oxide layer has a hexagonal pore structure located in the middle of the oxide layer [5].

2. RESEARCH METHOD

2.1. Equipment and Materials

The aluminum specimens used in this study were AA1100 plates with dimensions of 30 mm \times 20 mm \times 2 mm. Each experimental condition was carried out in triplicate (three replicates) to ensure the reliability and reproducibility of the results.

Table 1. Equipment and Materials

No.	Equipment	Materials
1	Rectifier	Aluminum 1100
2	Aerator	H ₂ SO ₄ 98%
3	Flowmeter	H ₃ PO ₄ 85%
4	Jumper cable	NaOH 5%
5	Beaker glass 250 mL	HNO ₃ 8%
6	Analytical balance	Aquades
7	Thermometer	Al Cathode
8	Stopwatch	Abrasive paper

2.2. Research Procedure

The research stages begin with the preparation of tools and materials needed for the anodizing process of Aluminum 1100. The workpieces to be anodized undergoes a series of initial treatments, namely surface sanding, cleaning with 5% NaOH solution, and etching with 8% HNO₃ solution. After that, the initial mass measurement is carried out. The workpiece is then anodized using a mixture of sulfuric acid (H₂SO₄) and phosphoric acid (H₃PO₄) electrolyte solutions with a concentration of 20%. This anodization process is carried out with two different systems, namely aeration and non-aeration and different time variations, namely 15 minutes and 30 minutes. During the anodizing process, the electrolyte temperature was monitored using a thermometer and maintained at room temperature (27 ± 2 °C) to minimize temperature fluctuations that could affect oxide layer formation. After the anodization process is complete, testing is carried out on the anodized metal which includes mass measurement, Vickers Hardness test, and microphotography test. After obtaining complete data, an analysis is then carried out and conclusions are drawn regarding the effect of electrolyte solution concentration, operating time, and aeration system on the anodization process.

2.3. Pre-Treatment

The 1100 Aluminum workpiece was sanded using abrasive paper to remove dirt and smooth the metal surface. After sanding, the cleaning process was carried out using 5% NaOH in a beaker. The aluminum metal was dipped for 5 minutes and rinsed using distilled water. The process aims to remove the remaining sanding process and other dirt from the surface. To neutralize the remaining base on the surface, an etching process was carried out by dipping the aluminum into an 8% nitric acid solution for 5 minutes and rinsing with distilled water

2.4. Initial Weight Measurement

After going through the sanding, cleaning, and etching processes, the initial weight measurement of the aluminum was carried out using a scale. This process is carried out to determine the initial weight before the aluminum is coated.

2.5. Anodizing Process

In this process, anodization is divided into two treatments, namely 20% electrolyte concentration with aeration and without aeration. The aluminum to be anodized acts as an anode and is then connected to the positive pole of the rectifier and the aluminum as the cathode will be connected to the negative pole of the rectifier. Both electrodes are dipped into a mixture of sulfuric acid-phosphoric acid electrolyte with a concentration of 20% and the current strength is set at 3A/dm² with a time interval of 15 minutes and 30 minutes. The aeration procedure is added with oxygen using an aerator and a flowmeter to regulate the air flow rate. In this study, an air flow rate of 4 lpm was used.

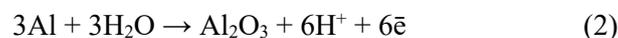
2.6. Result Testing

Testing the results of anodization on aluminum metal is in the form of oxide mass and process efficiency. The calculation of the mass of oxide formed is carried out using the Faraday's law equation by calculating the difference in weight of the workpiece after dissolution using a mixture of sulfuric acid and phosphoric acid and before the anodization process takes place so that the process efficiency value is obtained.

3. RESULT AND DISCUSSION

3.1. Anodizing Process

The anodizing process was carried out on the 1100 type Aluminum workpiece with variations in the electrolyte solution used, namely a mixture of 20% sulfuric acid and phosphoric acid with the composition of the acid concentration used, namely sulfuric acid 15%, 16%, 17%, 18%, 19%, 20% and phosphoric acid 5%, 4%, 3%, 2%, 1%, 0% respectively. The operating conditions used during the anodizing process were at room temperature, current density 3A/dm², aluminum cathode, time 15 minutes and 30 minutes and aerated and non-aerated electrolyte solutions. During the anodizing process, an oxide layer is formed when current is passed through the acid mixture solution, ions from the aluminum workpiece move to the cathode and ions from the acid mixture solution will move to the anode. The anodic oxidation process leads to the formation of an aluminum oxide (Al₂O₃) layer that grows both inward and outward from the metal surface. The presence of sulfuric acid in the electrolyte promotes oxide layer formation, while phosphoric acid contributes to pore development due to its ability to dissolve the oxide layer partially. Therefore, the balance between oxide formation and oxide dissolution strongly influences the thickness and structure of the anodic layer. This chemical exchange occurs on the surface of both electrodes. At the aluminum cathode, reduction occurs and at the aluminum anode 1100 oxidation occurs. The entire process is shown by the reaction equation:



