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Research Article

Investigation of friction behavior of TiO₂: Al₂O₃ composite coating formed on AA2024 alloy by micro-arc oxidation

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ABSTRACT

In this study, an alumina (Al₂O₃) ceramic coating and a composite coating containing titanium dioxide (TiO₂) particles were prepared on surfaces of AA2024 substrates by micro-arc oxidation (MAO) treatment. The effects of micro-sized (≈20-30 µm) TiO₂ particles incorporated into electrolyte on the microstructure, phase and chemical composition, thickness, and friction behavior of the coating were investigated. The surface morphologies of the coatings were analyzed by scanning electron microscopy (SEM). The phase and chemical compositions of the coatings were evaluated by means of X-ray diffraction (XRD) and energy-dispersive X-ray spectrometry (EDS), respectively. The friction coefficient of the coatings was investigated using a pin-on-disc tribometer under condition of dry slip sliding. Moreover, the wear tracks were analyzed by SEM. Addition of TiO_2 particles into the electrolyte resulted in the reduction in number of pores and formation of smaller and more uniform pores in comparison with the Al₂O₃ ceramic coating. XRD analyses demonstrated that the TiO₂:Al₂O₃ composite coating was composed of anatase-TiO₂ and rutile-TiO₂, as well as pure aluminum, α -Al₂O₃ and γ -Al₂O₃ which are the primary phases. The friction tests showed a significant reduction in the friction coefficient of the composite coated samples which contains titanium oxide (TiO₂) particles in comparison with Al₂O₃ coated and uncoated samples.

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INTRODUCTION

The usage of aluminum (Al) alloys is increasing rapidly every year in industrial and engineering applications such as automotive, rail transport, shipbuilding, aviation, aerospace and construction sectors. [1-9]. The main causes for the increasing popularity of Al alloys are their versatile properties such as high specific strength, low weight, relatively low cost, good corrosion resistance, formability and recyclability. [6, 10]. However, their poor surface characteristic, especially low surface hardness, high friction coefficient and poor wear resistance have greatly limited their range of tribological applications. [2, 11]. Thus, surface treatment technologies have been playing a key role in enhancing their usability of aluminum alloys. [12-17].

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Micro arc oxidation (MAO) is a simple and effective surface treatment for forming ceramic-like coating films on metals. [12, 16]. This method is a rapid development new technology in recent years. To date, the materials most coated with MAO treatment are aluminum [18], titanium [19], magnesium [20], zinc [21] and their alloys. The coating films fabricated by MAO can have a series advantage such as high corrosion resistance, high wear resistance, uniform thickness and ideal adhesive strength to the substrate. [12, 16-18]. The composition of substrate materials and electrolyte, electrical parameters, processing temperature, oxidation time, type and size of additive have a large impact on the properties of MAO coatings. [17]. Thus, the ideal properties can be achieved by controlling the process parameters.

The substrate material plays a critical role in the components and properties of MAO coatings. In the other words, the dominant compositions of the MAO coatings depend on the substrate material. For example, the main content of the coatings formed on aluminum, magnesium, titanium and their alloys are Al_2O_3 , MgO and TiO₂, respectively. Furthermore, the additions of different particles in the electrolyte can affect the MAO treatment, and significantly change further the microstructure and the property of the coatings. [12, 22]. Recent studies have shown that the composite coatings obtained by adding of TiO₂ into the electrolyte have given better wear performance. [23-25]. However, there has been limited study on the tribological properties of TiO₂-doped Al_2O_3 composite coating formed on AA2024 alloy. [24].

Since the differences in substrate material result in different growth rates, structures, phase composition and element distribution of the MAO coatings, $TiO_2:Al_2O_3$ composite coating was formed on AA2024 aluminum alloy by MAO treatment with present study. The main motivation for this study is to add a new resource to the limited publication. The characterization of the coating surface was analyzed scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy-dispersive X-ray spectrometry (EDS), respectively. The friction behavior of the coating was determined using a pin-on-disc tribometer at the dry sliding conditions. The effect of the TiO_2 particles incorporated into electrolyte on the surface morphology, coating thickness and coefficient of friction was investigated.

MATERIALS AND METHODS

AA2024 is one of the most widely used groups of aluminium alloys and have been extensively used for

automotive and aerospace industries due to high strength and excellent machinability. [9, 27]. AA2024 alloy was used to as the substrate material. The chemical composition of AA2024 is shown in Table 1.

