

International Review of Mechanical Engineering (IREME)

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International Review of Mechanical Engineering (IREME)

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The *International Review of Mechanical Engineering (IREME)* is a publication of the **Praise Worthy Prize S.r.l.**. The Review is published bimonthly, appearing on the last day of January, March, May, July, September, November.

Published and Printed in Italy by **Praise Worthy Prize S.r.l.**, Naples, May 31, 2016.

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Properties of Electrochemical Impedance and Surface Characteristics of Anodized AA 6061

Putu Hadi Setyarini, Rudy Soenoko, Agus Suprpto, Yudy Surya Irawan

Abstract – This paper was intended to figure out the corrosion resistance and surface morphology of AA6061 that is anodized with 1M phosphoric acid and cathode titanium in several potential variations. The surface morphology was examined by scanning electron microscopy (SEM), the surface roughness was investigated by Portable Surface Roughness Tester and finally the corrosion resistance was analyzed by the electrochemical impedance spectroscopy (EIS). The surface morphology showed that an increased potential of electricity supplied during the anodizing process leads to enlarged pore size larger or equitable to that occurring on the surface of the specimen, leading to decreased surface roughness values. Surface roughness tests indicated the lowest roughness values achieved from the anodizing with 30V potential was 0.674 μm . From the data, XRD showed the most significant influence on the process of anodizing, visible from the top of the resulting diffraction. As the potential increases, the intensity will decrease while the plane increases. The examination of corrosion resistance with EIS showed that the first step in the process of anodizing is an increase of R_{elp} associated with an increased area coated by a layer of oxide. **Copyright © 2016 Praise Worthy Prize S.r.l. - All rights reserved.**

Keywords: Anodizing, Potential, Surface Characteristics, Roughness, Corrosion

I. Introduction

Aluminum alloy is a very promising material because it has an excellent strength to weight ratio, low density, high thermal conductivity, high dimensional stability, good machinability and the ability to be recycled [1]-[4].

It is also available in many forms (e.g., rolling, extrusions, stampings, forgings, castings, and composites like Al-MMC) with relatively low production costs and suitability for a wide range of merger and surface treatment techniques [5]. Unfortunately, the overall advantage is limited due to inferior corrosion resistance and wear. When this material is used in industrial environments, it requires a further treatment to improve the corrosion resistance, such as the anodizing process. Numerous authors have managed to increase the wear and corrosion resistance of aluminum alloys through this anodizing method, among others [3]-[4], [6]-[7].

One of the factors greatly affecting the anodizing process is the potential. The applied potential will affect the thickness of the oxide layer formed after the anodizing process. The higher the used potential is, the higher the potential difference and energy yield.

This will result in a greater energy needed to break the bonds of ions at the cathode so that the ion-ion cathode will be separated from the bond. The more the ion-ion cathodes are separated from the bond, the greater the energy is produced, and more and more ion of cathodes are attached to the aluminum surface [8].

On the other hand, the use of different cathodes during anodizing process has not been widely explored.

The cathode can later protect aluminum from corrosion by forming an oxide layer on the surface of the anode (aluminum). Some metals commonly used as cathode are corrosion-resistant steel [9], platinum [10], and lead [11].

Setyarini et. al [12] found out that titanium positioned as the cathode in the aluminum anodizing process can penetrate into the film formed after the process is completed. In this study, the aluminum anodizing process is performed using titanium as a cathode on the variation of potential between 15-30V.

Due to its high corrosion resistance, titanium is expected to improve the corrosion resistance of aluminum. The focus of this study was on the morphological characteristics and corrosion resistance after the anodizing process is completed.

Morphological characteristics were determined using SEM and XRD before the corrosion resistance was evaluated by electrochemical impedance (EIS).

II. Experimental Method

II.1. Materials

The aluminum alloy used in this study was AA6061 with a composition of Si 0.4%, Mg 0.8%, Fe 0.7%, Cu 0.15%, Zn 0.25%, Ti 0.15%, Mn 0.15%, Al 97.55%.

The dimensions of the specimens used were 30 mm \times 5 mm \times 5 mm.

Specimens were smoothed using grit paper grade ranging from coarse to fine.

II.2. Method

II.2.1. Pretreatment

Oil or fat contained in the aluminum alloy surface was removed by degreasing prior to the anodizing process.

Degreasing was done with a solution of 15% sulfuric acid and heated at a temperature of $60 \pm 2^\circ\text{C}$ for 10 minutes, then rinsed with distilled water. Aluminum surfaces were polished by means of etching, which also aims to remove the layer of aluminum oxide film.