In addition, the aeration system introduces oxygen bubbles into the electrolyte, which enhances electrolyte mixing and improves mass transfer near the electrode surface. This condition can accelerate ion transport and stabilize the oxide formation process, resulting in improved oxide layer growth compared with the non-aerated system.

3.2. Characteristics of Aluminum Mass Changes from Anodizing

Calculation of the difference in aluminum weight by subtracting the weight of the initial aluminum (after pre-treatment) from the weight of the aluminum after anodizing. A positive weight difference (+) indicates that there is an increase in weight as an indication of the formation of an oxide layer on the aluminum plate 1100.

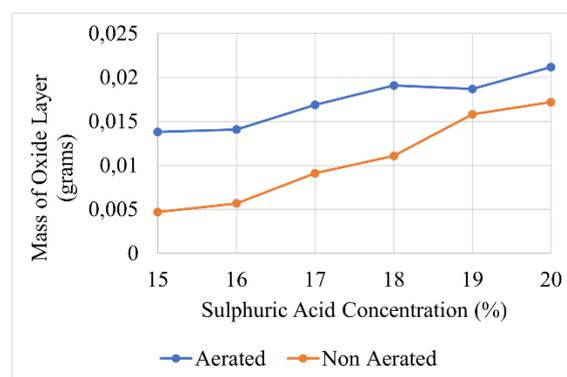


Figure 1. Curve of the effect of electrolyte solution concentration on the mass of the oxide layer with an operating time of 15 minutes

Based on the research data, in the aeration system, a relationship between the composition of the acid mixture and the mass of the oxide layer was obtained which was directly proportional, the higher the concentration of sulfuric acid used, the higher the mass of the oxide layer produced. This trend indicates that increasing the sulfuric acid concentration enhances the oxidation reaction rate, which promotes faster formation of the anodic aluminum oxide layer. Higher sulfuric acid concentration increases electrolyte conductivity, allowing more efficient electron transfer and accelerating oxide layer growth.. However, in an electrolyte solution with a concentration of 19% sulfuric acid and 1% phosphoric acid, there was a decrease in the mass of the oxide layer formed. This phenomenon can be explained by the simultaneous occurrence of oxide formation and oxide dissolution during anodizing. When the electrolyte becomes too acidic, the dissolution rate of the oxide layer increases, resulting in partial removal of the formed oxide and consequently reducing the measured oxide mass.. This can happen because the metal erosion process or the formation of Al^{3+} ions is faster than the formation of the oxide layer. The average mass of the oxide layer formed in the aeration system is 0.0173 grams with the largest oxide layer mass at a sulfuric acid concentration of 20%, namely 0.0212 grams. While in the non-aeration system, a directly proportional relationship is also obtained. The average mass of the oxide layer formed in the non-aeration system is 0.0106 grams. When compared to the average mass of the oxide layer in the aeration system, the average mass of the oxide layer in the non-aeration system has a smaller value. This difference is caused by the use of an aeration system that can increase the oxygen supply to the electrolyte solution so that it forms a porous oxide layer and the metal binds to the oxygen to form a larger oxide layer [6]. The higher oxide mass observed in the aeration system indicates that oxygen bubbling improves electrolyte circulation and enhances the supply of dissolved oxygen near the electrode surface. This improved mass transfer condition facilitates the formation of a more porous and thicker oxide layer, resulting in a higher oxide mass compared with the non-aerated system.