The samples with dimension 20x20x2 mm were successively polished with silicon carbide (SiC) papers of grit numbers of 400, 800 and 1200. Then, polished samples were rinsed with ethanol and dried prior to MAO process. The surface roughness values of the polished samples were measured approximately Ra≈0.10-0.13 µm by MarSurf PS1 tester. The MAO treatment of AA2024 substrates were carried out by the MAO system which was designed and manufactured by Plasma Technology Ltd. Two different electrolytes were prepared for the MAO process. The base electrolyte consisted of KOH (1 g/L), Na₂SiO₃ (2 g/L) and Al_2O_3 (3 g/L) and distilled water. The second electrolyte contained TiO₂ (3 g/L) particles, approximately \approx 20-30 µm in size, incorporated into the base electrolyte under similar conditions. An AC power supply was used in bipolar mode. The MAO process was performed with frequency of 700 Hz and a voltage of 550 V at 10 minutes operating time. The AA2024 samples and stainless-steel bath walls were set as the anode and cathode, respectively. The temperature of electrolytes was kept below 30 °C using a cooling system. After the MAO treatment, the samples were cleaned with ethanol and dried.

The surface morphologies and thickness of the coatings were analyzed by scanning electron microscopy and chemical composition of the coatings by SEM-EDS (Model: QUANTA 250 FEG). The coated samples were immersed in a liquid nitrogen bath and broke brittle to establish the coating thickness. The phase was analyzed by means of X-ray diffraction (Model: Malvern Panalytical Empyrean). XRD analysis was conducted with a scanning rate of 2°/ min from 20° to 90° (2 θ) at a wavelength of λ =1.5404 A°. The phases were defined by comparing the reflections obtained after the analysis with the peak lists of the JCPDS (Joint Committee on Powder Diffraction Standards). After MAO treatments, the friction behavior of the coatings was investigated under condition of dry slip sliding. The friction coefficient of uncoated and coated samples against the Al₂O₃ ball were determined under 2N load. Then, the wear tracks were analyzed by SEM.

RESULTS AND DISCUSSION

The surface morphologies of the Al_2O_3 coating and $TiO_2:Al_2O_3$ composite coating are shown in Figure 1. In general, the MAO is carried out at the voltages higher than

Table 1. The Chemical Composition of AA2024 Substrate

Element	Cu	Mg	Mn	Fe	Si	Zn	Cr	Al
(wt.%)	4.4	1.5	0.6	0.5	0.5	0.25	0.1	Balance

the breakdown voltage of the passive oxide film preformed on the surface and it is characterized by electrical sparks appeared on the anode surface. [12, 16]. During the process, micro-pores develop in the layer which grows by the continuous discharge process. These micro-pores allow continuing current flow and thus film growth. [16, 17]. As seen in Figure 1 (a, b), it is observed that the micro-pores have different diameters and random location on the surfaces. During the process, the irregular melting of Al has resulted in formation of some porosities, empty inclusions and volcanic formations. Moreover, there is few micro-cracks on the surface of MAO coating (Figure 1(a)). These pores and cracks formations are caused by molten oxide and gas bubbles thrown out of micro-arc discharge channels as a result of the nature of the MAO process. The cracks resulted from the thermal stress due to the rapid solidification of the molten oxide. [16-18, 28]. When Figure 1(b) is examined detail, it can be seen that less porous surface of composite coating which doped with TiO₂ particles. Moreover, pores are more

uniform in both size and shape. This is due to the strong adhesion of the additive to the surface and the filling of the discharge channels by TiO_2 particles. [24, 29]. In other words, the addition of TiO_2 particles relatively resulted in a decrease of pore diameter. Moreover, the addition of TiO_2 particles resulted in an increase of the coating compactness.

Figure 2 (a), (b) shows the cross-sectional images of Al_2O_3 coating and composite coating contained TiO_2 particles, respectively. When the coating thicknesses grown on the surface are examined, the thickness of layer was obtained as $\approx 24 \,\mu\text{m}$ in the Al_2O_3 coated sample, while the thickness was obtained as approximately $\approx 32 \,\mu\text{m}$ in the TiO_2 doped Al_2O_3 coated sample. This growth is thought to be due to the TiO_2 particles were placed among the micropores. This trend is the expected situation in MAO processes. It can be seen that these pores in Figure 1 (b) are fuller and uniform. This is the explanation that the pores are filled during the coating. In fact, TiO_2 particles increased the conductivity of the MAO electrolyte. The previous studies based



Figure 1. SEM images of (a) Al₂O₃ coating, (b) TiO₂:Al₂O₃ composite coating.



Figure 2. Cross-sectional microstructure for (a) Al₂O₃ coating, (b) TiO₂:Al₂O₃ composite coating.

on production of composite coatings containing micro or nano TiO_2 particles have shown an increase in coating layer thickness. [24, 29].

