Effect of Plasma Enhanced Chemical Vapor Deposition of Tetraethylorthosilicate on the Friction and Wear Loss of Plasma Electrolytic Oxidized Aluminum 6082

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Abstract The surface of 6082 aluminum alloy was coated by plasma electrolytic oxidation (PEO) and then treated by plasma enhanced chemical vapour deposition (PECVD) of tetraethylorthosilicate (TEOS), oxygen and argon. The PEO electrolyte was alkaline and consisted of potassium hydroxide and sodium aluminates. A pulsed DC power supply with the frequency of 18 kHz was utilized to perform PEO and PECVD treatments. In the PEO process, the electrolyte was at boiling temperature. PECVD was carried out at temperature of 400°C. The working pressure of vacuum chamber was 10 mbar. The surfaces were characterised using XRD, optical, AFM and SEM microscopy, EDX analysis, Vickers microhardness test and ball on disc wear test methods. The thickness of PEO coated layers was more than 80 μm . $\alpha\text{-Al}_2\text{O}_3$ was the main oxide compound in the coatings. PECVD treatment resulted in the diffusion of silicon in the surface oxide layer. However, up to 16% silicon was identified in the top surface layers after PECVD treatment, no considerable variation in the thickness of the alumina layer or new layers were observed on the surface of the samples. PEO coating generated very hard surfaces with highly varying coefficient of friction. PECVD treatment reduced the hardness slightly, modified the friction behavior and reduced the wear loss several times.

Keywords Adhesive Wear, Surface Roughness, Hardness, Coating, Wear Resistance, Stick-slip

1. Introduction

Industrial applications are demanding wear resistant light alloys, such as surface treated aluminum alloys, instead of steel. This requires surface hardness of more than 700 HV. Hard anodizing has been experienced as one the methods for improving the surface properties of light alloys. After hard

anodizing the surface hardness and the wear resistance of coatings on aluminum are not as high as hard resisting layers on steel [1]. PEO has offered new developments in aluminum oxidation. Several studies have attempted to produce uniform oxide layers on Al alloys with conventional anodizing or PEO processes in different electrolytes. Nevertheless, researchers have yet to produce a uniform and dense oxide layer free from surface porosities.

Spark discharges in PEO process create discharge channels on the surface of the samples. Melting, oxidation and ejection of the elements in these channels lead to growth of the nano or microcrystalline structure of the coating [2, 3]. This technique is also named micro-arc oxidation. The plasma phenomena in micro arc oxidation (MAO) or Plasma electrolytic oxidation (PEO) give unique effect to the coatings which increase the life of industrial parts with favorable properties such as high hardness and anti-corrosion abilities [4, 5]. Industrial applications of these coatings are widely ranging from automotive to aerospace, textile and oil and gas industry [6].

Aluminum oxide coatings do not show low or smooth friction behavior on the surface of aluminum alloys. Sometimes the high coefficient friction or in fact stick slip friction behavior of these coating may result in localized wear or spallation of oxidized surfaces. It has been found that PEO of Al-Si alloy with 7% silicon content in alkali silicate solution mainly result in the formation of $\alpha\text{-Al}_2\text{O}_3$, $\gamma\text{-Al}_2\text{O}_3$ and SiO_2 and some percentage of mullites ($3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) in the coating. These investigations have shown that different combinations of $\alpha\text{-Al}_2\text{O}_3$ and SiO_2 reduce the friction on the alumina surfaces [7]. It is proposed that it would be beneficial to produce mullites like phases or combinations of $\alpha\text{-Al}_2\text{O}_3$ and SiO_2 on PEO surfaces to reduce the friction coefficient of oxide layer and consequently increase the wear resistance of these surfaces.

It has been observed that oxides deposited from organosilicates compounds such as tetraethylorthosilicate (TEOS) show low friction properties on the surface of light alloys. Therefore, in this paper we have studied the effect of

PECVD of tetraethylorthosilicate (TEOS) for production of low friction compounds on the surface and reduction of the wear loss of PEO coated aluminum alloy 6082.

