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THE EFFECT OF VARIATIONS IN SULFURIC ACID - OXALIC ACID ELECTROLYTE CONCENTRATION AND ADDITIONAL AERATION ON 1100 SERIES ALUMINUM ANODIZATION RESULTS

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ABSTRACT

Aluminum is one of the materials used in making various types of goods because it has good properties such as being light, ductile and has good corrosion resistance. One of the disadvantages of aluminum is its low level of hardness. This can be overcome by treating aluminum, one of which is the anodization process. In this research, variations in the type of electrolyte used were sulfuric acid-oxalic acid, the electrolyte concentration was 16% with an interval of 0.5, the temperature used was room temperature with a current density of 3/dm2 and a coating time of 30 minutes. The aim of this research is to determine the acid concentration and the effect of the aeration system on mechanical properties. From the data obtained, it was found that the difference in weight of the best metal with the addition of aeration at a concentration of 13.5+2.5 was 0.0411 gr. This shows that as the concentration of oxalic acid increases it can accelerate the process of aluminum oxide formation, then for electrolyte concentrations of 16+0, 15.5+0.5, 15+1, 14.5+1.5 and 14+2 the difference in mass weight metals increased respectively by 0.0337 gr, 0.0335 gr, 0.0366 gr, 0.0390 and 0.0411 gr. In the anodization process without additional aeration, the best metal weight difference is found at an electrolyte concentration of 13.5+2.5 of 0.0401 gr. This shows that increasing the concentration of oxalic acid can accelerate the process of aluminum oxide formation. Then at electrolyte concentrations of 16+0, 15.5+0.5, 15+1, 14.5+1.5 and 14+2 the difference in metal mass weight gradually increases by 0.0182 gr, 0.0293 gr, 0.0318 gr, 0.0322 and 0.0362 gr.

Keywords: *Aluminum, Anodization, Oxide Coating, Electrolyte Solution, Sulfuric Acid, Oxalic acid*

1. INTRODUCTION

Aluminum is a type of metal that is widely used in industry and households. Aluminum is widely used because it has advantages including being light and easy to process into the desired shape [1]. Apart from these advantages, aluminum also has several disadvantages, including being easily deformed and having a low hardness value.

To improve these properties, aluminum needs to be treated, one of which is the anodization process. The anodization process is a surface treatment process carried out with the aim of improving or increasing the properties of a metal, including resistance to wear, increasing hardness, and the aim of beautifying the appearance itself [2]. The electrolyte solution used in the anodization process is sulfuric acid, because it can produce a thicker oxide layer compared to chromic acid [3]. However, the use of sulfuric acid causes large pores on the coated aluminum surface, which results in the metal's hardness level being reduced than it should be. This problem can be overcome by adding a weak electrolyte solution, such as oxalic acid, nitric acid, or phosphoric acid, which can reduce the formation of pores [4]. Research with variations in sulfuric acid concentration of 16%, 17%, 18%, 19% and 20% resulted in the highest oxide layer thickness of 16.5 μm occurring in anodization with a sulfuric acid solution concentration of 16% [5]. The difference in layer thickness is caused by the different conductivity of the electrolyte solution

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due to the difference in concentration of the sulfuric acid solution. Then, with a concentration of 12% sulfuric acid and 1% oxalic acid as electrolyte, as well as variations in current density of 2.8 A/dm2, 3 A/dm2, and 3.6 A/dm2 and a time of 30, 40, and 50 minutes, the results were obtained. the best hardness from 107.03 HVN to 301.03 HVN at 30 minutes and a current density of 2.8 A/dm2.

Most automotive companies use anodized aluminum because of its ability to create a surface that is hard, smooth, and does not wear out easily. Many vehicle components are made from anodized aluminum because the metal is hard, thick and does not wear out easily. An example of the use of anodized aluminum is on pistons, pistons in vehicle engines work by continuously rubbing together, so that the piston does not wear out easily, anodized aluminum is needed so that the piston continues to perform at its best and does not wear out easily.

Based on previous research, it is necessary to carry out new research by adding oxalic acid electrolyte as a weak acid to the weak acid electrolyte, this is expected to influence the oxide layer formed. Then with time variations to find out the optimum time for the anodization process. And adding an aeration system to the electrolyte. This research is expected to produce novelties regarding the effect of anodization on the mechanical properties of aluminum metal.

