The Influence of Electric Current and Electrolyte Solution on Porous Characteristics on Aluminum Anodizing

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Abstract. For decoration purposes, aluminum metal has widely used metal coloring techniques to enhance the aesthetic value of the metal. Most coloring techniques of the aluminum metal use aluminum metal dipping with dye or with chrome (Cr) directly. This coating technique has the disadvantage, such as the color fade easily or the chrome layer peels off. The anodizing method becomes a potential method to improve coloring techniques because this process is able to provide a protective layer and improve surface appearance through staining and can change the aluminum surface to aluminum oxide, high hardness, and corrosion resistance. This study aims to analyze the surface condition of the aluminum after the anodizing process with several variables. In this research uses the variation of H\textsubscript{2}SO\textsubscript{4} electrolyte solution concentration and electric current as variables to improve aluminum surface quality. Anodizing process is carried out with electrolyte concentration up to 20\% and current up to 5 A. The Characteristic of an aluminum oxide layer is tested using hardness test, optical and Scanning Electron Microscope to observe the hardness, the pores distribution and the average of pores diameter. The results of the hardness show that increasing electric current will decrease the hardness of the Aluminum surface. The significant results are also showed in electrolyte concentration variables, increasing electrolyte concentration will reduce hardness and enlarge pores diameter.

1. Introduction

Aluminum metal is one of the metal materials that commonly used in daily life and Industry, such as kitchen equipment, the automotive industry, aircraft manufacturing materials and so on. Aluminum has superior properties such as high strength, lightweight, and good formability [1]. Recently, the world market needs aluminum metal is not only limited to the physical properties of Aluminum but also related to aesthetic aspects. Most aluminum metal coloring techniques use dye or with chrome (Cr) for the coating techniques of aluminum metal using [2]. This technique has a major disadvantage, for the example; it is easy to fade or peel up. Anodizing technique is believing as the most suitable technique to produce an oxide layer in aluminum surface. The oxide layer has the porous structure that can support the coloring process and change the aluminum surface to be more decorative and corrosion resistant.

Through the principle of electrochemistry and utilizing aluminum properties which have a high chemical affinity for oxygen, the anodic porous of the aluminum oxide is formed by involving anodic oxidation to obtain porous alumina layers, both in micro and nano scales [3]. The electrolyte used is sulfuric acid to
function as an oxidizer. Besides being inexpensive and easy to obtain, oxide layer results using sulfuric acid electrolytes have extensive aesthetic and functional properties [4]. From previous research, anodizing process can increase the hardness of the aluminum surface [5]. The quality of the anodized product is determined by the thickness of the pores formed and the distance between pores. The distance between pores formed must be close together to avoid uneven coloring.

In this study, anodic porous alumina will be made through an anodizing process by regulating processes and variables such as current strength and electrolyte concentration, so that the quality of the coloring is good and the pore size is evenly distributed.

2. Experimental Method

This research use aluminum 1100 series and then this metal will be put as an anode. At the cathode, use a Lead metal plate that placed around the Aluminum as shown in Fig.1. The electrolysis process occurs in the electrolyte solution with variations of 10%, 15% and 20% of sulfuric acid to distilled water. The condition of the anodizing process will be encoded as shown in table.1.

Before electrolysis process, it is necessary to do the surface preparation. In this research, there are four steps to prepare the surface of the anodizing specimen. For the first step the specimen surface is cleaned using sandpaper then second dipped into sodium carbonate solution for 2 minutes to increase the clean power in the washing process, and continue to rinse in distilled water. After that, the surface will be etched to remove the oxide layer on the aluminum surface which cannot be removed by the previous process. The etching process uses soda caustic that dissolve into distilled water with a concentration 100 g/liter then the specimen is dipped again into the water for 1 minute to clean soda caustic solution. The last step is desmutting process. This process aims to remove smuts on the aluminum surface. Smut is a thin gray to black layer derived from aluminum metal-forming alloy materials which cannot dissolve in etching solutions. In this process, the specimen is immersed into desmut solution that contain 75% phosphate, 15% sulfuric acid (H₂SO₄) and 10% nitric acid (HNO₃) for 1 minute.

Anodizing process conduct in current from 3 to 5 A of the direct current power supply with voltage 25 Volts, the electrical circuit can be seen in Fig.1. The electric current in the power supply is set after the specimen has been dipped in the electrolyte solution with a current of 1 Ampere. The time for immersion is 60 minutes. After the anodizing process complete, then rinse using distilled water, before proceeding to the colorizing process.