2. Materials and Methods

Cylindrical samples of 5 mm thickness and 40 mm diameter were cut from aluminum alloy 6082 (0.6% Si, 0.6% Fe, 0.1% Cu, 0.7% Mn, 0.25% Cr, 0.3% Mg, 0.1% Zn, remaining Al). Two treatments of PEO and PECVD applied on the samples. Before PEO coating, aluminum samples were cut and ground by SiC papers to achieve a uniform surface roughness of approximately 6–7 μm . The samples were degreased with acetone and alcohol and immersed in the electrolyte.

The aluminate electrolyte was prepared from mixtures of KOH and NaAlO_2 in distilled water. A 10 KW pulsed DC power supply was utilized for the application of voltages up to 100, 150 and 220 V and max 50 amps (Table 1). The frequency of the power supply was 18 kHz. A 3 liters stainless steel container was used as the cathode. After PEO treatment, the samples were treated by plasma enhanced chemical vapor deposition of tetraethylorthosilicate (TEOS) in a gas mixture argon and oxygen at 400°C (Table 1).

Table 1. The conditions of PEO and PECVD treatment of aluminum alloy 6082

Treatment	PEO	PECVD
voltage	100-150 and 220 V	-350 V
Composition of electrolyte	aqueous solution of 10 g/l NaAlO_2 , 5 g/l KOH	
temperature, °C	Boiling point of the electrolyte	400 °C
Time, min	10, 20, 30, 40, 50, 60, 90 and 120	60
The ratio of gas mixture	-----	Tetraethylorthosilicate (TEOS): O_2 :Ar=1:50:15
Flow rate of TEOS	-----	4 sccm
Flow rate of oxygen	-----	200 sccm
Flow rate of argon	-----	60 sccm
Pulsed DC power	1-2 kW	300-400 W

The different phases of the coatings were investigated with a Philips X-ray diffractometer (XRD) equipped with Cu K α radiation and the scans were performed with 0.02° step size in the 2 θ range of 20–90°. The microstructure, thickness and topography of the surfaces were studied using optical, scanning electron microscopy (SEM) and AFM. EDX analysis helped to identify the elemental composition of the coatings.

For microscopy studies, the samples were mounted with cold setting resin. The mounted samples were ground using successive grades of silicon carbide emery papers from 80 to 1,200 grit. To achieve a scratch-free surface, the ground samples were finally polished using 0.05 μm alumina powder. Kroll's chemical reagent was used to reveal the

microstructure of the samples.

The microhardness of the coating layers was measured in different places by a Vickers microhardness test machine. The cross section profile of the wear tracks was measured by a DEKTAK profilometer. The friction and wear tests were performed in a ball on disk configuration under normal loads of 5 and 30 N at 0.1 m/s speed for maximum 350 m sliding distance. The volume loss of the samples was measured by multiplying the diameter of the wear tracks to the area of the cross section of the wear tracks.

3. Results and Discussions

(a)

(b)

(c)

Figure 1. Graphs show (a) the effect of voltage at constant concentration and time, (b) the effect of NaAlO_2 concentration at constant voltage and time and (c) the effect of time at constant voltage and concentration on the thickness of PEO oxide layers.

The early experiments of PEO coatings showed that at voltages less than 100V the coating is very thin and at voltages higher than 150 V does not increase strongly (Figure 1a). The least effective amount of NaAlO_2 was 10 percent. The higher percentages of NaAlO_2 did not dissolve properly in the aqueous solution and did not increase the thickness of the coating significantly (Figure 1b). After 60 min PEO coating, the approximately linear growth rate of the oxide layer was very low and did not increase considerably (Figure 1c). Therefore the samples treated at 150 V, for 60 min in aqueous solution of 10 g/l NaAlO_2 and 5 g/l KOH were selected for further investigation in this paper.

