P-ISSN : 2598-1277
E-ISSN : 2621-3966

JOURNAL OF GREEN SCIENCE AND TECHNOLOGY

ENHANCING HARD ANODIZATION OF ALUMINUM 1100 USING VARIATED CONCENTRATION AND AERATION IN 20% SULFURIC AND PHOSPHORIC ACID ELECTROLYTES

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ABSTRAK

Aluminum is widely used in manufacturing, especially in automotive and aerospace, due to its light weight and easy form, although it is worn. The purpose of this research is to get the highest layer formation efficiency. During the application, hard anodizing creates a new layer in the form of a anatural protective layer on the metal surface. In hard anodized aluminum, the specimen sanded, degreasing with base, and acid neutralization. Then, hard anodizing is performed by connecting the workpiece to the positive pole and the cathode to the negative pole with a current density of 3A/dm² for 30 minutes in an electrolyte solution at a temperature of 5°C. The variations used are the mixture composition between sulfuric acid (concentration 15% - 20%) and low concentration phosphoric acid (0%, 1%, 2%, 3%, 4%, 5%) which are differentiated into aerated and non-aerated systems. Then weigh the anodized work piece to determine the weight gain of the metal due to the formed layer. The weight of the obtained layer is used to determine the efficiency of the formation of oxide layer. The best layer formation efficiency is found in the sulfuric acid - phosphoric acid concentration variation (15% - 1%) in the aerated system at 73.47%. While the non aerated system at 73.28%. The aerated condition shows superior results compared to the non-aerated system, yielding better efficiency values than the non-aerated system.

Keywords: *Aluminum 1100, Hard Anodizing, Mechanical Properties, Aeration, Electrolyte*

1. INTRODUCTION

Aluminum is a type of metal commonly used, particularly in the automotive and aerospace industries. This metal is frequently utilized due to its advantages, such as being lightweight, having good thermal and electrical conductivity, corrosion resistance, and ease of forming, especially as a material for automotive and aircraft components [1]. Therefore, this metal is often used as a material for components that operate in harsh environmental conditions (pistons, aircraft gears, pulleys, etc.).

In addition to its advantages, aluminum also has weaknesses, such as being prone to wear due to its low hardness. Therefore, when used in harsh operating conditions such as friction and high temperatures, this metal is susceptible to wear, which can reduce the lifespan of a component [2]. Therefore, hard anodizing is used to improve the mechanical properties of aluminum by producing an aluminum oxide layer on the metal surface [3]. Hard anodizing is an electrochemical metal coating process that generates an Al_2O_3 layer on the metal surface. This layer enhances the metal's weight, which contributes to increased thickness and hardness of the coating.

According to [4], the type of aluminum that can be hard anodized is 1100. Several factors affect the results of hard anodizing, including the type of acid, concentration, time, temperature, current density, and cell aeration. Previous research has conducted hard anodizing with a sulfuric-phosphoric acid electrolyte at 15%. The best aluminum oxide layer was achieved with a thickness of 23.61 μm with the addition of 2% phosphate [1]. Further research was conducted on hard anodizing with a sulfuric-phosphoric acid electrolyte at 15% with voltage variations of 10 V and 20 V. The best layer was achieved at 20V with a process efficiency of 83.05% and a layer thickness of 11.43 µm [5]. Another study was conducted with the same electrolyte at temperature variations of 5°C, 10°C, and 15°C. The best layer was obtained at an electrolyte temperature of 5°C with the best oxide mass addition of 0.255 g and a process efficiency of 69.03% [6]. Due to several studies that have been conducted, none have investigated the effect of adding aeration to hard anodizing with a sulfuric acid and phosphoric acid electrolyte solution. This research uses a sulfuric-phosphoric acid electrolyte at 20%, varying its concentration and differentiating between aeration and non-aeration systems. The aim of this research is to determine the influence of concentration and cell aeration on oxide mass addition in the hard anodizing process. This research is expected to provide novel insights compared to previous studies.

2. LITERATURE REVIEW

2.1. Aluminum Alloy 1100

Aluminum 1100 is a widely used metal. It is typically alloyed with other elements according to the specific requirements and applications, making the metal stronger [7]. According to [8], the aluminum content in the 1100 alloy should be at least 99% of the total compounds present in the metal. The hardness value of the 1100 aluminum alloy ranges between 35 and 55 HVN [1].

