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Statistical Optimization of Exploring the Effect of Electrolyte Types and Additives on the Hardness of Aluminum 6061 Alloy during Microarc Oxidation process

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Abstract - Aluminum 6061 alloy is the most frequently used aluminum alloy in automobile, aircraft, and aerospace industries. However, poor wear and corrosion resistances, considerably affecting their scaled-up applications. Microarc oxidation (MAO) is ecofriendly, straightforward, efficient, and rapid technique for improving the surface characteristics of aluminum alloys and proves to be a highly effective surface treatment technique to enhance the corrosion and wear resistances of aluminum alloys. This article describes progresses realized in recent years for understanding the effects of electrolyte type, electrolyte composition, and additives on the morphology and properties of MAO coating film, and on the hardness of MAO coatings .Usually, applying an electrolyte system and introducing some additives in appropriate concentrations can improve the quality of the MAO coatings and their hardness, whereas some additives have negative effects on the hardness which is related to the corrosive behavior of aluminum. The statistical analysis showed that the microhardness values of the MAO coatings which was prepared in Na_2AlO_2 electrolyte was greater than the one which was prepared in Na_2SiO_3 electrolyte with percentage of 62 %. It is also found that the coatings generated by using pulsed-bipolar mode power source in MAO gives superior characteristics, and adding additives to electrolyte is not always enhance hardness and wear resistance.

Keywords: *Microarc oxidation, 6061 Aluminum alloys, Corrosion resistance, Wear resistance, Coating, Electrolyte.*

1. Introduction

Aluminum alloys are widely used in various sectors, such as aerospace, automotive, and shipbuilding industries owing to their remarkable mechanical properties, high strength-to-weight ratio, and rapid and low-cost production. These alloys can incorporate different alloying elements, including copper, magnesium, zinc, silicon, manganese, and lithium, which classify aluminum alloys into specific series. For instance, alloys containing copper are categorized under 2000 series, 3000 series for manganese, 6000 series for magnesium and silicon, etc. [1]–[5].

Aluminum 6061 alloy which called “structural Al,” is the most frequently used aluminum alloy in automobile, aircraft, and aerospace industries. The remarkable benefits of aluminum alloys make them a compelling alternative for replacing or substituting steel and cast-iron parts. Moreover, these alloys exhibit excellent formability, high extractability, and moderate strengths [6]–[9]. Magnesium and silicon are two major constituents of aluminum 6061 [10], [11]. The chemical composition of aluminum 6061 alloys is presented in Table 1 [12], [13].

Table 1. Chemical composition (wt.%) of Aluminum 6061 Alloy.

Elements in Al 6061 alloy	Fe	Si	Cr	Zn	Mn	Ti	Cu	Mg	Al
wt %	0.36	0.6	0.23	0.22	0.12	0.14	0.18	0.93	Rest

Despite possessing good mechanical properties, aluminum and its alloys exhibit poor wear and corrosion resistances, considerably affecting their scaled-up applications [14]. Therefore, they need surface treatments to enhance their service life. The phase composition, microstructure, and stress–strain conditions should be considered when selecting the appropriate surface treatment techniques [15]. Several effective surface modification techniques can be employed for Al alloys, such as anodization of coatings, chemical conversion, electrical discharge machining, electrochemical deposition, dip-coating [16]–[18], microarc oxidation (MAO), and anodic spark deposition [19]–[21].

The conventional anodizing process is a time-honored surface treatment method employed to augment the surface properties. It converts aluminum to aluminum oxide (alumina, Al_2O_3) using an acid or a complex anodizing electrolyte. However, this technology has certain technical limitations, including restricted thickness capabilities, low growth rates, and potential health concerns, because of the use of hazardous electrolytes. Furthermore, anodizing produces porous anodic oxide layers that provide some corrosion protection; however, they interact with the working environment because of their porous nature, leading to localized corrosion caused by the presence of aggressive species [22]–[24].