Etching was done by immersing the aluminum in a solution of caustic soda. The temperature employed was generally about $30 \pm 2^\circ\text{C}$ for 5 minutes, followed by a rinse with distilled water. Any black spots formed on the etching were removed by a desmutting process.

This process used a solution of nitric acid with a concentration of 10% carried out at a temperature of $30 \pm 2^\circ\text{C}$ for 5 minutes, then rinsed with distilled water.

II.2.2. Aluminum Anodizing

After the pretreatment process is finished, the aluminum was connected to the anode (positive pole) in the power supply and then immersed in an electrolysis bath measuring $12\text{ cm} \times 12\text{ cm} \times 10\text{ cm}$ containing 1M phosphoric acid solution at a temperature of 5°C . On the other side, a cathode (negative pole) in the power supply was connected to a titanium plate, according to the potential and anode-to-cathode spacing that had been planned. The power supply was then turned on and the walking process allowed to run for 60 minutes.

II.3. Data Collection Procedures

II.3.1. The Characterization of Coatings

Phenom scanning electron microscopy (SEM) was used to observe the surface morphology.

Qualitative analysis of the film coating was done by energy dispersive x-ray (EDX), and the phase analysis was carried out by the pan-analytical x-ray diffraction (XRD) with Cu K α radiation ($\lambda = 1.5425\text{ \AA}$).

II.3.2. Surface Roughness Testing Procedures

The roughness testing of materials in this study was performed using a SurfTest Mitutoyo SJ-301 Portable Surface Roughness Tester. Some tests were performed on three different surface samples of each variable, and each test was repeated three times for each sample.

II.3.3. Polarization Testing Procedures

The polarization was tested using an electrochemical method on a 3.5% NaCl solution. The test was performed using a potentiostat Autolab (PGSTAT302N) to investigate the corrosion properties.

A three-electrode system was used here, including the working electrode exposing 1 cm^2 surface area for

anodizing, a saturated calomel electrode (SCE), and a platinum plate, each of which is used as both a reference electrode and a counter electrode. Electrochemical impedance measurements were carried out at room temperature with AC amplitude in open circuit potential of 10 mV and within a frequency range of 100 kHz - 1MHz, with 5 points per decade. The software used was NOVA 1.7.

III. Results and Discussions

III.1. Structural Analysis

The structural characteristics of the coating film obtained from the anodizing process with some variation of the potential on phosphoric acid electrolyte are shown in Fig. 1. This image shows the structure of amorphous alumina as it has been reported in the literature [6].

Diffraktogram XRD showed the highest peak on 38 and 45 that can be assumed with a face-centered cubic (JPCDS 04-0787), as reported by another authors using sulfuric acid electrolyte [13]-[14].

Overall potential produces the same phase with different peak heights.

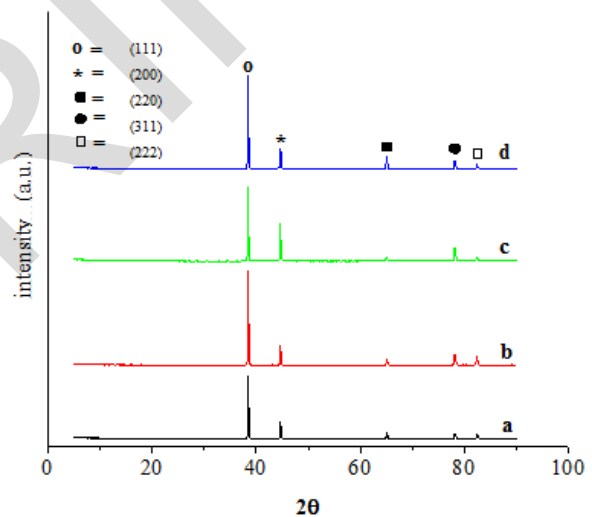


Fig. 1. XRD pattern of anodic layer formed with different potentials: (a) 15V, (b) 20V, (c) 25V, (d) 30V

The primary effect of the anodizing process appears at the top of the resulting diffraction. At low potentials, the diffraction peaks correspond to the (222) plane as shown in the peak of 83.

As potential increases, the intensity will decrease and increase the plane (111). This change is likely to occur because during the anodizing process, Al atoms in the plane (220) and (200) are reduced and the Al atoms in the plane (111) are relatively constant.

III.2. Morphological Analysis

The SEM of aluminum films are shown in Figs. 2 and that of aluminum substrate without anodizing process is shown in Fig. 2(a).