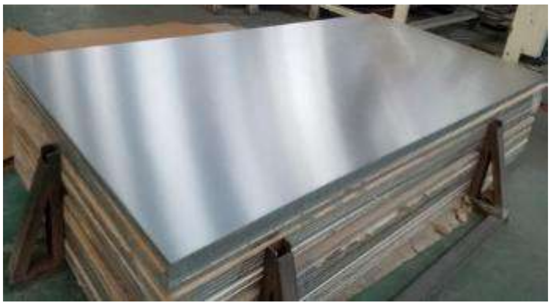

Figure 1. Aluminum Alloy 1100

2.2. Hard Anodizing

Conventional and hard anodizing are electrochemical treatments that began in the 1930s. Both processes involve converting aluminum into its oxide by selecting the appropriate type of electrolytic bath, temperature, voltage, and current density [9]. Hard anodizing is an electrolytic metal coating process that produces a superior aluminum oxide layer compared to conventional anodizing. This process uses higher voltage or current density and lower electrolyte temperatures. Hard anodizing techniques are typically used to enhance the mechanical properties of aluminum [10].

2.3. Aluminum Oxide Layer

The result of the anodizing process is the formation of an aluminum oxide (Al_2O_3) layer that adheres well to the surface of the aluminum metal, serving as a protective coating. The reaction that occurs during the anodizing process is as follows.

$$3\ Al + 3\ H_2O\ à\ Al_2O_3 + 6\ H^+ + 6e^- \qquad (1)$$

In general, there are two types of oxide layers on aluminum: barrier-type oxide films and porous oxide films. The oxide layer formed has a porous structure with a hexagonal pattern, where the pores are located at the center of the structure.

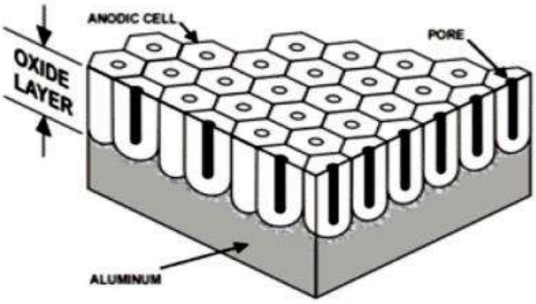

Figure 2. Aluminum Oxide Layer Structure

At the beginning of the anodizing process, a thin base layer with a non-porous dielectric structure known as the barrier layer is formed. This layer grows proportionally to the applied voltage until it reaches a thickness of about 0.02 µm [11]. The barrier layer is a thin, dense coating that acts as an intermediary between the porous layer and the base metal. It protects the metal from further corrosion and is resistant to electrical currents. The porous structure of the oxide layer results from the balance between oxide formation and oxide layer dissolution [12].

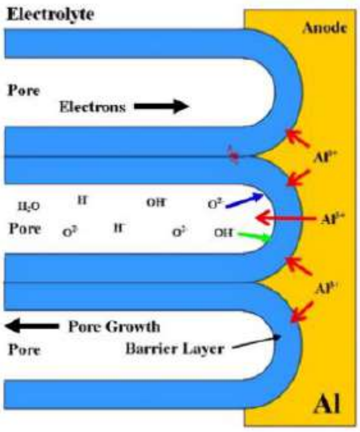

Figure 3. Scheme of the Pore Layer Resulting from Anodizing [13]

2.4. Influencing Factors

2.4.1. Electrolytes

Typically, electrolyte solutions commonly employed for the anodizing process include chromic acid and sulfuric acid. Alternatively, a blend of sulfuric acid and phosphoric acid can also be utilized. This mixture serves to generate an aluminum oxide layer that not only enhances corrosion resistance but also improves the adhesive properties of the metal surface, thereby enhancing its overall durability and performance in various applications. [14] [15].

2.4.2. Concentration

The increase in electrolyte concentration has a significant impact on the characteristics of the oxide layer formed during the anodizing process. This alteration can influence the rate of metal loss that occurs while the metal is being anodized. If metal loss continues unchecked, the base material of the workpiece will eventually be consumed, leaving only the oxide layer behind. In such a scenario, the oxide layer no longer serves as a protective barrier for the underlying metal; instead, it effectively becomes part of what was originally the base material. Consequently, this change undermines the layer's intended function of protecting the metal, altering its role entirely [16].

2.4.3. Temperature

The operational temperature during anodizing, particularly in hard anodizing, plays a crucial role in determining the quality and characteristics of the oxide layer formed on the metal surface. Lower temperatures are preferred as they facilitate quicker and more even growth of the oxide layer, thereby enhancing the effectiveness of the coating process compared to higher temperatures. This controlled temperature environment ensures optimal conditions for the formation of a robust and uniform oxide layer, which is essential for achieving desired properties such as improved durability and corrosion resistance in the treated metal [6].