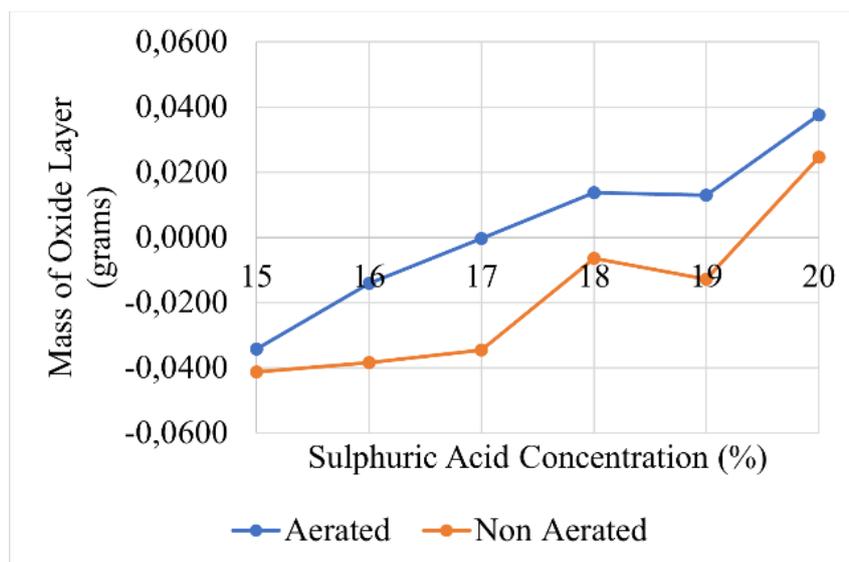


Figure 2. Curve of the effect of electrolyte solution concentration on the mass of the oxide layer with an operating time of 30 minutes

At longer anodizing times (30 minutes), the oxide layer mass generally increases because the anodic oxidation process continues over time, allowing the oxide layer to grow thicker. However, excessive acidity in the electrolyte can accelerate the dissolution of the oxide layer, which may explain the decrease in oxide mass observed at certain electrolyte compositions. The higher the concentration of phosphoric acid solution, the faster the formation of Al^{3+} or Al erosion than the formation of the oxide layer [3], [7]. Phosphoric acid acts as a pore-forming agent that increases oxide solubility, which can reduce the overall mass of the oxide layer if the dissolution rate exceeds the formation rate. This condition occurs because the acidity level of the electrolyte solution and also the optimum point of the electrolyte solution concentration has been reached, this is what makes the data obtained have a minus

value. If seen in Figure 3 above, it shows a decrease in the mass of the oxide layer in the electrolyte solution for a non-aerated system with a sulfuric acid solution concentration of 19% and phosphoric acid 1%, the results of this data are one example of a condition that occurs where the layer mass experiences a decrease in mass because it has reached its optimum point so that Al erosion becomes faster. If observed as a whole, the graph has an upward trend, but both in the aerated and non-aerated systems for electrolyte solutions with a concentration of 19% sulfuric acid and 1% phosphate do not show an increase in the graph but produce a flat graph or even tend to decrease, where this can be caused because the electrolyte solution has reached its optimum value which accelerates erosion of the Al^{3+} layer. This condition indicates that the electrolyte composition approaches an optimum acidity level where oxide dissolution becomes dominant, leading to a reduction in the net oxide mass formed on the aluminum surface [8], [9].

3.3. Characteristics of the Thickness of the Anodizing Aluminum Oxide Layer

The thickness of the oxide layer formed based on Figure 4 tends to be uneven. This non-uniformity may be caused by fluctuations in the current density distribution across the aluminum surface and local variations in electrolyte concentration. Such conditions can lead to localized dissolution of the oxide layer, resulting in variations in thickness [10].

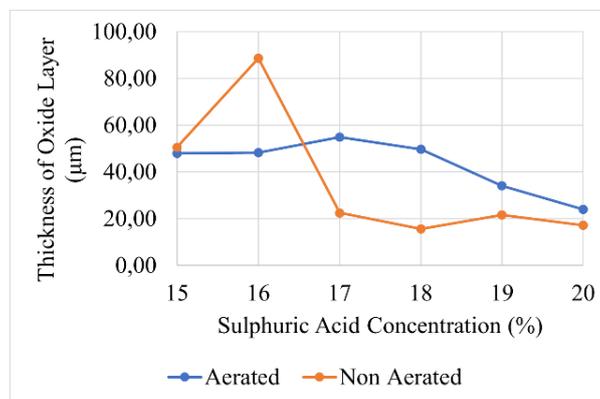


Figure 3. Curve of the effect of electrolyte solution concentration on the thickness of the oxide layer with an operating time of 15 minutes

Meanwhile, at an operating time of 30 minutes with an aeration system as in Figure 4, it has an upward trend, this is in accordance with the results of previous studies, namely that the thickness will increase with increasing anodization time [11]. In the aeration system for 30 minutes with a mixture of 19% sulfuric acid electrolyte solution and 1% phosphoric acid obtained the highest oxide layer thickness of 47.94 µm.