It appears that the surface has a natural film layer with uneven size as well as excessive pitting arising on the surface. After the anodizing process, as shown in Figs. 2(b)-(e), a change in surface morphology occurred in which the entire area was covered with a layer of pores related to one another.

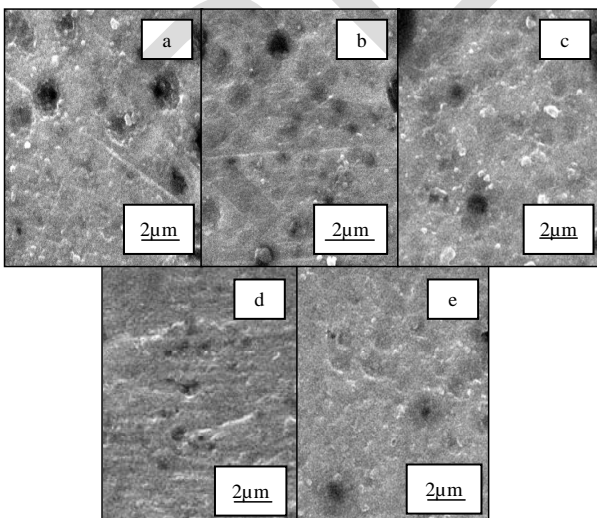
The results showed that a higher potential of electricity supplied during the anodizing process led to larger pore size evenly occurring on the surface of the specimen, in turn resulting in increased surface roughness values. This is in accordance with the roughness test results that can be seen in Fig. 3.

One factor that affects the formation of surface morphology of porous coating on the aluminum anodizing process is an O₂ bubble [7]. The same phenomenon has been seen in H₃PO₄, an electrolyte used by Habazaki *et. al.* in their research using titanium [15]. It can be concluded that pits, cavities and cracks formed during the anodizing process are likely to occur due to the formation of O₂ bubbles during the process.

In Fig. 3, it can be seen that surface roughness decreases along with increased potential. Before the anodizing process, the surface roughness reached 1.266 μm, whereas after being anodized with a potential of 15 V, the roughness values decreased by 0.877 μm.

When the potential was increased to 30V, it resulted in a surface roughness of 0.674 μm, suggesting that the surface of the material after anodizing process will have a smoother and homogeneous surface compared with not anodized materials. In general, it can be said that the surface roughness and cracking occurred during the anodizing process would reduce the lifetime and corrosion resistance. Thus, the reduction of the amount of surface roughness formed on the oxide layer is assumed to be able to increase the lifetime of the anodizing results.

This corresponds to a study conducted by Shahzad *et.al.* stating that the fatigue life increased with decreased surface roughness [16].



Figs. 2. The surface morphology of anodized aluminum alloy at various potentials: (a) substrate 15V, (b) 20V, (c) 25V, (d) 30V

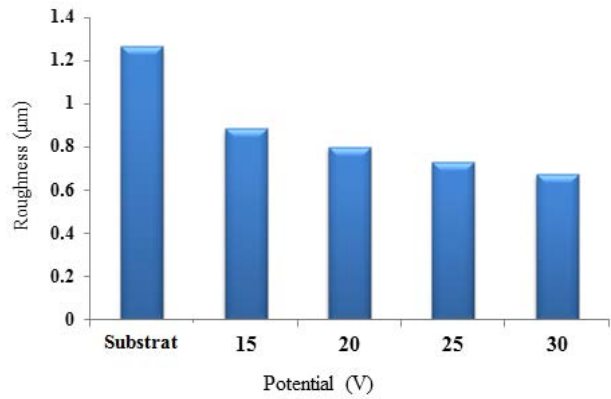


Fig. 3. Surface roughness after anodizing with different potentials

III.3. Anodic Films Impedance Spectra

An impedance diagram for some variations of potential applied to the 3.5% NaCl for the anodic film layer formed in the process of anodizing using phosphoric acid electrolyte is depicted in Figs. 4.

Nyquist plots are characterized by their capacitive loop. Some studies have found that a low frequency is associated with the properties of the barrier layer, while the medium to high frequencies influence the properties of the porous layer [17]-[19]. The anodizing process results in anodic layers consisting of two layers, namely barrier layer that is very thin and porous layer that is relatively thick.

Thus, in order to explain the EIS parameter on the anodic layers, the barrier layer and porous layer are distinguished into two different phases that are independent.

The electric and dielectric natures of each layer phase are described in the electrical equivalent circuit that consists of series and parallel resistors and capacitances [20], in which each barrier layer and porous layer has its own capacitive and resistor as modeled in Fig. 4(b).