In the coloring process then the material is dipped into a dye solution for 3 minutes. The solution made from dye powder with dissolved in distilled water as much as 10 g/l. This aims to add decorative value to the aluminum metal and in addition, it will be a protective layer on the oxide layer. The last step of coloring
anodizing is a sealing process. In this process, the pores of the oxide layers will be closed and color locking occurs. Sealing use acetate acid with concentration 20 ml/l and immerse for 10 seconds.

Table 1. Samples Encoding

| Sample Code | Electric Current (A) | [H$_2$SO$_4$] % | Holding Time (minute) | Voltage (V) |
|-------------|----------------------|-----------------|-----------------------|-------------|
| A           | 3 A                  | 10              |                       |             |
| B           | 4 A                  | 10              |                       |             |
| C           | 5 A                  | 10              |                       |             |
| D           | 3 A                  | 15              |                       |             |
| E           | 4 A                  | 15              | 60                    | 25 V        |
| F           | 5 A                  | 15              |                       |             |
| G           | 3 A                  | 20              |                       |             |
| H           | 4 A                  | 20              |                       |             |
| I           | 5 A                  | 20              |                       |             |

Optical Microscope observes the characteristics of pores including diameter average and distribution of pore. The thickness of the oxide layer can be seen under scanning microscope optic, and microhardness Vickers method is used to measure the hardness of oxide layers. The load is 10 grams and pressing for 6 seconds.

3. Result and discussion

3.1. Hardness Testing Results
The result of the hardness test shows that increasing the current, the hardness value tends to decrease. The higher electrical current causes the dimension of the pores to become larger. Colorizing process (warna) raise the hardness value, can be seen in Fig. 3. This happens due to after colorizing process, the pores are covered by coloring and sealing agent that believed will influence to the surface hardness.

![Hardness Testing Results](image)

Figure 2. The hardness of specimens before and after colorizing process.

Base on microhardness measurement, the concentration of the sulfate acid solution influences hardness value as well. Increasing solution concentration apparently reduce hardness value. The highest hardness
value was obtained in the sample with a condition, the electrical current variable of 3 Ampere and 10% electrolyte solution concentration. The hardness achieves to 59.1 VHN.

3.2. The Characteristics of Pores

Figure 3 is a representation of the pores appearance of the anodizing aluminum surface. The image on the left is an A sample with 10% of H$_2$SO$_4$ concentration that has the smallest average pore diameter of 17.53 µm, while on the right is a sample of H which has the highest average pore diameter of 29.82 µm. under optical microscope view, the difference is that sample A has more pores than sample H.

![Figure 3: Photos of a surface with Optical Microscope, Sample A x50 magnification (a) and Sample H x50 magnification (b)](image)

From Fig.4, The sample A, sample B, and sample C have the different electric current condition. They show the different average pore diameter respectively 17.53 µm, 20.88 µm, and 21.29 µm. It can be concluded that the higher electrical current will increase the pore diameter. This is caused by the high current reaction rate will also be faster and have an impact on the pore shape that is wide and not tight to each other. The results of this study which the results close to SNI No. 07-0734-1989 are sample A with an average pore diameter of 17.53 µm.

![Figure 4: Sample A (a), sample B (b), sample C (c)](image)

3.3. Oxide Layer Thickness

The Observations using the SEM can be seen the differences in the thickness between the oxide layer (a) and the base metal (b) (Fig 5). This data is taken after the aluminum through the colorizing process. The thickness is measured by drawing a line horizontally on the oxide layer which looks darker than the metal.
Specimen A that anodizing on the lower electric current and solution concentration than Specimen H, obtains average oxide layer thicker, is 103 µm while Specimen H has thickness 15.6 µm. It can be seen that the layer formed is not as thick as the layer in the A sample.

Figure 5. The thickness of the oxide layer a and base metal b of (a) specimen A, and (b) Specimen H

4. Conclusions
Anodizing process can produce porous on the aluminum surface. Something that we consider is higher electric current and solution concentration can cause the size of pores wider and the hardness value decrease although colorizing process can raise hardness value. The thickness of the oxide layer formed decrease along with increasing electric current. From available data, the higher the concentration of the electrolyte solution, the lower the yield of the oxide layer formed on the surface anodized aluminum metal.

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