2.4.4. Time

The duration of the anodizing process is directly related to the thickness of the oxide layer that forms on the metal surface. In general, the longer the metal remains exposed to the anodic electrolyte solution, the thicker the resulting oxide layer becomes. This correlation means that extending the anodizing time allows for a more substantial accumulation of the oxide layer on the surface of the metal [17].

2.4.5. Current Density

The application of current density plays a significant role in determining the characteristics of the resulting oxide layer. When the current density is increased, the electric field around the electrode intensifies, which enhances the electrolysis process. This optimization leads to a greater amount of oxide layers adhering to the surface of the workpiece. Specifically, an increased current density facilitates the formation of a thicker and more robust oxide layer. For instance, the hardness of the aluminum surface is notably higher at a current density of 3 A/dm² compared to other variations of current density [18].

2.4.6. Aeration

The purpose of incorporating aeration is to augment the concentration of dissolved oxygen within the electrolyte solution. This addition facilitates the introduction of extra oxygen into the electrolyte, thereby mitigating any oxygen deficiency that may arise during the anodizing process [19].

3. RESEARCH METHODOLOGY

3.1. Research Stages

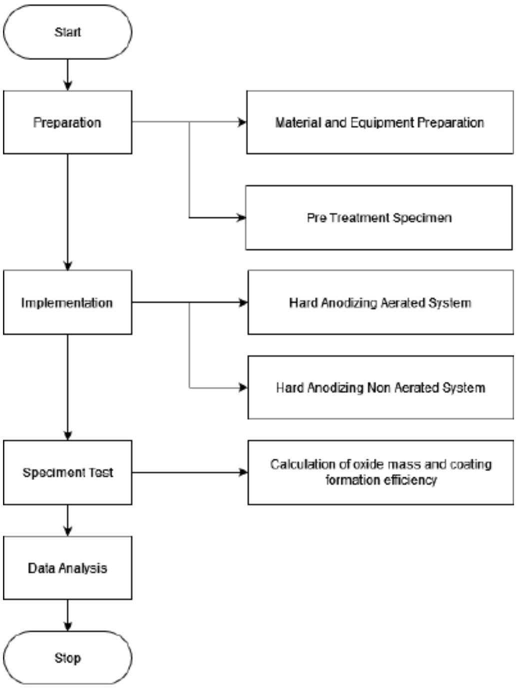

Figure 4. Hard Anodizing Stages

3.1.1. Preparation

3.1.1.1. Material and Equipment Preparation

The equipment and materials required during the hard anodizing process are presented in the following table:

Table 1. Material and Equipment

Equipment	Material
Double Jacketed Beaker Glass	Sulfuric Acid
MGW Lauda Water Bath	Phosphoric Acid
Analytical balance	NaOH
Cathode (Al)	HNO_3
Rectifier	Aquadest
Beaker Glass	Ice
Hot Plate	Resin
Measuring glass	Aluminum
Thermometer	
Abrasive grinder	
Aerator	

3.1.1.2. Material and Equipment Preparation

The workpiece made of aluminum 1100 is initially subjected to sanding to eliminate any dust or rust present on its surface. This sanding process is carried out under a continuous flow of water to prevent the metal from oxidizing due to exposure to the surrounding air. Following this, the workpiece undergoes a reduction process for a duration of 5 minutes using a sodium hydroxide (NaOH) solution, which serves to open the pores on the metal surface. The workpiece is then thoroughly washed with clean water to remove any residual NaOH solution and is subsequently neutralized by immersing it in a nitric acid (HNO_3) solution for 1 minute. The purpose of this reduction process is to ensure the complete removal of any remaining base from the metal surface, thereby making it cleaner and resulting in a brighter finish on the anodized product.