In general, it can be said that the shape of the curve with different potentials for specimens after being exposed to the 3.5% NaCl solution is quite similar, where there is an increase in diameter loop with increased capacitive potential applied during the anodizing process.

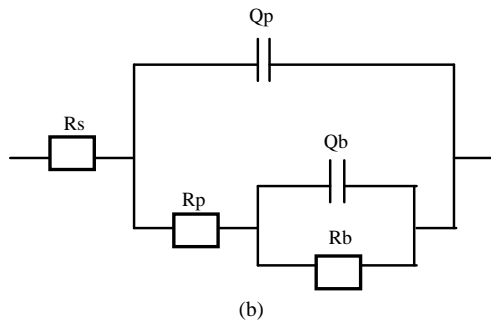
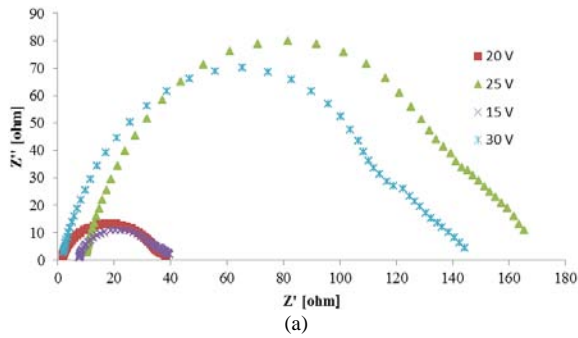
The modeling of the capacitances from the rail solution between the reference electrode and anodic films indicates that Rel is usually worth very little in NaCl solution. Cepp capacitance and resistance Rp represent the properties of the porous layer, while the barrier layer is indicated by CEPb capacitance and resistance Rb.

The constant phase element (CPE) is used to calculate the existing properties in the layers [17]. Thompson stated that the CPE can be calculated by the following equation [18]:

$$CPE = 1 / (2\mu JfC)^{\alpha} \quad (1)$$

As seen in Table I, Relp has a higher value than Rp.

This indicates that the resistance of the anodic film layer depends on the inner lining.



Figs. 4. (a) Nyquist diagram of the anodic film of AA 6061 (b) Equivalent circuit used to adjust the data of anodic film of AA 6061 impedance

TABLE I
CIRCUIT PARAMETER FROM EIS DATA

Potential (V)	Element Circuit					
	Relp	CPEp	Rp	Relb	CPEb	Rb
15 V	19.87	335.88	0.992	88.87	465.81	0.998
20 V	48.08	189.12	1.001	121.08	289.14	1.002
25 V	198.88	54.55	1.001	234.05	112.76	1.001
30 V	224.88	42.89	1.002	334.88	89.93	1.002

An increase in the value of Relp along with the increase of potential is in line with the increase in the thickness of the coating as shown in Fig. 4(a) [19].

This is consistent with research conducted by Wu et al. stating that the first step in the process of anodizing is an increase in Relp related to an increased area coated by a layer of oxide. At the end of the anodizing process, the improvement in Relp is associated with the increased thickness of the oxide layer.

There is a decline in value before the 30 V rail (Figs. 4), which indicates that the film at 30 V has the lowest roughness (Fig. 3). Anodic film layer has two layers of interface, the interface film/electrolyte interface and substrate/film. Rel reflects the homogeneity of interfaces formed. A high value for Rel indicates that the interface film/substrate is softer so the potential changes will not result in significant changes to corrosion by the electrolyte during the film growth process.

IV. Conclusion

This research developed a process of anodization of AA 6061 using low potential and a titanium cathode in a solution of 1M phosphoric acid. In this research, the observation on the surface morphology resulted after the

anodizing process as well as the corrosion resistance was conducted. It can be said that the higher the potential is applied, the smaller the size of the diameter of the pore is formed, decreasing the surface roughness.

During the process of the voltage increasing, the pore formation, formed after the anodizing process, will improve, so the higher is the voltage given, the more subtle and solid the appearance of the aluminum film, after the anodizing process since the pores formed, become smaller. This causes the decrease of surface roughness by 53% compared to the substrate. In addition, this will also lead to a decrease in surface roughness, creating increasingly subtle layers. This will result in an increased corrosion resistance so that the area of contact with the corroding substance becomes smaller.

Acknowledgements

The authors wish to thank Femiana Gapsari Madhi Fitri and Sulistiyana for the valuable discussions.

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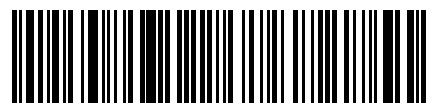
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Autorizzazione del Tribunale di Napoli n. 6 del 17/01/2007



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1970-8734(201605)10:3;1-3