2. LITERATURE REVIEW

2.1 Aluminium 1100

Most automotive companies use anodized aluminum because of its ability to create a surface that is hard, smooth, and does not wear out easily. Many vehicle components are made from anodized aluminum because the metal is hard, thick and does not wear out easily. An example of the use of anodized aluminum is on pistons, pistons in vehicle engines work by continuously rubbing together, so that the piston does not wear out easily, anodized aluminum is needed so that the piston continues to perform at its best and does not wear out easily.

Aluminum metal is widely used as a material in various industrial tools [6]. Aluminum is one of the basic materials in making various types of goods because it has good properties such as light weight, high strength and ductility, and has good corrosion resistance [7]. Metal materials such as aluminum are one of the materials that is often used for the manufacturing industry and also the automotive industry. In the manufacturing industry, aluminum is often used because it has several advantages, such as being easy to shape into various shapes so that it can be made into various kinds of products, making it a choice in the manufacturing industry. Aluminum metal is also used in vehicles such as pistons. This component is a moving component and naturally must meet certain physical and mechanical properties such as wear resistance, heat resistance, hardness, etc. [2]. An example of an image of 1100 aluminum metal is in figure 1.

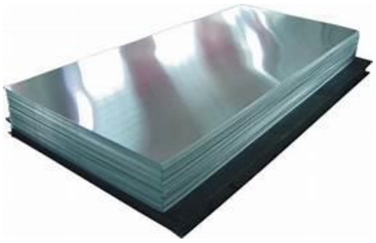

Figure 1. 1100 Series Aluminum Strip

Based on ASTM B-209-96, the Al compound content in 1100 aluminum alloy should be at least 99% of the total of all compounds contained in 1100 aluminum alloy [8].

2.2 Working Principle of Anodization

The anodizing process is a process to produce a thin layer of oxide from a metal and its alloys by means of electrolysis in a suitable solution [9]. Anodization is an electrolysis process with the basic principle of forming a controlled aluminum oxide layer through an aeration process to form a porous oxide layer [10]. The anodization process is expected to improve the mechanical properties of aluminum such as hardness and thickness.

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The following is an example of a reaction in the anodization process:

Reaction at the anode :

$2\ Al \rightarrow 2\ Al^{3+} + 6e^-$ (1)

Reactions on oxides/electrolytes:

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

Reactions on metals/oxides :

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

Reaction at the cathode:

$6\ H^+ + 6\ e^- \rightarrow 3\ H_2$ (4)

The total reaction of the anodization process is:

$Al + 3\ H_2O \rightarrow Al_2O_3 + 3\ H_2$ (5)

Aluminum in anodization is placed as an anode and then the metal is oxidized. An inert electrode is used as the cathode. The cathode and anode are immersed in an acid solution, so that a current occurs in the test cell, then the cathode and anode are connected to a direct current (DC), namely a rectifier, with aluminum connected to the positive pole and the cathode connected to the negative pole. When the rectifier is activated, current flows from the positive pole. This causes aluminum to release electrons, which can cause aluminum to oxidize and bind with oxygen to form an oxide layer [11].

The following is a schematic of the anodization process sequence, shown in Figure 2

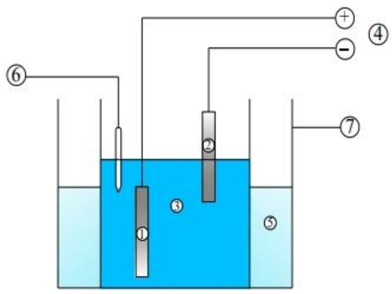

Figure 2 Anodization process sequence**[12]**

Information :

(1) Anode (coated material)

(2) Cathode (conductor)

(3) Electrolyte (H2SO4 solution)

(4) Direct current source.

(5) Ice as a coolant

(6) Thermometer

(7) Box cooler

2.3Oxide Layer

Anodizing produces an oxide layer on the aluminum surface. This oxide layer functions as a corrosion resistance that is useful for industry. The oxide layer formed from this process will increase abrasion resistance, electrical insulator capabilities. The oxide layer functions to protect aluminum against damage caused by its reaction with others, such as from the surrounding environment. In general, the

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anodized oxide layer has hard characteristics and has a hardness comparable to sapphire, is insulative and resistant to loads, is transparent, has no flakes [13].

The formation of an oxide layer can be seen and studied by observing changes in current at a fixed rectifier voltage or changes in voltage at a fixed current [14]. The process of forming the aluminum oxide layer has 4 stages as shown in Figure 3.

