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Alumina Formation on Pure Aluminum via PEO Process in Electrolytes with Different Silicate-Aluminate Ratios

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Abstract: This study systematically investigates the effect of electrolytes with varying silicate-aluminate ratios on the microstructure, phase composition, coating thickness, and surface roughness of aluminum coatings synthesized via the PEO process on pure Al. The coatings primarily consist of mullite ($Al_6Si_2O_{13}$), ɣ-Al_2O_3 and α-Al_2O_3 phases. Increasing the aluminate content promoted α-Al_2O_3 formation and a higher number of smaller discharge channels, while higher silicate content favored mullite formation. Coating thickness increased from approximately 32 µm to 50 µm with higher silicate concentrations, whereas surface roughness remained relatively constant (~5 µm) across different electrolyte compositions.

Keywords: Aluminum, silicate, aluminate, plasma electrolytic oxidation, micro arc oxidation.

Farklı Silikat-Alüminat Oranlarına Sahip Elektrolitlerde PEO Prosesi ile Saf Alüminyum Üzerinde Alümina Oluşumu

Özet: Bu çalışmada farklı silikat-alüminat oranlarına sahip elektrolitlerin saf Al üzerine PEO prosesi ile elde edilen kaplamaların mikro yapısı, faz bileşimi, kaplama kalınlığı ve yüzey pürüzlülüğü üzerindeki etkisini sistematik olarak incelenmiştir. Kaplamalar ağırlıklı olarak mullit ($Al_6Si_2O_{13}$), ɣ-Al_2O_3 ve α-Al_2O_3 fazlarından oluşmaktadır. Elektrolitteki alüminat oranının artması α-Al_2O_3 oluşumunu ve daha küçük, daha fazla deşarj kanalını teşvik ederken, silikat oranının artması müllit oluşumunu desteklemiştir. Kaplama kalınlığı, yüksek silikat konsantrasyonlarında yaklaşık 32 µm'den 50 µm'ye yükselirken, yüzey pürüzlülüğü farklı elektrolit konsantrasyonlarında nispeten sabit kalmıştır (~5 µm).

Anahtar Kelimeler: Alüminyum, silikat, alüminat, plazma elektrolitik oksidasyon, mikro ark oksidasyon.

Article

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1.Introduction

Aluminum and its alloys are widely used across various industrial sectors including automotive, aerospace, textile, construction, electronics, biomedical and metal forming industries owing to their favorable properties such as high specific strength, low density and excellent formability (Hirsch, 2014; Zhang et al., 2018). However, their applications are often limited by relatively poor surface characteristics, including low hardness, limited wear resistance, and inadequate corrosion resistance under certain environmental conditions (Berlanga-Labari et al., 2020). To overcome these drawbacks, two general approaches have been developed to enhance the performance of aluminum-based materials. The first focuses on improving bulk properties through alloying, heat treatment, and/or thermomechanical processing, while the second targets surface modification (Li et al., 2023). Surface modification is generally more cost-effective and has attracted increasing research interest in recent years (Jiang & Wang, 2010). Conventional surface modification methods such as sol-gel deposition, vapor deposition and thermal oxidation have been successfully applied; however, they often alter the substrate microstructure or involve environmentally harmful electrolytes (Jiang & Wang, 2010; Mora-Sanchez et al., 2021).

