National University of Science and Technology "POLITEHNICA" Bucharest
University Center of Pitesti
Faculty of Mechanics and Technology

SCIENTIFIC BULLETIN
AUTOMOTIVE series
year XXXI, no. 35

The Influence of PEO Treatment Duration on the Wear Behavior of Ceramic Coatings on Aluminum Alloys

Maria-Magdalena DICU*, **Daniel-Constantin ANGHEL**
National University of Science and Technology POLITEHNICA Bucharest, Pitesti University Centre
*Corresponding author e-mail: maria.dicu@upb.ro

Article history
Received 01.05.2025
Accepted 20.06.2025

DOI https://doi.org/10.26825/bup.ar.2025.002

Abstract. This paper presents the development and characterization of ceramic coatings obtained by Plasma Electrolytic Oxidation (PEO) on aluminum alloys. The electrolyte used is a mixture of sodium metasilicate (Na_2SiO_3) and sodium hydroxide (NaOH). The resulting surfaces were characterized using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), and tribological testing. Additionally, correlations between structure and performance were discussed.
Keywords: PEO, coatings, surface analysis, tribological testing

INTRODUCTION

In recent years, Plasma Electrolytic Oxidation (PEO) technology has attracted significant attention due to its ability to produce hard, adherent, and corrosion- and wear-resistant ceramic coatings on light alloys such as those based on aluminum and magnesium.

PEO is a complex electrochemical process that combines features of conventional anodizing with local electrical discharges occurring at high voltages, which promote the formation of thick and porous oxide layers. The composition of these layers varies depending on the processing conditions. During the process, several phenomena can be observed: oxide layer formation, gas emissions, and dielectric breakdowns, which in turn trigger micro-discharges on the surface of the substrate. These micro-discharges are accompanied by plasma chemical reactions and the emission of visible light, resembling a spark [1].

The micro-discharges also allow the incorporation of electrolyte species into the oxide layer being formed, thus supplying the oxygen required for oxide growth. Moreover, micro-discharges play a key role in producing liquid metal by partially melting the treated material [2].

PEO-synthesized coatings are used in various applications such as anticorrosion, antifriction, and photoactive layers [3–5].

Aluminum alloys offer an excellent combination of properties such as low density and high corrosion resistance, making them widely preferred materials in the automotive, aerospace, and electronics industries, as well as in nuclear engineering, mining, oil and gas production, construction, and other modern engineering fields [6].

Among the numerous technological parameters that influence the final properties of PEO coatings, the treatment duration is essential, as it determines the layer thickness, surface morphology, and the composition of the developed ceramic phases. While many studies have addressed the influence of electrolyte composition or applied voltage, the effect of oxidation time on the tribological performance of PEO coatings remains insufficiently explored.

In this context, the present study aims to investigate the wear behavior of PEO coatings obtained on an aluminum alloy by varying the treatment duration (2, 4, and 6 minutes), while keeping the processing voltage constant at 350 V. The samples were characterized using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) to highlight morphological and compositional changes in the coatings. Additionally, wear resistance was evaluated through cyclic tribological tests, with surface roughness (Ra) being recorded at different stress intervals (initially, after 500, 1000, and 2000 cycles).

The results contribute to a better understanding of how oxidation time influences the tribological performance of PEO coatings and provide practical guidelines for optimizing this treatment method in engineering applications involving friction and wear.

MATERIALS AND METHODS

Materials used

In this study, a high-strength aluminum alloy, AA2024-T351, was used. This alloy exhibits the following properties: a density of 2.78 g/cm³, ultimate tensile strength of 469 MPa, fatigue strength of 138 MPa, Young's modulus of 73 GPa, and a melting onset at approximately 500 °C.

The chemical composition of the 2024 aluminum alloy is presented in Table 1:

Table 1. The chemical composition of the alloy

The chemical composition of the 2024 aluminum alloy (%)								
Al	**Cu**	**Mg**	**Mn**	**Fe**	**Zn**	**Si**	**Ti**	**Zr**
base	4.4	1.5	0.6	0.14	0.12	0.08	0.05	0.02

The 2024 aluminum alloy is known for its high strength, as copper, magnesium, and manganese significantly increase the strength of aluminum alloys. It exhibits excellent machinability, good workability, high strength, and can be fabricated to resist corrosion, making it an optimal choice for applications in aircraft and vehicle manufacturing.

Before starting the treatments, the samples were polished using SiC abrasive paper (#800 – #1200), degreased, and cleaned in an ultrasonic bath with ethyl alcohol and distilled water, successively.

PEO Process

The equipment used for Plasma Electrolytic Oxidation (PEO) is similar to that used for conventional anodizing, but more complex—particularly due to the potential need for higher power and pulse control.

The PEO installation used for the treatments was built at the University of Pitești. It consists of an electrolytic cell made of stainless steel with a stirrer and a pulsed DC bipolar power supply. The aluminum alloy sample was used as the anode, while the stainless-steel cell served as the cathode.

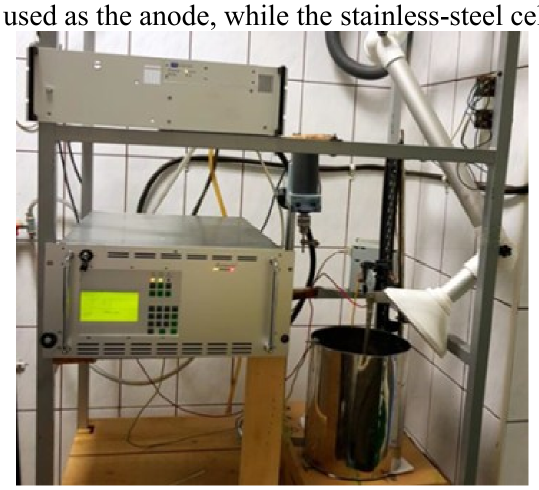

Figure 1. The PEO installation

In PEO treatments, alkaline electrolytes are the most commonly used, as they are more environmentally friendly than the acidic electrolytes employed in conventional anodizing. The most widely used PEO electrolytes are silicates, phosphates, tungstates, and aluminates, known as inorganic polymers [7–9].