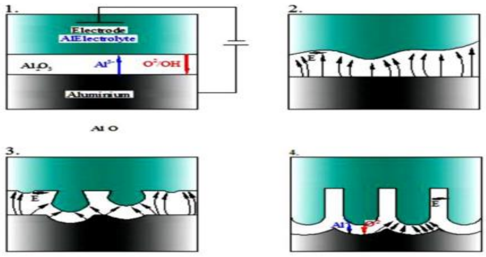

Figure 3 Stages of oxide layer formation **[14]**

1. Formation of barrier layer

2. Beginning of pore formation

3. Pores begin to form and develop

4. The pores formed are more stable

Anodization is influenced by several factors including current density, immersion time and voltage which causes the physical and mechanical properties of aluminum to vary [15].

2.4 Influencing Factors

2.4.1 Voltage

Voltage has a significant influence on the surface roughness of aluminum anodization results using titanium as a cathode [16]. Because when the voltage is increased, the electric field formed between the electrodes will increase. This increase in the electric field will attract more ions from the electrolyte solution towards the surface of the workpiece (aluminium), so that the process of forming an oxide layer during the anodization process on the surface will increase and produce a thicker layer.

2.4.2 Temperature

The factor that will greatly influence is temperature, at a higher temperature the anodized layer will become thinner and softer, the pore diameter will be larger, and the pore absorption capacity will be higher [17].

2.4.3 Electrolyte composition and concentration

In the anodization process, the electrolyte used is a strong acid such as sulfuric acid, oxide acid, phosphoric acid and other types of acid. The concentration of the electrolyte solution greatly influences the anodization process. The maximum oxide layer formed will have a good thickness and if it exceeds the maximum limit the oxide layer formed will decrease [5].

2.4.4 Time

Time can influence the anodization process. The longer the anodization time, the thicker the oxide layer produced so that the hardness of the aluminum resulting from anodizing increases, however, the oxide layer produced has a maximum thickness so that if anodization is carried out for more than 120 minutes, the hardness tends to be constant or decrease [18].

3. RESEARCH METHODOLOGY

3.1 Tools and materials

The tools and materials used in this research include tools: Al cathode, beaker, rectifier, analytical balance, aerator, thermometer, measuring cup, abrasive paper. Ingredients: aluminum 1100, sulfuric acid, oxalic acid, NaOH, HNO3, Aquadest, and ice cubes.

3.2 Research Stages

The stages of this research begin with the preparation of tools and materials, then continue with the pre-treatment stage. In the pre-treatment stage there are 3 processes, namely, the metal sanding process, then after the metal has been sanded, it continues with the degreasing process using a 5% NaOH solution. After the degreasing process, the neutralizing process continues using the solution. HNO3. After the pre-treatment process is complete, the workpiece will be anodized with the following operating conditions: room temperature 25°C - 30°C, immersion time 30 minutes, and current density of 3 A/dm^2

3.3 Pre-Treatment Work Unit

This process refers to ASTM D-2651-01 [19] using an acid-base electrolyte solution which aims to clean or remove contaminants in aluminum metal such as oil, dust and other impurities so that they do not hinder the anodization process or inhibit the formation of an oxide layer on the metal. The material used is aluminum 1100, a sanding process is carried out first to remove the natural oxide layer on the metal and even out the surface. This sanding process uses abrasive paper and aluminum by rubbing the abrasive paper on the aluminum and running water until the aluminum surface is flat and clean. This is to prevent metal oxidized by the surrounding air. The workpiece is then subjected to a degreasing process aimed at removing oil, fat and other organic substances from the metal surface using a 5% sodium hydroxide (NaOH) solution for 5 minutes, then rinsed with clean water and dipped in 8% HNO3 nitric acid solution for 1 minutes to neutralize the remaining acid-base solution in the degreasing process and aims to make the aluminum surface cleaner and the anodization results brighter.

3.4 Anodization Process

Aluminum in the anodization process is placed as an anode and then the metal undergoes oxidation. An inert electrode is used as the cathode. The anode and cathode are immersed in an acid solution so that a current is formed in the test cell. Then the anode and cathode are connected with direct current (DC) through a rectifier, with aluminum connected to the positive pole and the cathode to the negative pole. When the rectifier is activated, current flows from the positive pole, causing the aluminum to release electrons which bond with oxygen to form an oxide layer. This process uses a 20% sulfuric acid electrolyte solution with the addition of varying concentrations of oxalic acid at 1% intervals and additional aeration. The operating conditions used in this anodization process are 25-30ºC, current density 3A/dm2, and anodization process time of 30 minutes. After that, proceed with calculating the difference in metal weight compared to aluminum 1100 before and after anodizing.

3.5 Weight Difference Measurement

After the anodization process is complete, the weight of the sample in the anodization process is measured using an analytical balance. Then obtain the calculated weight difference value and compare the results of the initial weight and final weight in the anodization process.