3.1. Microstructure

The PEO process generally resulted in porous layers on the surface of 6082 aluminum alloy. The PECVD of tetraethylorthosilicate (TEOS) in Argon and Oxygen mixture did not change the appearance and thickness of the surface layers significantly. It has frequently observed that deposition of TEOS by PECVD treatment has resulted in very thin layers on different substrates. However, the thin layers were very scratch resistant [8].

Figure 2 shows two samples treated with successive PEO and PECVD processes. These samples were plasma electrolytic oxidised in an aqueous solution with concentration of 10 g/l NaAlO_2 and 5 g/l KOH. As seen in the Figure 2a, the thickness of the sample coated by PEO at voltage of 100 V and then deposited by PECVD of TEOS was less than 10 μm . Also, some debris exists on the surface which may belong to any top surface layer that separated during metallography of the samples.

Figure 2b shows another sample that PEO coated at 150 V and then deposited by PECVD of TEOS at 400°C. This coating has small porosities and no distinct layers are observed in the sample. The thickness of the oxide compound layer was around 85 μm (Figure 2a). Some porosity can be observed on the cross section of the surface layer. However there is not a big variation in the structure of the fine grains of the oxide compound layer.

(a)

(b)

Figure 2. SEM micrographs of the sample plasma electrolytic oxidised in aqueous solution of 10 g/l NaAlO_2 and 5 g/l KOH for 60 min at (a) 100 V and at (b) 150 V, followed by PECVD of TEOS for 60 min at 400°C for both (a) and (b).

It is proposed that the layer deposited by PECVD of TEOS has been very thin to be observed in the top surface of the PEO coated surface.

The layer deposited from TEOS may have been integrated in the top layers. The incorporation of silicon in the top surface layer was later identified by EDX analysis.

3.2. Phase Identification

XRD of aluminum alloy 6082 (Figure 3a) showed only four distinct peaks in the pattern in which (311) peak was very strong. After PEO treatment, peaks of $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ were identified in the XRD pattern (Figure 3b).

However, small peaks of Aluminum alloy 6082 were also observed in the pattern. It can be attributed to the thick layer of $\alpha\text{-Al}_2\text{O}_3$ that has prevented the enough penetration of X-ray into the aluminum substrate. Consequently the peaks of the oxide layer have appeared stronger in the patterns. Also, as seen in Figure 3b, the peaks of $\alpha\text{-Al}_2\text{O}_3$ are much stronger than γ -alumina. This is strongly related to the higher amount of $\alpha\text{-Al}_2\text{O}_3$ than that of γ -alumina. It has been reported that the γ -alumina phase prevails in the early stage of the PEO process, and the $\gamma \rightarrow \alpha$ phase transition occurs in those layers that are thicker than 30–40 μm [1].

PECVD treatment did not result in any distinctive peaks of SiO_2 (Figure 3c). This is correlated to the very thin layers of SiO_2 or dissolved SiO_2 compound in the top layers of $\alpha\text{-Al}_2\text{O}_3$ layers. Many investigations in the field of SiO_2 deposition from PECVD of TEOS and oxygen have produced a few microns or very thin layers of SiO_2 on different substrates. These thin layers have been identified mostly by FTIR or Raman spectroscopies and have not been shown by XRD analysis [9, 10]. From another hand, the peak positions of aluminosilicate or mullites compounds such as $(3\text{Al}_2\text{O}_3, 2\text{SiO}_2)$ in the XRD patterns have the same position as $\alpha\text{-Al}_2\text{O}_3$ [11, 12].

Figure 3. XRD patterns of (a) untreated Aluminum alloy 6082, (b) Aluminum alloy 6082, PEO treated at 150 V for 60 min in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH, (c) the treatment as (b) and then PECVD treated for 60 min at 400 °C and (d) the same as (c) at incident grazing angle of 9 degree.