The MAO technique is a modified version of anodizing and is a noble, ecofriendly [25]–[27], straightforward, efficient, and rapid technique for improving the surface characteristics of aluminum alloys. This surface coating technique uses in situ growth of a thick metallurgical oxide protective coating on metal surfaces, such as aluminum and magnesium [28]–[33]. Furthermore, MAO method overcomes the limitations associated with the conventional hard anodizing process. MAO method is effective in treating challenging Al–Si alloys and addressing the tendency to produce crystalline phases of Al_2O_3 instead of amorphous Al_2O_3 [34]–[36].

MAO is an anodic oxidation process, but the oxide growth mechanism is a different and complicated process [37], [38]. The process is conducted in a stainless-steel tank containing an electrolyte solution. During the process, the anode alloy is oxidized, and then a substantial voltage (ranging from tens to hundreds of volts) is administered across the anode and second electrode. The magnitude of this voltage is sufficient to generate persistent sparks within the electrolytic medium connecting the electrodes. The process lasts several tens of minutes to grow a uniform, thick (10–100 μm), and dense oxide film with suitable porosity and chemical compositions [39]–[44].

1.1 MAO Coating Growth Mechanism

Whether employing AC (alternate current) or DC (direct current) electrical power, the fundamental principles of MAO coatings are the same. As the applied voltage increases, a permeable insulation layer are formed, which is characterized using a columnar arrangement, generating many gas bubbles, as shown in Figure1. When the voltage reaches the breakdown voltage in a few isolated weak areas within the insulating layer, dielectric breakdown and spark discharge occur. This results in the generation of numerous fine, evenly distributed white sparks on the sample surface. As the number of sparks drops, many small, uniform micropores are formed. During the MAO process, sparks undergo transition in their colour, shifting from white to yellow and finally to an orange-red hue. The final spark transition from yellow to orange-red in the microarc stage is related to a fast coating growth rate. As the voltage value and coating thickness increase, sparks decrease, but they become intense, leading to rough surface morphologies. With further increase in voltage, a porous and loose region of MAO coating is developed, which generates a splash of the coating materials and produces localized significant ablation features. Under MAO treatment, aluminum alloy develops two distinct layers: a thick layer with exceptional corrosion resistance and a porous layer that enhances the adhesion bonding strength of organic coatings applied to its surface [45]–[54].

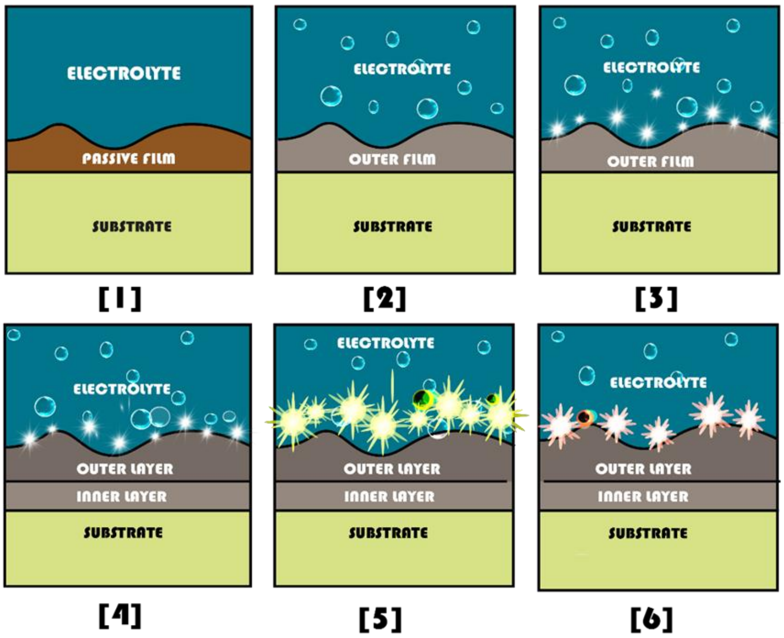

Fig. 1. Schematic of the growth mechanism of MAO coating. [1] passive film before applying high voltage, [2-3] dielectric breakdown and spark discharge occur after applying high voltage, [4] inner layer growth begins, [5] sparks shifting from white to yellow and [6] spark transition to orange-red hue