4. RESULT AND DISCUSSION

In this research, calculating the mass of the metal to determine the mass of the oxide after the anodization process, the calculation of the mass of the oxide can be calculated using the formula final weight - initial weight. Calculation of the oxide mass aims to determine the speed of the aluminum oxide formation process. The results of calculating the difference in metal weight are shown in Figure 4.

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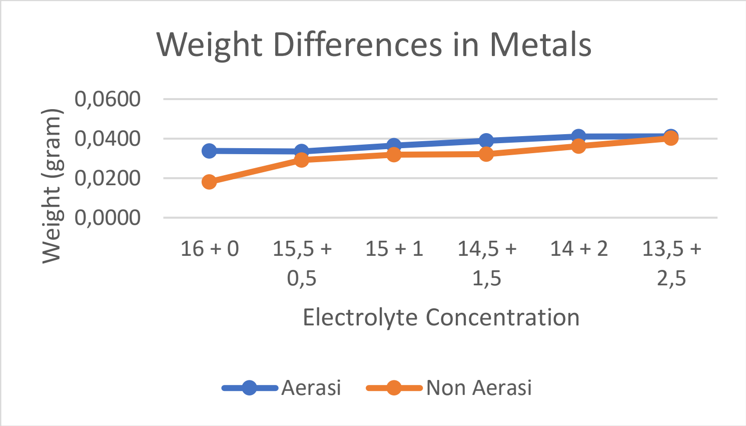

Figure 4. Relationship between changes in acid concentration and weight differences

Based on Figure 4, the comparison graph between the difference in weight and electrolyte concentration. Under aeration conditions, it shows a stable increase in weight difference around 0.033 to 0.041 gr. Then the non-aeration condition shows a more gradual, consistent increase in the weight difference compared to aeration. Increasing the concentration of oxalic acid affects metal loss in the anodization process, so that the eroded base metal is replaced by an oxide layer [20].

Based on observation data on the difference in weight from the anodization process. Seen from Figure 4. The difference in weight of the best metal with the addition of aeration at a concentration of 13.5+2.5 is 0.0411 gr. This shows that as the concentration of oxalic acid increases it can accelerate the process of aluminum oxide formation, then for electrolyte concentrations of 16+0, 15.5+0.5, 15+1, 14.5+1.5 and 14+2 the difference in mass weight metals increased respectively by 0.0337 gr, 0.0335 gr, 0.0366 gr, 0.0390 and 0.0411 gr. In the anodization process without additional aeration, the best metal weight difference is found at an electrolyte concentration of 13.5+2.5 of 0.0401 gr. This shows that increasing the concentration of oxalic acid can accelerate the process of aluminum oxide formation. Then at electrolyte concentrations of 16+0, 15.5+0.5, 15+1, 14.5+1.5 and 14+2 the difference in metal mass weight gradually increases by 0.0182 gr, 0.0293 gr, 0.0318 gr, 0.0322 and 0.0362 gr.

From these results it can be concluded that adding aeration produces a more consistent and higher weight difference compared to without adding aeration. This shows that additional aeration can help in the formation of a larger aluminum oxide layer. Meanwhile, conditions without additional aeration show that as the concentration of oxalic acid increases, the formation of the oxide layer becomes faster.

This anodization process with aeration produces a more consistent metal oxide mass weight and tends to be slightly higher compared to non-aeration. This shows that aeration can help in the formation of a more uniform oxide layer. Meanwhile, non-aeration shows a more gradual increase in the weight of the oxide mass, the process of formation of the oxide layer is slower but more stable, with the addition of oxalic acid concentration, the higher the concentration of oxalic acid added, the heavier the oxide mass formed.

From these results it can be concluded that adding aeration produces a more consistent and higher weight difference compared to without adding aeration. Meanwhile, conditions without additional aeration show a more fluctuating weight difference, because it is caused by a lack of oxygen transfer during the anodization process. This shows that the greater the additional concentration of oxalic acid has an optimum amount, if it exceeds the maximum limit, it results in a decrease in the formation of the aluminum oxide layer.

5. CONCLUSSION

Based on analysis, the conclussion of this research are follow:

1. The best concentration is found at an electrolyte concentration of 13.5+2.5, both with aeration and non-aeration with results of 0.0411 gr and 0.0401 gr.

2. The aeration system can form an oxide layer more quickly than without aeration as seen from the weight difference, namely at an electrolyte concentration of 13.5+2.5 it is 0.411 gr while without aeration the result is 0.0401 gr.

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