EDX spectra of three regions on PECVD treated sample showed on average 16 % silicon in addition to aluminum and oxygen. The higher content of aluminum and oxygen than that of silicon suggested that the amount of α -Al₂O₃ in the coating was in excess of SiO₂. Therefore it may be concluded that the deposition of TEOS by PECVD has resulted in very thin SiO₂ layers or a compound that integrated or mixed with α -Al₂O₃ during the formation process of the surface layers [13, 14]. Grazing angle XRD of PECVD treated sample showed only peaks of α -Al₂O₃ (Figure 3d). As seen in Figure 3d, there was not any peak of aluminum alloy and the peaks of γ -Al₂O₃ did not appear in the pattern. It can be concluded that the top surface layers have been modified with PECVD treatment and any layer of silicon compound has been very thin or dissolved in the top layers.

3.3. Surface Topography and Roughness

Surface morphology of the coatings after 60 min PEO treatment at 100, 150 and 220 V is shown in Figure 4. It can be seen that PEO coating at 100 V (Figure 4a) has resulted in deep and fine gained porosities on the surface that have broken out of the surface. This kind of surface roughness may be correlated to the less violence created in voltage of 100 V in comparison with higher voltages. These pores are actually residual discharge channel during the spark reaction. The irregular-shaped areas around discharge channel are formed due to the rapid cooling effect of the electrolyte [15]. At 150 V, SEM examination on the top surface of the specimen revealed the sink-hole type of pores in the discharge channels which are clear on the surface (Figure 4b).

These porous structures can be useful in keeping the lubricants at the sliding surfaces and less friction coefficient. At 220 V, the surface irregularity has increased and pores of sink-hole type have grown outward and varied to round type protrusions out of the surface. This type of surface roughness did not expect to have low coefficient of friction. After PECVD treatment of the PEO coated sample at 150 V, the surface did not changed strongly and some pores were filled with the compounds deposited from TEOS organometallic compound. This surface topography expected to improve the friction behavior of the material.

To evaluate the average surface roughness of treated surface, untreated aluminum alloy 6082, PEO treated sample at 150V in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH for 60 min and the sample PECVD treated for 60 min at 400°C after PEO treatment were examined by AFM microscopy (Figure 5).

As seen in Figure 5, the untreated material had an average roughness of 6.88 μ m (Figure 5a) and increased to 12.5 μ m by PEO coating (Figure 5b).

This is quite reasonable in PEO process. In this process the microdischarges take place over a very short time interval and quench very quickly that result in rough surface. This is in agreement with SEM micrographs from PEO coated surfaces (Figure 4).

Figure 4. SEM micrographs of surface topography of the samples plasma electrolytic oxidised at (a) 100 V, at (b) 150 V, at (c) 220 V at for 60 min in aqueous solution of 10 g/l NaAlO_2 and 5 g/l KOH and (d) the same as (b) followed by PECVD of TEOS for 60 min at 400°C.

After PECVD process the average roughness decreased to 4.81 µm (Figure 5c) which is less than that of the untreated material. It may be suggested that PECVD treatment had produced some compounds that have diffused into some pores and reduced the average surface roughness [11].

(c)

Figure 5. AFM micrographs of the surface roughness of (a) untreated Aluminum alloy 6082, (b) Aluminum alloy 6082, PEO treated at 150 V for 60 min in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH, (c) the treatment as (b) then PECVD treated for 60 min at 400°C.

3.5. Hardness of the Coatings

The surface hardness of the coatings is shown in Figure 6. The precise measurement of hardness impressions on the cross-section of coating was not possible. As they often end to cracking of the layer [16].

As can be seen in Figure 6, the PEO treatment resulted in very hard surface oxide layers. However the contribution of hard layers to surface hardness depends on the thickness of the layers. At 100 V PEO treatments, the surface oxide layer was very thin and therefore the whole hardness value was low because it had a major contribution of the soft aluminum substrate. At higher voltages the surface oxide layer was thick enough to show a high hardness.