1.2 MAO Process Parameters

Microstructure and composition of substrates are the primary factors determining the ultimate properties of the MAO coating [55], [56]. Additionally, the electrolyte composition, different concentrations, additives [57], [58], electrolyte temperature [59], [60], and other process parameters, such as current, voltage, frequency, and power density, influence the coating properties. Owing to the direct interaction between alloys and electrolytes during coating formation, the composition of the electrolyte has much more influence on the final properties of the coating compared to other factors. Adding different concentration and additives (i.e., Al_2O_3, MoS_2, TiO_2, ZnO, and Na_2WO_4) to the base electrolyte are highly effective methods to attain diverse improvements in coating properties, such as hardness, thickness, morphology, porosity, and wear resistance [61]–[68].

2. Objectives

The aims of this work are to improve the hardness and wear resistance of ceramic coatings on 6061 Al alloy prepared by MAO method. A statistical optimization study is used to investigate the suitable MAO process parameters of 6061 Al alloy, such as power supply, current density, oxidation time, and electrolyte type according to experiments of previous works.

3. Methods

In MAO process, micro arc is created through electrical potential in an electrolyte. Power supply, current density and oxidation time will be the main parameter. Electrolytes and additives will also play important roles in coating quality.

3.1 MAO process parameters

One of the most crucial components of MAO treatment is power supply, and the electric pulse substantially influences the quality of the oxide films and required processing time. Different electrical conditions can be employed during the MAO process, including DC, pulsed-DC, or pulsed-bipolar current modes. When compared to coatings generated using DC and pulsed-unipolar modes, utilizing a pulsed-bipolar mode power source in MAO yields coatings with superior characteristics. These coatings exhibit uniform thickness, reduced flaws, and a dense and compact structure [69]–[72]. Creating a compact oxide film that serves as a barrier layer on the anode surface during the MAO process correlates with an appropriate increase in current value and a reasonable current pulse frequency [73], [74].

Lin and colleagues [67] examined the surface properties of MAO coatings in aluminum 6061 alloys at various current densities to clarify the process of developing MAO coating. The findings proved that, among four different types of specimens, the coating layer of MAO generated at 15 A/dm^2 exhibited the highest hardness and optimum wear resistance.

As the oxidation time in the MAO process increases, the thickness of the ceramic coating created on aluminum alloy also increases, thereby reducing the coating wear. Moreover, with prolonged oxidation time, the content and thickness of crystalline substances within coatings increase. However, the growth rate of the coatings decreases over time. Additionally, coatings subjected to extended oxidation time display large pore sizes and low porosity on their surfaces [75], [76].

3.2 Electrolytes and additives

The commonly used alkaline electrolytes for MAO on aluminum alloys consist of sodium and potassium silicates, aluminates, and phosphates. These alkaline solutions are more ecofriendly than acidic anodized solutions, resulting in fewer disposal concerns and lesser potential environmental harm [77], [78].

Incorporating nanoparticles into an electrolyte solution considerably affects the morphology, hardness, corrosion resistance, and wear resistance of MAO coatings. Numerous nanoparticles have been studied for their corrosion performance on MAO coatings, including carbon nanotubes, sodium tungstate dihydrate ($Na_2WO_4{\cdot}2H2O$), silicon dioxide (SiO_2), TiO_2, Al_2O_3, zirconia (ZrO_2), ceria (CeO_2), yttrium oxide (Y_2O_3), silicon nitride (Si_3N_4), and borax [79]–[82].