Anodic oxidation is another widely used surface modification technique for aluminum alloys. Nevertheless, its broader application is constrained by slow coating growth rates, limited thickness, inadequate wear resistance, and the environmentally hazardous nature of the acidic electrolytes used in the process (Jiang & Wang, 2010; Mora-Sanchez et al., 2021). To address these challenges, Plasma Electrolytic Oxidation (PEO), also known as Micro-Arc Oxidation (MAO), has emerged as a promising alternative to conventional anodizing. PEO enables the formation of hard, adherent and corrosion-resistant ceramic-like oxide coatings on valve metals such as Al, Mg, Ti, and their alloys (Cengiz, 2020; Clyne & Troughton, 2019; Cosan et al., 2022; Gunduz et al., 2017). The process is characterized by the generation of localized micro-discharges once the dielectric breakdown potential of the oxide film is reached, initiating high-temperature plasma reactions that produce dense and crystalline oxide layers (Zhu et al., n.d.). Compared with conventional anodizing, PEO yields significantly thicker coatings with higher phase stability, superior tribological and corrosion performance, and reduced environmental impact. The characteristics and performance of PEO coatings are strongly influenced by several process parameters, including electrical conditions (voltage, current, frequency, and duty cycle), electrolyte composition and concentration, substrate characteristics, process temperature, and treatment duration (Clyne & Troughton, 2019; Fernández-López et al., 2024; Gamba et al., 2025; Y. Wang et al., 2024). Among these parameters, electrolyte chemistry plays a particularly critical role in determining coating growth kinetics, morphology, microstructure and phase composition. PEO treatments on aluminum and its alloys are typically carried out in alkaline electrolytes with a pH range of approximately 7-12. Within this range, the presence of sufficient OH^- ions ensures adequate electrical conductivity, which is essential for sustaining the plasma discharges responsible for coating formation. The electrical conductivity of the electrolyte directly affects the breakdown voltage and growth rate, higher conductivity generally leads to lower breakdown potential and faster coating development (Fernández-López et al., 2024; Gamba et al., 2025). Common electrolytes for PEO include silicate-, phosphate-, borate-, and aluminate-based systems (G. Lv et al., 2006; S. Wang et al., 2020). In addition, particulate-incorporated electrolytes containing oxide- or non-oxide-based particles have also been employed (Gamba et al., 2025). Silicate-based electrolytes are widely used due to their stability and ability to produce relatively thick coatings with good corrosion resistance. However, in alloys such as Al-Si, they can promote excessive formation of silicon oxides, mullite and aluminosilicate phases, which reduce the proportion of hard aluminum oxides and impair tribological performance (Fernández-López et al., 2024). In contrast, aluminate-based electrolytes favor the formation of α-Al_2O_3, resulting in coatings with improved hardness, wear resistance and corrosion performance, particularly in cast Al-Si alloys where aluminum oxides help balance silicon oxide formation (Fernández-López et al., 2024; Gamba et al., 2025; XIE et al., 2017). Recent studies have shown that using a mixture of silicate and aluminate electrolytes can affect coating thickness, surface structure, and phase stability. PEO treatment of Al-Cu-Li alloys in aluminate and silicate electrolytes demonstrates that electrolyte composition both phase transformation behavior and coating growth kinetics (Arboleda & Dela Peña, 2025). The aluminate-based electrolyte produces a smoother coating/substrate interface, however, its coating thickness (92 μm) is significantly lower than that of the silicate-based coating (154 μm). XRD analyses performed on the coatings reveal that the aluminate-based coatings contain a higher amount of α-Al_2O_3 compared to the silicate-based ones. This difference is attributed to the suppression effect of silicon on the α-Al_2O_3 phase transformation (Arboleda & Dela Peña, 2025). In the plasma electrolytic oxidation (PEO) of pure titanium, electrolyte chemistry plays a decisive role in coating growth and microstructural evolution (He et al., 2025). Studies on mixed aluminate-silicate-phosphate electrolytes show that variations in electrolyte concentration and the aluminate-to-silicate ratio significantly influence coating morphology and phase formation. Lower electrolyte concentration (C2) increases the proportion of aluminate and silicate species, resulting in thicker but highly porous coatings enriched in Al_2TiO_5. In contrast, higher electrolyte concentration (C1) promotes the development of more compact oxide layers with reduced Al_2TiO_5 content and a higher fraction of TiO_2, thereby enhancing corrosion resistance. Cross-sectional measurements confirm this trend: C2 electrolytes produce thicker coatings (52.61 μm and 32.60 μm for Ti-C2-V1 and Ti-C2-V2, respectively), whereas C1 electrolytes yield comparatively thinner layers (45.39 μm and 23.80 μm for Ti-C1-V1 and Ti-C1-V2). Despite the lower thickness, the C1 electrolyte provides better corrosion resistance, as demonstrated by the Ti-C1-V1 sample, which exhibits a more noble corrosion potential (E_{corr}=−0.170 V) and a significantly lower corrosion current density (I_{corr}=1.730 x 10^{3-} $mA.cm^{2-}$) (He et al., 2025). However, there are still few studies that systematically examine how changing the ratio of silicate to aluminate affects the structure, surface features and composition of PEO coatings. Understanding this relationship is important for designing coatings with specific mechanical strength or corrosion resistance.