XRD graphs of the AA2024 sample and coated samples are shown in Figure 3. It is found that coated samples are mainly composed of Al, α -Al₂O₃ and γ -Al₂O₃. In addition, Al peak with intensity and much stronger was observed in the uncoated sample. After both MAO treatment, it was clearly seen that the intensity of Al peak has decreased. It can be said that the high-density peaks of Al come from the AA2024 substrate. The γ -Al₂O₃ characteristic peaks were observed at $2\theta \approx 38^{\circ}$, $2\theta \approx 44^{\circ}$, $2\theta \approx 66^{\circ}$ and $2\theta \approx 78^{\circ}$ corresponding to planes (311), (400), (440) and (533), respectively. The α -Al₂O₃ characteristic peaks were also detected at $2\theta \approx 46^{\circ}$, $2\theta \approx 66^{\circ}$ and $2\theta \approx 84^{\circ}$ corresponding to planes (012), (202) and (223), respectively.

These peaks correspond to both γ -Al₂O₃ and α -Al₂O₃ as mentioned in previous literature. [30- 32]. High pressure



Figure 3. XRD graphs of AA2024 sample, Al_2O_3 coating and TiO₂:Al₂O₃ composite coating.

and temperature helped to form the α -Al₂O₃ and γ -Al₂O₃ phases easily. The XRD graphs in Figure 3 show clear differences in phases between the Al₂O₃ coating and the TiO₂-doped Al₂O₃ composite coating. The anatase-TiO₂ and rutile-TiO₂ phases on surface of the TiO₂:Al₂O₃ composite coating indicates that TiO₂:Al₂O₃ composite coating had been successfully grown on AA2024 alloy using MAO treatment. Similar to the literature, the intensity of anatase-TiO₂ and rutile-TiO₂ phases was determined at $2\theta \approx 25.2^{\circ}$, $2\theta \approx 36.9^{\circ}$, $2\theta \approx 37.7^{\circ}$, $2\theta \approx 38.5^{\circ}$, $2\theta \approx 42.3^{\circ}$, $2\theta \approx 48^{\circ}$, $2\theta \approx 55.1^{\circ}$ and $2\theta \approx 62.60^{\circ}$ corresponding to planes (101), (103), (004), (200), (111), (200), (211) and (204), respectively. [33, 35].

The energy-dispersive X-ray spectrometry (EDS) analysis of the surface of TiO2:Al2O3 composite coating is shown in Figure 4. The results showed that the coating consists mainly of aluminum (Al), oxygen (O), titanium (Ti) and silicon (Si) with an atomic concentration of 41.85%, 52.14%, 3.61%, and 2.41%, respectively. The concentration of the titanium in the coating is greatly lower than aluminum and oxygen. From the literature, it is well known that silicon comes from the composition of electrolyte used in the growth of the composite coating. [29]. In fact, Figure 1 (b) shows that the TiO_2 particles were dispersed all over the surface of coating. Also from Figure 2, it is seen that the phases of TiO₂ particles were detected on the surface of coating. Lastly, the EDS results indicated that TiO₂:Al₂O₃ composite coating has been successfully grown on AA2024 alloy using MAO treatment.

The friction coefficient (COF) of the uncoated AA2024 alloy and Al_2O_3 coated and $TiO_2:Al_2O_3$ composite coated samples were evaluated by pin-on disk tribometer using Al_2O_3 ball as counterpart material. Firstly, the COF graphs were determined. Then the wear tracks were analyzed by SEM in order to identify the wear mechanism of coatings. The experiments were performed at total sliding distance of 60 m under 2N load for all samples. Figure 5 shows the changes in the COF as a function of sliding distance.



Figure 4. EDS analysis of the TiO₂:Al₂O₃ composite coating.

AA2024 substrate showed that the average COF value of 0.57-0.58 during the steady stage. The friction coefficient increased rapidly in the initial stages and then gradually decreased. After 10m, the friction coefficient became stable. Al₂O₃ coating and TiO₂:Al₂O₃ composite coating exhibited lower initial COF value than the uncoated AA2024 sample. During the test, it was observed that there was severe plastic deformation on the substrate.