3.2.1 Silicate electrolyte system

A silicate electrolyte bath is effective for the MAO process [83] because it provides a wide range of temperatures and currents for the electrolyte. Furthermore, it promotes passivation of the alloy surface and forms an oxide film containing silicon dioxide, resulting in a high surface hardness [84], [85]. Bosta and coworkers [86] subjected aluminum 6061 alloys to MAO under various alkali silicate electrolyte temperatures. Notably, the electrolyte temperature had a considerable effect on all surface properties.

3.2.1.1 Potassium hydroxide (KOH) and sodium metasilicate (Na_2SiO_3) electrolyte system

Sharma and coworkers [87] studied the effects of composition KOH:Na_2SiO_3 ratio on hardness. The results showed a slight increase in hardness when changing the KOH:Na_2SiO_3 ratio from 5:5 to 5:10 for up to 30 min. However, it was observed that the rate of increase was considerably high within the 60-minute timeframe when the ratio was 10:10. Jayaraj and coworkers [88] tested MAO coatings on aluminum 6061 alloys using a silicate electrolyte and then optimized the MAO parameters to achieve coatings with the smallest porosity and maximum hardness. The optimized hardness value was 1360 HV at an MAO current density of 0.11 A/cm^2, an oxidation time of 26.61 min, and an inter-electrode distance of 6.33 cm.

In contrast, Wang and colleagues [89] conducted an optimization study on the AC voltage value during the MAO process using a mild alkaline silicon electrolyte on an aluminum 6061 alloy substrate. The experiment involved treating the alloy substrate for 5 min at various AC voltages. The findings indicated that the hardness of the outer layer exhibited an increasing trend when voltage in the range of 200 V was applied, but the hardness decreased when the AC voltage reached 220 V. Similarly, Wang [90] studied the optimal DC voltage of the MAO process executed on Al 6061 alloy substrate using a mild alkaline silicon electrolyte for 5 min while maintaining a constant amplitude of 200 V AC. They found that the microhardness gradually increased, reaching 1900 HV at 220 V. Furthermore, Jadhav [91] investigated the impact of current, voltage, electrode gap, and the existence of graphene in the electrolyte on the formation of the coating layer. Findings indicated that the MAO process did not result in considerable graphene absorption. A maximum thickness of 200 μm in the sample cross-section was achieved with an applied voltage of 197 V, an oxidation time of 30 min, an electrode distance of 20 mm, and a current of 0.3 – 1.0 A.

3.2.1.2. Silicate electrolyte with binary additives of $(NaPO_3)_6$ and H_3BO_3

Zhu and coworkers [92], [93] studied the effect of additives, such as $(NaPO_3)_6$ and H_3BO_3, on silicate electrolytes to improve the quality of MAO coatings on aluminum 6061 alloys. They subsequently investigated the prepared layers using SEM and found that $(NaPO_3)_6$ and H_3BO_3 could improve the MAO layers by developing thick, compact coatings with few defects. However, it was observed that as the percentage of $(NaPO_3)_6$ increased, the microhardness of the coatings decreased.

3.2.1.3. Silicate electrolyte with Al_2O_3 micropowder as additive

Wang and colleagues [94] studied the impact of adding Al_2O_3 micropowder into the silicate electrolyte to improve the quality of MAO coatings on aluminum 6061 alloys. SEM images showed that the dimensions of micropores on the MAO coatings decreased when Al_2O_3 was added compared to when it was absent from the electrolyte. Furthermore, the surface microhardness of the coatings increases up to 2.0 g/L and decreased subsequently.

3.2.1.4. Silicate electrolyte with MgO micropowder as additive

Wang and colleagues [95] studied the effect of incorporating MgO micropowder in different percentages to silicate electrolytes to improve the quality of MAO coatings on aluminum 6061 alloys. The coating thickness was analysed using SEM, and the hardness of the coating was measured. The highest hardness was observed at an MgO concentration of 6 g/L; beyond this concentration, the hardness slightly decreased.