In this study, pure aluminum substrates were coated using PEO in electrolytes with controlled silicate to aluminate ratios. The resulting coatings were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and surface profilometry to evaluate phase evolution, microstructure, coating thickness and surface roughness. The study aims to provide fundamental insights into how electrolyte composition governs plasma discharge behavior and phase transformations during PEO coating growth.

2. Experimental

2.1. Preparation of Substrates

Commercially pure aluminum plates with dimensions of 10 × 15 × 20 mm^3 were used as the substrate in the present study. All samples were polished using emery paper to achieve a uniform surface roughness (R_a) of 0.2 ± 0.1 μm. Surface roughness measurements were performed using a Veeco Dektak 8 profilometer. Prior to PEO coating, the aluminum

samples were ultrasonically cleaned in ethanol for 5 minutes and subsequently dried with warm air.

2.2. PEO Coating Process

The PEO coatings were produced using a homemade PEO system. Aqueous electrolytes were prepared using silicate (Na_2SiO_3) and aluminate ($NaAlO_3$) components, with compositions listed in Table 1. The electrolytes were labeled A1 to A11, with each prepared by dissolving a constant amount of 2 g/L KOH (0.036 mol/L) and a total of 0.1 mol/L silicate and aluminate. The PEO treatment was conducted for 60 minutes at a maintained electrolyte temperature of 25 ± 2 °C using a cooling system. A constant current density of 0.25 A/cm² was applied, while the voltage increased gradually over time. Samples were coded according to the electrolyte in which they were treated.

Table 1. The chemical compositions of the electrolytes and the corresponding sample codes.
Tablo 1. Elektrolitlerin kimyasal kompozisyonları ve numune kodları.

Sample Code	Electrolyte (mol/l)	
	Na_2SiO_3	$NaAlO_3$
A1	0.01	-
A2	0.09	0.01
A3	0.08	0.02
A4	0.07	0.03
A5	0.06	0.04
A6	0.05	0.05
A7	0.04	0.06
A8	0.03	0.07
A9	0.02	0.08
A10	0.01	0.09
A11	-	0.10

2.3. Characterization of the Coatings

The thickness of the PEO coatings was measured using an eddy current instrument (Fischer Dualscope MP20). For each sample, thickness values were recorded at ten different locations on the surface, and the average value was calculated. The surface roughness of the coatings was determined using a Veeco Dektak 8 profilometer, with measurements taken at 10 randomly selected positions to obtain the average R_a value.

Phase analysis of the coatings was carried out by a Bruker Advance D8 Diffractometer (Cu K_α radiation) in Bragg-Brentano geometry, over a 2θ range of 10º-90º, with scanning rate of 2 º/min and a step size of 0.02º at operating conditions of 40 kV and 40 mA. The surface morphology and chemical composition of the coatings were examined using a Philips XL30 FEG scanning electron microscope (SEM) equipped with an AMETEK EDAX energy-dispersive X-ray spectroscopy (EDS) detector. Surface observations were performed in secondary electron (SE) mode at various magnifications. For cross-sectional analysis, the coated samples were sectioned and mounted in epoxy resin. The cross sections were ground using SiC papers with grit sizes ranging from 180 to 1200, followed by polishing with a diamond suspension (1-3 µm particle size) to obtain a mirror-like finish suitable for microstructural examination.

3. Results and Discussion

3.1. XRD Analysis of the Coating

The XRD patterns obtained from the surface of the PEO coatings are presented in Figure 1. The surface XRD results show that the coatings consist primarily of mullite ($Al_6Si_2O_{13}$), ɣ-Al_2O_3 and α-Al_2O_3 phases. With increasing sodium aluminate concentration in the electrolyte, the relative intensity of the α-Al_2O_3 peaks increases, while that of mullite decreases. This can be attributed to the formation of mullite in coatings produced from sodium silicate electrolytes during the PEO process. The SiO_3^{2-} component in the sodium silicate electrolyte is easily incorporated into the coatings through chemical reactions, contributing to an increase in coating thickness. While the α-Al_2O_3 phase is thermodynamically stable at all temperature including room temperature, the ɣ-Al_2O_3 is metastable phase. However, ɣ-Al_2O_3 can form easily and transform into α-Al_2O_3 at temperatures between 800 °C and 1200 °C. Previous studies have reported that an increase in coating thickness facilitates the formation of α-Al_2O_3 and the amount of this phase increases accordingly. This phenomenon has been explained by the fact that strong discharge events can heat the coating to the temperatures required for the ɣ→α phase transformation (Rodriguez et al., 2023).