Figure 6. Surface microhardness of the samples treated with PEO coating for 60 min at different voltages in 10 g/l NaAlO₂ and 5 g/l KOH and subsequent PECVD of TEOS for 60 min at 400 °C.

The high hardness value is also related to the existence of hard α -Al₂O₃ phase in the coating. Other investigations have shown that higher ratio of α -alumina to γ -alumina in the coatings on aluminum alloy substrates leads to greater hardness.

As γ -Al₂O₃ phase has a low hardness of around 200 HV, it may be suggested that the main phase of the coatings in our investigations was composed of α -Al₂O₃ [17].

It seems that PECVD treatment after PEO treatment has not varied the hardness value significantly. This is attributed to the very thin oxide layers resulting from PECVD treatment. From another hand, it has been observed that coatings with mullites phase or the coatings with silicon compounds show less hardness than coating without silicon. It has been shown that aluminum-silicon-oxygen compounds have a comparatively lower hardness value than that of pure aluminum oxide compounds [18].

3.4. Friction Behaviour and Wear Loss

The friction behaviour of untreated Al 6082 and the samples treated with PEO and PECVD treatments under 5 and 30 N derived from the ball-on-disc sliding wear tests are shown in Figure 7.

As can be observed in Figure 7a, the friction behaviour of untreated aluminium 6082 was quite varying. It seems that normal load of 5N has not been enough to deform the oxide asperities and the steel slider has adhered intermittently with the surface that resulted in high varying friction behaviour.

However the same behaviour was observed under 30 N up to 30 m distance (Figure 7b). Afterwards the friction variations showed a *typical stick-slip behavior* that was highly repeatable. This behavior is strongly attributed to the repeated adhesion and separation of the steel slider with surface asperities of untreated aluminum alloy 6082 under normal load of 30 N.

PEO treatment resulted also in high variation of friction behavior (Figure 7c). Under normal load of 5 N, friction behavior was highly varying. The varying intervals were shorter than that of untreated sample and did not repeat regularly. The friction coefficient value under normal load of 30 N was high but the variations were less than that of 5 N load. The curve of 30 N load shows that the friction coefficients were high at the beginning, then after some fluctuation gradually decreased and remained constant with distance similar to that of 5 N loads.

The trend of the initial 'running in' stage in sliding wear test is due to the surface structure, roughness and adhesion between the pair of sliding components.

High surface roughness and granular and irregular coatings on the surface create a resistance against sliding and cause an increase in the coefficient of friction.

As can be seen in Figure 7, the mean coefficient of friction is at least 0.2 under 5 N load and increases to approximately 0.45 under normal load of 30 N. This is mainly due to the higher normal load of 30 N if compared with that of 5 N.

Figure 7. Friction behavior of untreated aluminum alloy 6082 under normal loads of (a) 5 N, (b) 30 N, (c) PEO coated surface at 150 V for 60 min in aqueous solution of 10 g/l NaAlO_2 and 5 g/l KOH under normal load of 5 and 30 N and (d) PECVD treated surface by TEOS for 60 min at 400°C after PEO treatment under normal load of 5 and 30 N.

In comparison with untreated surface, the friction behavior of PEO coated surface may be related to the weaker adherent junctions or less adhesion between the slider and PEO treated surface than those between slider and untreated material.

PECVD treatment after PEO reduced the variation of friction coefficient significantly (Figure 7d). Under both 5 and 30 N loads the variation of friction coefficient reduced strongly. The average values of 0.25 and 0.35 can be estimated for the coefficients of friction under 5 and 30 N respectively. These are less than those for untreated and PEO treated surfaces. It may be proposed that PECVD of TEOS has reduced the adhesion between the slider and PECVD treated surface more than that of PEO coated surfaces [19]. The smoother friction behavior of this treatment resulted in less wear loss than other treatments.