3.2.1.5. Silicate base electrolyte containing cellulose $(C_6H_{10}O_5)n$ as additive

In a study by Song and coworkers [96], the MAO coating of aluminum 6061 alloy was prepared using a silicate-based electrolyte that included cellulose. The primary objective of the study was to evaluate the tribological properties of the resulting layer. X-ray diffraction (XRD), SEM, and energy dispersive X-ray spectroscopy (EDS) determined that the presence of cellulose had a minimal impact on the hardness of the coating. However, an increase in cellulose concentration improved the roughness and thickness of the coating layer.

3.2.1.6. Silicate electrolytes with titania (TiO_2) as additive

Jiang and coworkers [97] studied the impact of TiO_2 nano-additive concentrations in silicate electrolytes on the formation of an MAO layer on aluminum 6061 alloys. The specimens were examined using SEM and EDS. The findings indicated that by adjusting the TiO_2 concentration, MAO coatings could reduce the mean depth of erosion rate. Nevertheless, elevating the TiO_2 concentration reduced the surface hardness of MAO coatings, resulting in large erosion pits on the surface of the coatings.

3.2.1.7. Silicate electrolytes with additive Na_2WO_4

Liu and colleagues [98] conducted a study to examine the influence of Na_2WO_4 additive on the corrosion resistance of MAO coatings formed on aluminum 6061 alloys using a silicate electrolyte system. Surface morphology analysis was conducted using SEM and XRD. The results revealed that incorporating Na_2WO_4 facilitated the formation of MAO coatings with high impedance and increased thickness. Notably, at a Na_2WO_4 concentration of 5 g/L, the corrosion resistance of MAO coatings was enhanced by 1.6 times compared to that formed solely in the silicate electrolyte.

3.2.2. Aluminate and phosphate electrolytes

A study by Peng and colleagues [99] aimed to investigate the impact of phosphate and aluminate electrolytes on the formation of MAO coatings on aluminum 6061 alloy. The cross-section microstructures and surface of the MAO coatings were examined using EDS and SEM. Furthermore, the corrosion resistance and wear properties of MAO coatings were assessed. The findings indicated that phosphate electrolytes resulted in the formation of a

bilayer coating structure. In contrast, aluminate electrolyte resulted in the formation of a single-layered dense Al_2O_3 coating with a hardness of 1300 HV. Consequently, the aluminate MAO layer exhibited a lower wear rate than the phosphate MAO layer. Wang and coworkers [100] studied MAO coatings on aluminum 6061 alloys using different electrolytes (Na_2SiO_3, Na_2AlO_2, and Na_3PO_4). The microhardness values of the MAO coatings were calculated as 1400, 2000, and 2180 HV for Na_2SiO_3, Na_3PO_4, and Na_2AlO_2 electrolytes, respectively.
Furthermore, Liu and coworkers [101] studied the effect of various electrolytes at a constant current (8 A/dm^2) for 80 min and an electrolyte concentration of 8 g/L using the hardness test. The results were consistent with those of Wang and coworkers, showing that hardness was the highest with Na_2AlO_2 electrolyte (~1270 HV) and lowest with Na_2SiO_3 electrolyte.

4. Results

The following chart summarizes the optimization of the hardness value for MAO coating under different conditions and electrolytes, as derived from different studies.