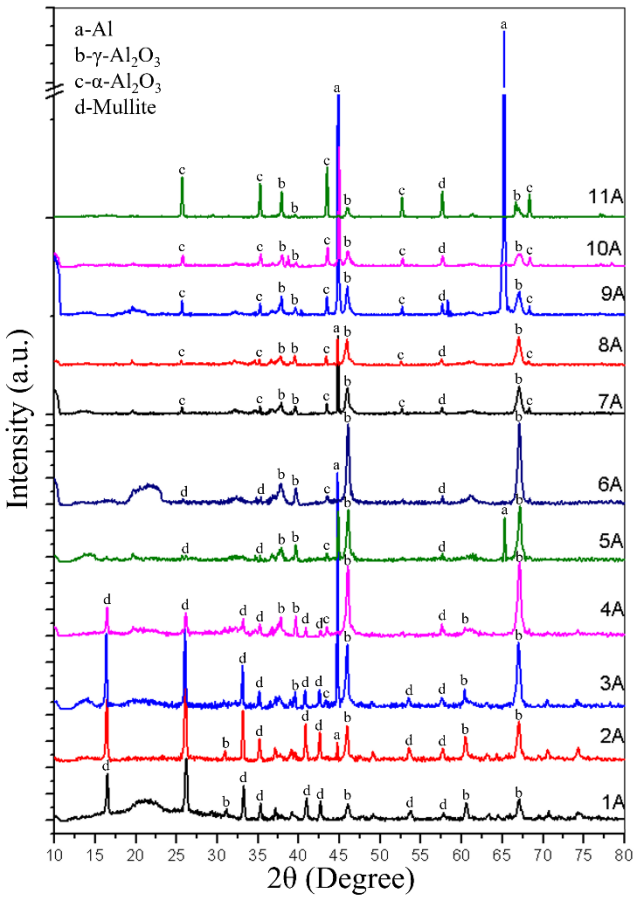

Figure 1. XRD patterns obtained from the surface of the PEO-coated samples.
Şekil 1. PEO kaplanmış numunelerin yüzeyinden elde edilen XRD paternleri.

3.2. Surface and Cross-Sectional SEM Analysis of the Coating

Figure 2 shows the surface morphologies of the pure Al samples coated via PEO process in different electrolyte concentrations for 60 minutes. The surface SEM images clearly show the presence of discharge channels and the number of these channels tends to increase as the electrolyte composition changes from A1 to A11. On the other hand, the mean sizes of the discharge channels decrease with the change in the solution in the same order. The surfaces of the A1-A4 samples exhibit glassy phases and bowl-like features, typical of PEO coatings (Figure 2(a-d)). With increasing sodium aluminate concentration, these glassy phases progressively transform into a sponge-like morphology, as observed in the

A5-A8 samples (Figure 2(e-h)). Open pores are evident on the surface of the A9 sample and the openings of the discharge channels appear narrower compared with those of the previously discussed samples. Additionally, the regions surrounding the discharge channels appear smoother than the other areas on the surface. The surfaces of the A10 and A11 samples (Figure 2(j, k)) are representative examples illustrating the so-called “pancake” structure. In this structure, electrical discharges occur at the center of the pancake, causing molten metal to be ejected. The formation of this structure results in relatively smoother surfaces compared with the earlier samples. Cracks were observed on the surfaces of all samples. The cracks on the A6, A7 and A8 samples are particularly larger and more pronounced. Furthermore, localized spalling was detected on the surface of the sample coated in the A6 electrolyte. Particulate formations were also observed on the oxide surfaces, except for the A1 sample.