The wear loss of the samples was examined under ball-on-disc configuration. The profiles of cross section of the wear scars are shown in Figure 8. These profiles produced using a DEKTAK surface profilometer which moved in the direction of the radius of circular wear tracks and passed the worn surface area. As can be seen in the Figure 8, the depth of the wear track on untreated sample was much larger than that of other samples.

(c)

Figure 8. The profiles show the cross section of the wear tracks developed under normal load of 30 N on the surface of (a) untreated aluminum alloy 6082, (b) PEO coated sample at 150V for 60 min in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH and (c) the sample PECVD treated by TEOS for 60 min at 400°C after PEO treatment.

The low depth of wear track in PEO and PECVD treated surfaces is connected to a structural change in the coatings [20]. These coatings were strongly more wear resistant than that of untreated sample. The reason of this was due to the high content of α -Al₂O₃ phase with high density and hardness on treated surfaces. However it seems that PEO treated wear track has worn in some parts deeper than other parts. While the PECVD treated surface had the same resistance to wear all over the wear track. The reaction of the samples against sliding was evaluated and represented in Figure 9.

Dry ball-on-disc wear tests were performed on Aluminum-alloy 6082 substrate, PEO treated sample and PECVD treated samples, under a normal load of 30 N, 0.1 m/s sliding speed for sliding distances up to 350 m and humidity of 50% RH against SAE 52100 bearing steel.

The lower wear loss of PEO sample is quite reasonable and is due to the α -Al₂O₃ oxide layers that are hard and wear resistant [21]. In fact the low shear force that involved during sliding resulted from a hard surface cutting a soft counterpart [22].

The much lower wear loss of PECVD treated surface might be due to some reasons. It may be stated that some of the discharge channels got closed while some of them remained open during the PECVD treatment of PEO coated surface and provided a smoother or less adhering surface.

The surface of the wear track is more flat and the sink-hole type structure more or less flattened out and merged into each other forming a more uniform surface (Figure 4d) [23]. The other reason may be the formation of a solid lubricant due to the introduction of silicon compounds to the surface.

In our results, the PECVD treated surface gives a smoother friction behavior if compared to PEO treated surface. This indicates that hard and soft rubbing pair generated lower friction and higher wear resistance on the surface.

Figure 9. The bar graph shows the wear volume loss under normal load of 30 N for (a) untreated aluminum alloy 6082, (b) PEO treated sample at 150V for 60 min in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH and (c) the sample PECVD treated by TEOS for 60 min at 400°C after PEO treatment.

4. Conclusions

Aluminum alloy 6082 was plasma electrolytic oxidized (PEO) in NaAlO₂: KOH aqueous solution at different time and voltages.

According to an elementary investigation, it was observed that the reliable coating thicknesses did not increase significantly by voltages more than 150 V for 60 min in aqueous solution of 10 g/l NaAlO₂ and 5 g/l KOH. Therefore friction and wear tests of the samples were compared at these conditions.

To achieve new surfaces, the PEO treated samples were deposited by Plasma Enhanced Chemical Vapor Deposition (PECVD) of TEOS, Ar and O₂ for 60 min at 400°C.

The microhardness of PEO treated surface was much higher than that of untreated aluminum alloy 6082. While the hardness of PECVD treated surface was slightly less than that of PEO coated surface.

Surface topography of PEO coated surfaces at 150 V was relatively smoother than those treated at other voltages. However PECVD treatment produced a relatively more uniform and smoother surface.

Aluminum alloy 6082 showed a typical stick-slip behavior. The friction behavior of PEO coated surface was highly varying and increased if compared to that of untreated material. PECVD treatment decreased the variation and average value of friction coefficient.

The wear loss of PEO treated surface was 40 times less than that of untreated aluminum alloy 6082. PECVD treatment reduced the wear loss of PEO treated surface several times.

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