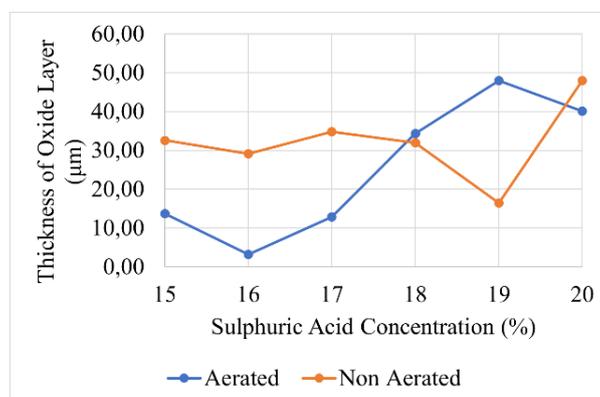


Figure 4. Curve of the effect of electrolyte solution concentration on the thickness of the oxide layer with an operating time of 30 minutes

3.4. Surface Hardness of Anodized Aluminum

After anodizing on aluminum, a Vickers test was carried out to determine the hardness value of the metal after anodization. The Vickers hardness testing mechanism is carried out by indenting a rhombus-shaped diamond into the metal surface [10], [12], [13]. The most optimum hardness value was obtained from the anodization results at an operating time of 15 minutes and an aeration system in a mixed electrolyte solution of 15% sulfuric acid-5% phosphoric acid with a hardness value of 352.60 HVN. The increase in hardness is closely related to the microstructure and porosity of the anodic oxide layer. At moderate sulfuric acid concentrations, a relatively dense oxide layer can be formed, resulting in higher hardness values. However, when the sulfuric acid concentration increases excessively, the oxide layer tends to become more porous, which can reduce its mechanical strength and hardness. This happens because the higher the concentration of sulfuric acid tends to increase the porosity level of the anodized metal. The addition of phosphoric acid solution is done to reduce the formation of pores so that metal with a more optimum hardness level can be produced [14], [15], [16].

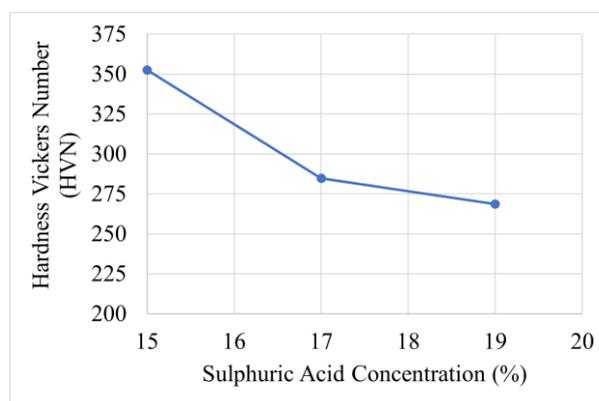


Figure 5. Effect curve of electrolyte solution concentration on hardness value with an operating time of 15 minutes

The anodization results with an operating time of 30 minutes tend to produce an oxide layer with a higher thickness level, but its large porosity level can result in a less than optimum hardness value. This phenomenon occurs because thicker oxide layers often contain higher porosity levels due to prolonged exposure to the acidic electrolyte. High porosity reduces the compactness of the oxide structure, which can lower the hardness value despite the increased thickness of the oxide layer. Therefore, an optimal balance between oxide thickness and porosity is required to achieve maximum surface hardness in anodized aluminum.

4. CONCLUSION

Based on the results of anodization research on 1100 aluminum plates, it was found that the anodizing process produces an oxide layer (Al_2O_3) on the surface of 1100 type aluminum plates which functions to protect the metal from corrosion. The highest oxide layer mass value was obtained from an anodization study with a mixture composition of 20% sulfuric acid without the addition of phosphoric acid with an aeration system and an anodization time of 30 minutes, with an oxide layer mass of 0.0375 grams. It caused by the higher the concentration of sulfuric acid solution and the lower the concentration of phosphoric acid and the use of an aeration system resulted in the formation of an increasingly optimum oxide layer. From the results of the microphotography test, it was found that the highest oxide layer thickness of 47.94 μm was obtained from anodization with an operating time of 30 minutes and an aeration system using a mixture of 19% sulfuric acid and 1% phosphoric acid electrolyte solution. In addition, the Vickers Hardness test results showed that the highest oxide layer hardness, namely 352.60

HVN, was obtained from the anodization process in a mixture of 15% sulfuric acid-5% phosphoric acid at an operating time of 15 minutes with an aeration system.

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