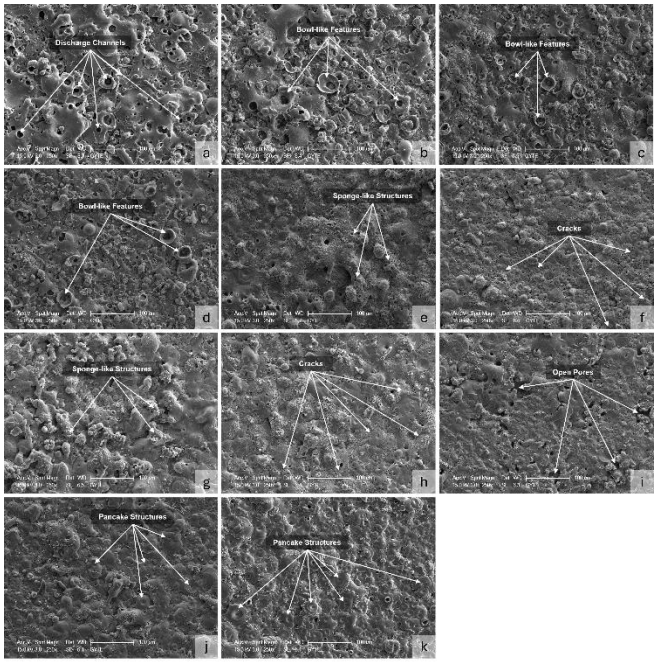

Figure 2. Surface SEM micrographs of the samples coated in different electrolytes, a) A1, b) A2, c) A3, d) A4, e) A5, f) A6, g) A7, h) A8, i) A9, j) A10, k) A11.

Şekil 2. Farklı elektrolitlerle kaplanmış numunelerin yüzey SEM mikrografları, a) A1, b) A2, c) A3, d) A4, e) A5, f) A6, g) A7, h) A8, i) A9, j) A10, k) A11.

Figure 3 shows cross-sectional SEM images of the PEO-coated samples. No spallation was observed, indicating good adhesion of the oxide coatings to the substrates. Cross-sectional SEM images further showed compact, band-like structures (Figures 3(g, h)). Based on the XRD results and comparisons with similar findings reported in the literature (Cengiz, 2020; Cosan et al., 2022; Gencer & Gulec, 2012; Oter et al., 2015), these band-like and compact structures can be reliably attributed to α-Al_2O_3. During polishing with alumina, the relatively soft γ-Al_2O_3 undergoes selective wear, which further highlights the harder α-Al_2O_3 bands. As the silicate content in the electrolyte increased, the α-Al_2O_3 phase initially decreased and eventually disappeared (Figures 3(a-e)). In contrast, increasing the aluminate content led to a broadening of the α-Al_2O_3 bands (Figures 3(g-k)). Furthermore, the amount of the mullite phase associated with the glassy morphology increased with increasing silicate content. Previous studies support these observations. Xie et al. investigated the effect of $NaAlO_2$ concentration on the PEO behavior of cast Al-Si (A356) alloys. Three electrolyte solutions with different $NaAlO_2$ concentrations (2 $g.L^{1-}$, 16 $g.L^{1-}$, and 24 $g.L^{1-}$), each supplemented with a constant amount of KOH (1 $g.L^{1-}$), were compared to a conventional silicate-based electrolyte (8 $g.L^{1-}$ Na_2SiO_3 + 1 $g.L^{1-}$ KOH). Their results showed that the electrolyte containing 24 $g.L^{1-}$ $NaAlO_2$ produced a single-layer coating with superior wear and corrosion resistance, attributed to the substantial presence of α-Al_2O_3 (XIE et al., 2017). Similarly, Wang et al. fabricated MAO coatings on LY12 alloy using electrolytes with varying Na_2SiO_3 and KOH concentrations. They observed that the intensity of the α-Al_2O_3 peak gradually decreased with increasing Na_2SiO_3 concentration, suggesting that higher Na_2SiO_3 inhibits the γ→α transformation. This effect is attributed to the lower voltages achieved with higher Na_2SiO_3 content, which reduces the temperatures and pressures during the PEO process, preventing γ-Al_2O_3 from transforming into the more stable α-Al_2O_3 phase (C. Wang et al., 2020).