3.1.2. Implementation

The hard anodizing process is categorized into two distinct methods: hard anodizing with aeration and hard anodizing without aeration. The pre-treated aluminum 1100 workpiece will be connected to an electrical current via a rectifier, maintaining a current density of 3 A/dm². Following this, the metal is immersed in an electrolyte solution, which features various concentrations and types of acid, to undergo the hard anodizing process. This procedure is conducted at a temperature of 5°C ± 1°C for a duration of 30 minutes. The specific types of acids utilized, along with their concentration variations, are detailed in the following table:

Table 2. Electrolytes Composition

	Concentration (20%)					
Sulfuric Acid	20	19	18	17	16	15
Phosphoric Acid	0	1	2	3	4	5

3.1.3. Specimen Testing

This quantitative analysis method is used to determine the amount of substance based on weight measurement. It is used to measure the weight and efficiency of the oxide produced from the hard anodizing process. Before the coating process, the initial weight of the workpiece is recorded as the initial weight. After the coating process is complete, the workpiece is weighed again to obtain the final weight. The difference between the final weight and the initial weight indicates the weight of the oxide mass formed during the anodizing process, which will be used to calculate the efficiency of the oxide layer formation process.

3.2. Hard Anodized Data Collection

Once the anodizing process has been fully completed, the sample undergoes a meticulous weight measurement and testing procedure. Initially, the weight of the workpiece is recorded before the hard anodizing treatment begins. After the anodizing process is finalized, the workpiece is weighed again to determine any changes in its mass. This post-anodizing weight is then carefully compared to the initial weight of the workpiece to assess the effect of the hard anodizing process on the sample's overall mass.

4. ANALYSIS AND RESULT

4.1. Oxide Layer Addition Efficiency

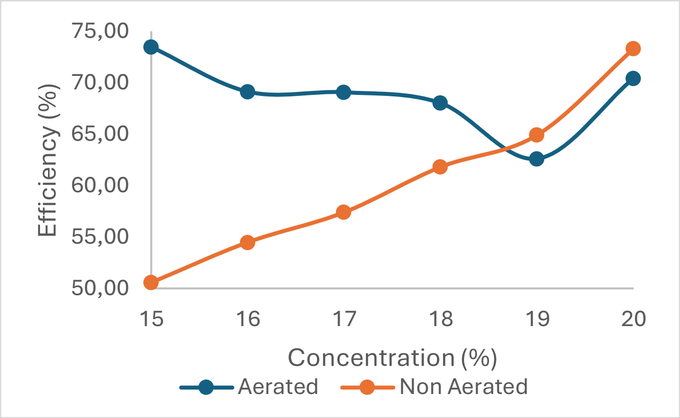

Figure 5. Relation between Efficiency and Concentration

The efficiency of aluminum oxide layer formation in the aeration system is superior compared to the non-aeration system. In the aeration system, metals coated with sulfuric acid and phosphoric acid electrolytes tend to experience an increase in efficiency with the addition of phosphoric acid at 1% intervals. Conversely, in the non-aeration system, metals experience a decrease in efficiency with the addition of phosphoric acid at 1% intervals. This indicates that the optimal point for adding phosphoric acid concentration in the aeration system is at 5% phosphoric acid addition, while in the non-aeration system, the optimal point is at 1% phosphoric acid addition.

The best efficiency value is found in the variation of 15% sulfuric acid concentration plus 5% phosphoric acid in the aeration system, with an efficiency value of 73.47%. Meanwhile, in the non-aeration system, the best average weight gain of the metal is found in the variation of 20% sulfuric acid concentration without the addition of phosphoric acid, with a value of 73.28%. This may be due to the formation of pyrophosphoric acid ($H_4P_2O_7$) in the electrolyte solution, where two phosphate molecules can combine with water. Pyrophosphoric acid in the electrolyte solution can affect the rate of pore layer formation on the metal surface, especially in the formation of aluminum oxide nanofibers. However, if the anodization treatment lasts too long, these aluminum oxide nanofibers can degrade [20].

5. CONCLUSSION

The efficiency of aluminum oxide layer formation is significantly higher in the aeration system when compared to the non-aeration system. Within the aeration system, the incremental addition of phosphoric acid at 1% intervals notably enhances efficiency, reaching an optimum at a 5% phosphoric acid addition. Conversely, in the non-aeration system, efficiency tends to decrease with the addition of phosphoric acid, achieving an optimum point at just 1% phosphoric acid addition. The highest efficiency value in the aeration system is observed with a sulfuric acid concentration of 15% combined with 5% phosphoric acid. On the other hand, the non-aeration system attains its best efficiency value with a 20% sulfuric acid concentration without any added phosphoric acid. This discrepancy is likely attributed to the formation of pyrophosphoric acid in the electrolyte solution, which influences the rate of pore layer formation and the development of aluminum oxide nanofibers. Consequently, further analyses are essential, such as measuring the thickness of the aluminum oxide layer and testing the hardness of the metal, to gain a deeper understanding of this phenomenon.

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