The friction coefficient of both coated samples are much lower than that of the substrate. However, the samples covered by the progress of the test period have been found to have significant fluctuations in the coefficients of friction. This can be explained by the rapid occurrence of three body wear, especially in coatings produced with the addition of TiO₂ particles. [23, 24]. Adding TiO₂ particle affected the friction coefficient values. The lowest friction coefficient value was obtained with novel TiO₂ doped Al₂O₃ composite coated sample. The friction coefficient of the TiO₂:Al₂O₃ composite coating was obviously decreased because of the strong adhesion of the TiO2 particles to the surface and the filling of the discharge channels by these particles. [23-26, 29, 35]. Previous studies has shown that the anatase- TiO_2 , rutile-TiO₂ particles lead to achieve a compact and uniform morphology of coating surface that can reduce the friction coefficient of the substrate. [26, 35, 36]. Some TiO₂ particles



Figure 5. Coefficient of friction graphs of (a) AA2024 sample, (b) Al₂O₃ coating, (c) TiO₂:Al₂O₃ composite coating.

that are not tightly connected act as the third body rigid particles that are harmful to the coating itself. However, it is known that the rutile phase of TiO_2 is a phase that is more resistant to wear and can contribute in addition to the lower wear performance. [26].

Figure 5 also displays the SEM images of the wear tracks morphologies of the AA2024 substrate, Al₂O₃ coating and TiO₂:Al₂O₃ composite coating. As shown in Figure 5, the average width of the wear marks decreases as a function of the coating. This can be explained by the sample being worn and the applied coating creating a barrier against wear on the surface. Compared to the AA2024 substrates, both coatings have been observed to be shallow and narrow. AA2024 substrate showed signs of plastic deformation in combination with adhesive and abrasive wear. Some micro cracks were also observed and most of loosen wear debris were found in the wear track as shown in Figure 5 (a). The wear mechanism of the Al₂O₃ coating consisted mainly of micro cracks and the removal of the coating as flakes accompanied by the formation of the protective transfer layer as shown in Figure 5 (b). It is thought that the roughness on the surface of the Al₂O₃ coating ensures the adherence of the tribolayer by mechanical interlocking. [18]. Meanwhile, AA2024 substrate is directly oxidized to become α -Al₂O₃ and γ -Al₂O₃ phases due to a high temperature in the microarc zone. So, there is an ideal adhesion between the Al₂O₃ ceramic coating and AA2024 substrate. [37, 38]. The addition of TiO₂ particles resulted in an increase of the coating compactness and affect wear mechanism of the coating. The addition of TiO₂ particles into electrolyte the abrasive wear and detachment of the coating was effectively reduced as shown in Figure 5 (c). This is believed to be due to an increase in the thickness of the coating with the addition of TiO2 particles, as well as the capacity to carry loads.

CONCLUSION

In this study, Al_2O_3 coating and $TiO_2:Al_2O_3$ composite coating layer were formed on AA2024 alloy by micro arc oxidation (MAO) method. The effects of TiO_2 particles incorporated into electrolyte on the microstructure, phase and chemical composition, thickness, and friction behavior of the coating were investigated. The results from this study obtained are summarized below.

- MAO coating was successfully applied to AA2024 alloy for the TiO₂-doped Al₂O₃ ceramic coating.
- ii. It was concluded that the addition of TiO_2 particles into the electrolyte resulted in the reduction in number of pores and formation of smaller and more uniform pores in comparison with the Al_2O_3 coating.
- iii. With X-ray Diffraction (XRD) analyses, it was found that both coated samples are mainly composed of Al, α -Al₂O₃ and γ -Al₂O₃. Moreover, presence of rutile and anatase phases characterizing TiO₂ was detected on the surface of composite coating. The micro pores and

micro cracks on the composite coating were filled with ${\rm TiO_2}$ particles in the MAO treatment.

- iv. The EDS results showed that the composite coating consists mainly of aluminum (Al), oxygen (O), titanium (Ti) and silicon (Si) with an atomic concentration of 41.85%, 52.14%, 3.61%, and 2.41%, respectively.
- v. The average coefficient of the Al_2O_3 coating and $TiO_2:Al_2O_3$ composite coating are 0.5 and 0.36, respectively. Adding TiO_2 particle affected the friction coefficient values. The lowest friction coefficient value was obtained with novel TiO_2 doped Al_2O_3 composite coated sample.
- vi. Compared to AA2024 alloy, an improvement of approximately 60% was observed in the COF value with the composite coating containing titanium dioxide (TiO_2) particles formed on surface of AA2024.
- vii. The addition of TiO_2 particles resulted in an increase of the coating compactness and affect wear mechanism of the coating.
- viii. The addition of TiO_2 particles resulted in an increase of the coating thickness, as well as the capacity to carry loads.
- ix. The addition of TiO_2 particles into electrolyte the abrasive wear and detachment of the coating was effectively reduced.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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