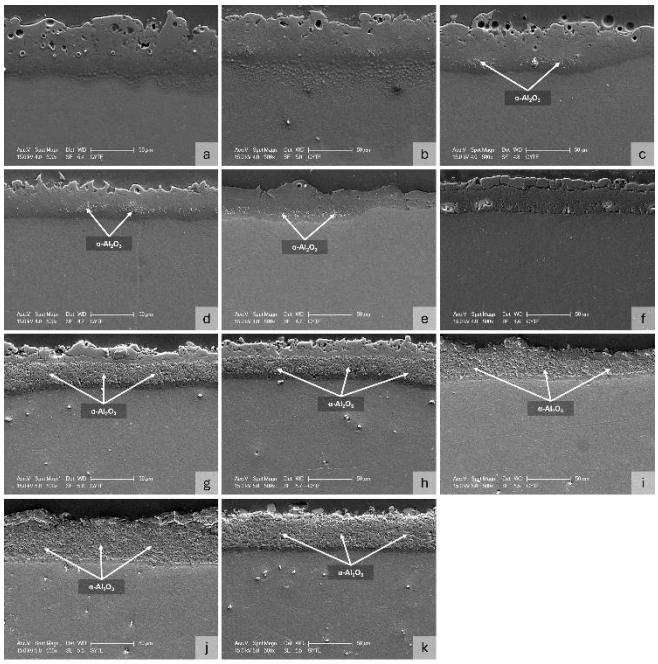

Figure 3. Cross-sectional SEM micrographs of coatings, a) A1, b) A2, c) A3, d) A4, e) A5, f) A6, g) A7, h) A8, i) A9, j) A10, k) A11.

Şekil 3. Kaplamaların kesit SEM mikrografları, a) A1, b) A2, c) A3, d) A4, e) A5, f) A6, g) A7, h) A8, i) A9, j) A10, k) A11.

3.3. Coating Thickness and Surface Roughness

The coating thickness and surface roughness of the PEO-coated samples are presented in Figure 4. The surface roughness (R_a) was approximately 5 µm, with some scatter, and was not significantly affected by the electrolyte concentration. In contrast, the coating thickness decreased from A1 (average ~50 µm) to A11 (average ~32 µm), showing considerable variability. Increasing the sodium silicate concentration in the electrolyte enhanced the coating thickness and accelerated the deposition rate. Lv et al. investigated the effect of electrolyte composition on the coating thickness of ceramic layers formed on 7075 aluminum alloy via PEO using aluminate-, silicate- and phosphate-based electrolytes. Consistent with these findings, their results showed that the average coating thickness varied depending on the electrolyte, with the silicate-based electrolyte producing the thickest coating. This difference in thickness was attributed to the amount of molten alumina expelled from the discharge channels during the PEO process. The study further emphasized that thicker coatings provide a more effective barrier against corrosive ions, highlighting the importance of coating thickness in enhancing the protective performance of PEO layers (X. Lv et al., 2019).

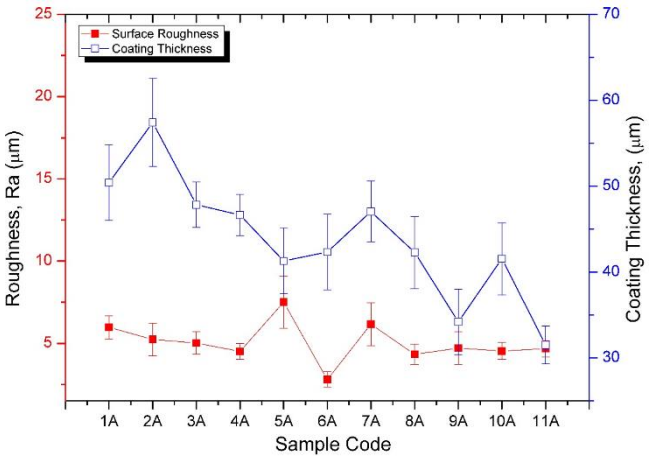

Figure 4. The change in the coating thickness and the surface roughness with different electrolyte content.

Şekil 4. Farklı elektrolit içeriğine bağlı olarak kaplama kalınlığının ve yüzey pürüzlülüğünün değişimi.

4. Conclusions

The present study investigated the influence of electrolytes with varying silicate-aluminate ratios on the microstructure, phase composition, thickness, and surface roughness of PEO coatings on aluminum substrates. The main conclusions can be summarized as follows:

1. All coatings formed in the different electrolytes consisted primarily of mullite ($Al_6Si_2O_{13}$), ɣ-Al_2O_3 and α-Al_2O_3 phases.

2. Increasing the aluminate concentration in the electrolyte promoted the formation of α-Al_2O_3 and led to a higher number of discharge channels with smaller sizes, while increasing silicate content favored the formation of mullite with glassy morphology.

3. Coating thickness decreased from ~50 μm to ~32 μm as the electrolyte composition changed from silicate-rich to aluminate-rich, whereas surface roughness (R_a= ~5 μm) was not significantly affected by electrolyte composition.

4. Higher sodium silicate concentrations enhanced coating thickness and accelerated deposition rates.

5. Declaration of Competing Interest

The authors declare that they have no conflicts of interest. This work is original, unpublished, and not under consideration for publication elsewhere.

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