



Effect of current density and concentration on microstructure and corrosion behavior of 6061 Al alloy in sulfuric acid



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ABSTRACT

Oxide coatings were prepared on 6061 aluminum alloys (AA6061) using anodization process in the electrolyte solution of H₂SO₄. The effects of applied current density (0.3–3 A/dm²) and sulfuric acid concentration (1–5 M) in a wide range on the morphology, thickness, phase formation, elemental composition, and corrosion resistance of alumina ceramic coatings have been investigated using X-ray diffraction, scanning electron microscopy, energy dispersive X-ray spectrometer and potentiostatic polarization test. Applying high current density resulted in higher growth rate of the oxide, but made the oxide layer more porous and less protective on the AA6061 alloy. Increasing sulfuric acid concentration also enhanced the reaction rate. A vigorous reaction taking place in the electrolyte with a high sulfuric acid concentration of 3–5 M led to the deposits cracked which deteriorated corrosion resistance. The coating with the best corrosion resistance ($I_{\text{corr}} = 1.203 \times 10^{-10}$ A/cm²) was obtained by anodizing at the current density of 1 A/dm² in an electrolyte with the sulfuric acid concentration of 1 M.

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1. Introduction

The aluminum alloy has favorable properties such as low density (2.71 g/cm³), high ductility and good machinability. These properties make it valuable in a number of applications including communications and computer components, living commodities, photoelectric and semiconductor devices [1]. Nevertheless, because aluminum is one of the most reactive metallic elements, aluminum and its alloys are prone to corrosion that restrains the application potential. For example, the oxide formed on the 6061 aluminum alloys (AA6061, Al-Mg alloy) with more amount of magnesium surface is not able to protect the substrate sufficiently. Therefore, developing an effective corrosion protection technology is important for utilizing the advantages of the aluminum alloy. Anodization is a useful technique for forming protective films on aluminum and aluminum alloys. High purity aluminum (>99.99%) has very low impurity and formed anodization easily that has attracted extensive researches [2–5]. But high purity aluminum has low strength that limits practical application. The most popular commerce available high-strength AA6061 alloy has been widely used in many component products especially in the semiconductor, optoelectronics and mobile industry. During anodization, the direct current (DC) electricity was generally applied to the aluminum as the anode and the other metal as cathode in an acidic electrolyte such as sulfuric acid. When current flowed through the cell, the oxygen and hydroxyl

ions migrated from the electrolyte onto the surface of the anodizing aluminum alloy, where they reacted with the dissolved aluminum ions to form a sealed aluminum oxide barrier layer. On the other hand, hydrogen ion was attracted to the cathode, where it formed hydrogen gas bubbles and dissipates into the air [6].

The quality and thickness of anodized coating can be controlled by the current density, concentration, compositions, temperature of electrolyte and anodization time. Generally, current density is one of the most important parameters affecting microstructure and properties of the coating. Additionally, the attack on the anodized coating by the acid will reduce the strength and result in the anodized coatings with varied properties. Although a variety of acids are used such as sulfuric acid [7,8], phosphoric acid [9,10] and oxalic acid [11–13], the most common acid used for aluminum alloy anodizing is sulfuric acid. This is ascribed to the fact that the sulfuric acid is not only obtained easily but also can lead to forming barrier layer quickly. Most researches used high-purity Al [3,4] or low-purity 1050 Al alloy [8] for anodization at a low concentration of 0.3–1 M and a voltage of 17–27 V or a current density of 1.2–1.8 A/dm² in electrolyte solution of sulfuric acid. The microstructure and optical property were studied. No corrosion behavior was reported above. Regarding AA6061, Cheng and Chou [7] studied the breakdown voltage and hardness of anodized AA6061 at concentration of 0.1–0.3 M and voltage of 25–60 V. Sayuri et al. [14] studied anticorrosion behavior of AA6061 deposited by cerium based conversion coating in 0.5 M NaCl aqueous solution. The best I_{corr} of 1.4×10^{-7} A/cm² was obtained by some coating parameters that affected microstructure. It is of thus interest to understand the microstructure and corrosive

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behavior of AA6061 anodized at a wide process window. In this article, we have investigated the AA6061 anodization in wide range of current density (0.3–3 A/dm²) and sulfuric acid concentration (1–5 M) for the evolution of microstructure. The microstructure characterization and influence on corrosion behavior were performed. A potentiostatic polarization method was adopted to evaluate corrosion characteristics of metal specimens [14,15]. The measured corrosion current provided a valuable means of rapidly determining the corrosion resistance of a metal. In a typical polarization curve, lower corrosion current density corresponded to lower corrosion rate and better corrosion resistance of the coating. The best corrosion could be obtained by anodization at the current density of 1 A/dm² and sulfuric acid concentration of 1 M.

2. Experimental procedure

2.1. Substrate preparation

The rectangular samples (100 mm × 40 mm × 1 mm) of AA6061 alloy were prepared as the substrates for the experiments. The nominal composition of this alloy by wt% is 0.90% Mg, 0.66% Si, 0.40% Fe, 0.24% Cu, 0.15% Cr, 0.07% Mn, 0.05% Zn, and Al was the balance. Prior to anodizing, the following procedures were performed for a clean and smooth surface. Specimens were abraded with the abrasive papers of successive grades from #800 to #2000 and then ultrasonically degreased in the acetone and followed by the isopropyl alcohol for 15 min each. To remove the oxide layers the specimens were alkali washed in a 1 M sodium hydroxide solution for 10 min and then were ultrasonically washed in de-ionized (DI) water for 15 min. After alkali wash, the samples were immersed in 1 M H₂SO₄ to remove aluminum hydroxide residue and then rinsed with DI water to get the clean surface of the specimens.