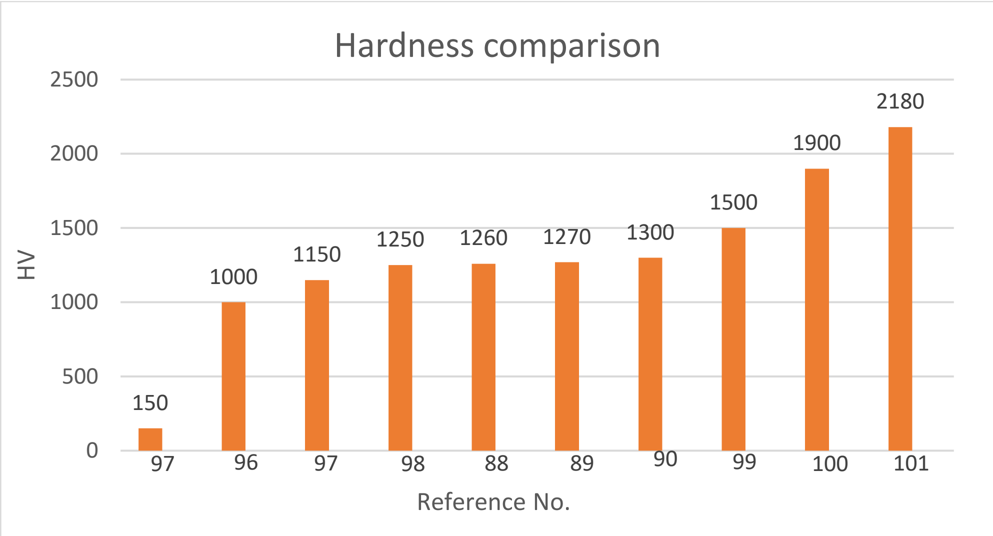

Fig. 2. Hardness values of MAO coatings that are developed using different electrolytes and conditions (the values extracted from references [88], [89], [90], [96], [97], [98], [99], [100], and [101]).

Figure 2 clarifies the effect of the MAO process parameters on the hardness of Al 6061 alloy. Before the MAO process, the hardness of the alloy was 150 HV, which increased considerably (≥1000 HV) after the MAO process depending on the electrolyte and other factors.
According to a study [101], a hardness value of 2180 HV was achieved using a Na_2AlO_2 electrolyte at a constant current (8 A/dm^2) for 80 min, which is the highest obtained value.
When silicate electrolytes are used in MAO coatings prepared for aluminum alloys, the resulting phases, such as silica, have lower hardness compared to the primary phases of α-Al_2O_3 and γ-Al_2O_3 in MAO coatings prepared in aluminate electrolytes. This implies that MAO coatings prepared in silicate electrolytes on aluminum alloy may have lower resistance to wear than those prepared in aluminate electrolytes [102], [103]. However, aluminate electrolytes are rarely employed owing to their instability, particularly at high aluminate concentrations [104], [105].

5. Discussion

To enhance the durability and wear resistance of ceramic coatings applied to 6061 aluminum alloy through the MAO method, a statistical optimization study was conducted. This study aimed to determine the optimal MAO process parameters for 6061 aluminum alloy, were investigated through experimentation, drawing on insights from prior research endeavors as follows:

1. Coatings formed by using pulsed-bipolar mode power source in MAO generate coatings with superior characteristics than other modes.

2. The most appropriate current density to produce suitable properties is $15A/dm^2$ with oxidation time range from 30 min to 80 min.

3. A silicate electrolyte bath is effective for the MAO process because it provides a wide range of temperatures and currents for the electrolyte. Furthermore, it promotes passivation of the alloy surface and forms an oxide film containing silicon dioxide, resulting in a high surface hardness.

4.In a silicate electrolyte without additives, the highest hardness value achieved at different parameters is 1900 HV.

5. Most studies have focused on improving the hardness and corrosion resistance of MAO coatings in silicate electrolytes with additives and investigated that some additives have negative effects on hardness such as $(NaPO_3)_6$ and TiO_2 nano additives, However, others have positive effects on microhardness as providing the additives at some percentage such as Al_2O_3 , MgO micropower, and some had a minimal impact on the hardness of the coating such as $(C_6H_{10}O_5)_n$.

6. Some additives just had influence on the coating thickness and corrosion resistance such as TiO_2 nano additives and Na_2WO_4.

7. The highest microhardness values of the MAO coatings is the film prepared in Na_2AlO_2 electrolyte.

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