2.2. Anodizing

Fig. 1 shows the schematic apparatus of the anodization experiment. The electrochemical cell consisted of a two-electrode system e.g. the Ti alloy plate acting as the counter electrode and the AA6061 sheet acting as the working electrode i.e. the anode. The anodization process was carried out at room temperature (25 °C) for 30 min in a water-cooled electrochemical cell containing diluted sulfuric acid with agitation. The

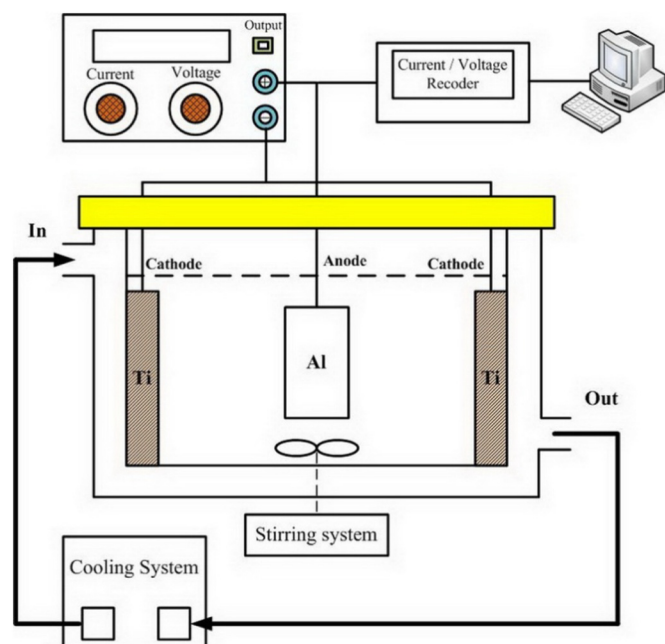


Fig. 1. Schematic diagram of the anodization apparatus.

Table 1

Anodization conditions and the resulting thickness as well as the corrosion potential (E_{corr}), current density (I_{corr}) of the anodized coatings.

Specimen	Anodized film formation conditions			Sample characteristics		
	Sulfuric acid concentration (M)	Current density (A/dm ²)	Reaction time (min)	Thickness (μm)	E_{corr} (V vs.SCE)	I_{corr} (A/cm ²)
A	1	0.3	30	2.17	-1.5459	1.150×10^{-9}
B	1	1	30	7.64	-1.2029	1.203×10^{-10}
C	1	3	30	25.14	-1.0565	2.259×10^{-10}
D	3	1	30	7.75	-1.1918	1.314×10^{-9}
E	5	1	30	8.42	-1.1593	3.213×10^{-9}

anodic AA6061 alloy substrate was immersed in the center of electrolyte. Two Ti alloy plates mounted on the inner surface of the tank were used as a cathodic counter electrode. The distance between the working electrode and counter electrode was 4 cm. The anodization process was performed by a constant current control mode, so the anodic voltage varied with anodizing time. Five alloy samples, namely A, B, C, D and E, were anodized in the sulfuric acid bath at different experimental conditions to study the evolution of microstructure and corrosive behavior. The experimental parameters such as current density, sulfuric acid concentration and reaction time were listed in Table 1.

2.3. Characterization of anodized coatings

Scanning electron microscopy (SEM, Hitachi S-3000N, Japan) was used to examine the morphology and thickness of the anodized samples. The film composition of the anodized samples was analyzed by using Energy Dispersive X-ray Spectrometer (EDX) which was attached to SEM. The crystal structure was characterized using X-Ray Diffractometer (Bruker D8 Discover) with Cu K α source ($\lambda = 0.154 \text{ nm}$) scanned at a wide angle of $2\theta = 20^\circ\text{--}100^\circ$.

2.4. Electrochemical test

Fig. 2 shows the schematic potentiostatic test to investigate the corrosion resistance of the anodized films obtained under different

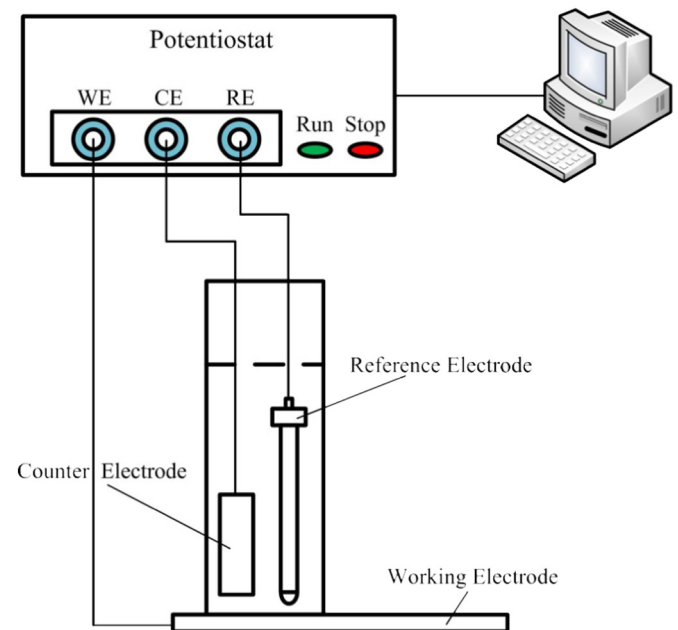


Fig. 2. Schematic diagram of the apparatus used for the determination of polarization curves of the anodized AA6061 in a solution using a potentiostat.

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