For this study, an aqueous solution consisting of 0.2 mol/L of sodium metasilicate (Na_2SiO_3) and 0.2 mol/L of sodium hydroxide (NaOH) dissolved in distilled water was used.

PEO coatings were performed at a voltage of 350 V for treatment durations of 2, 4, and 6 minutes. After each treatment, the samples were rinsed with distilled water and dried at room temperature.

Microstructural and Compositional Characterization

SEM – Scanning Electron Microscopy

The surface morphology of the treated samples was observed using a scanning electron microscope (SEM, Hitachi SU5000). The SEM was operated at an accelerating voltage of 25 kV.

EDX – Energy Dispersive X-ray Spectroscopy

The elemental composition was analyzed using an energy dispersive X-ray spectrometer (EDX) integrated into the SEM. EDX analysis was conducted at an acceleration voltage of 20 kV.

2.4. Tribological Testing

Test Type Used

To evaluate the wear behavior of the coatings obtained through plasma electrolytic oxidation (PEO), tribological tests were carried out using a plane-on-cylinder configuration on a custom-designed laboratory device.

This configuration involves direct contact between a flat surface (the PEO-treated sample) and a rotating cylindrical surface, generating controlled dry friction conditions.

The cylindrical surface was fabricated by 3D printing and then covered with P600 grit abrasive paper, in order to simulate a uniform and reproducible abrasive interaction.

The samples were held in fixed contact with the rotating cylinder under a constant vertical load.

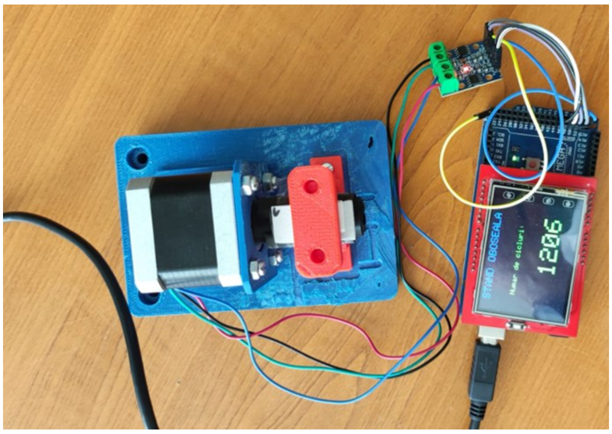

Figure 2. The wear testing rig used for the cyclic wear tests

Loading Parameters

The tribological tests were conducted under identical conditions for all three samples, with the following specifications:

- Configuration: plane-on-cylinder, dry friction;
- Cylinder material: 3D-printed (PLA), covered with P600 grit abrasive paper;
- Applied normal force: constant, 0.3 N;
- Relative motion: rotation of the cylinder under load;
- Number of loading cycles: 500, 1000, and 2000 cycles;
- Ambient conditions: temperature 23 ± 1°C, relative humidity 50 ± 5%.

This method allowed for a comparative evaluation of the wear behavior of the ceramic layers obtained through PEO with treatment durations of 2, 4, and 6 minutes, under strictly controlled conditions.

Roughness (Ra) Measurement Methodology

The surface topography changes resulting from the wear test were evaluated by measuring the arithmetic roughness Ra [µm], using a contact profilometer (Mitutoyo SJ-210). Measurements were carried out at four distinct points for each sample:

- Initial Ra: before testing;
- Ra 500c: after 500 cycles;
- Ra 1000c: after 1000 cycles;
- Ra 2000c: after 2000 cycles.

Figure 3. Surface roughness measurement of the sample

For each measurement, three different areas of the worn surface were selected, and the average value was considered representative. The evaluation length was 4 mm, with a measurement step of 0.25 mm, in accordance with ISO 4287. A Gaussian filter was applied to eliminate high-frequency noise, ensuring high accuracy of the results.

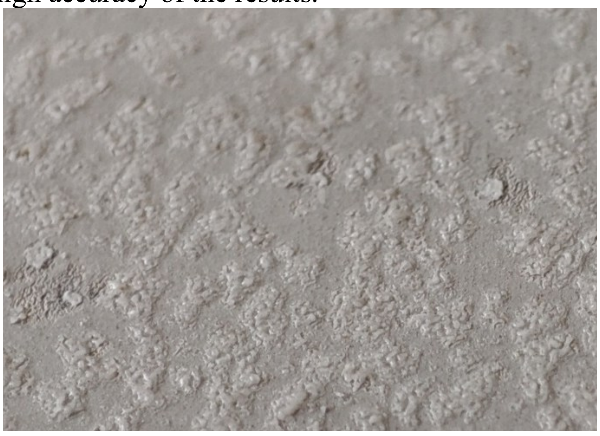

Figure 4. Surface of the sample before wear

Figure 5. Surface of the sample after applying the wear cycles

The data obtained are presented in Section 3.3, where the trends in roughness evolution and their implications on the wear resistance of PEO coatings are discussed, as a function of treatment duration.

RESULTS AND DISCUSSION

Surface Morphology of PEO Coatings

Figure 6 (a, b, and c) shows SEM images of the layers deposited on the AA2024 aluminum alloy using the PEO technique.

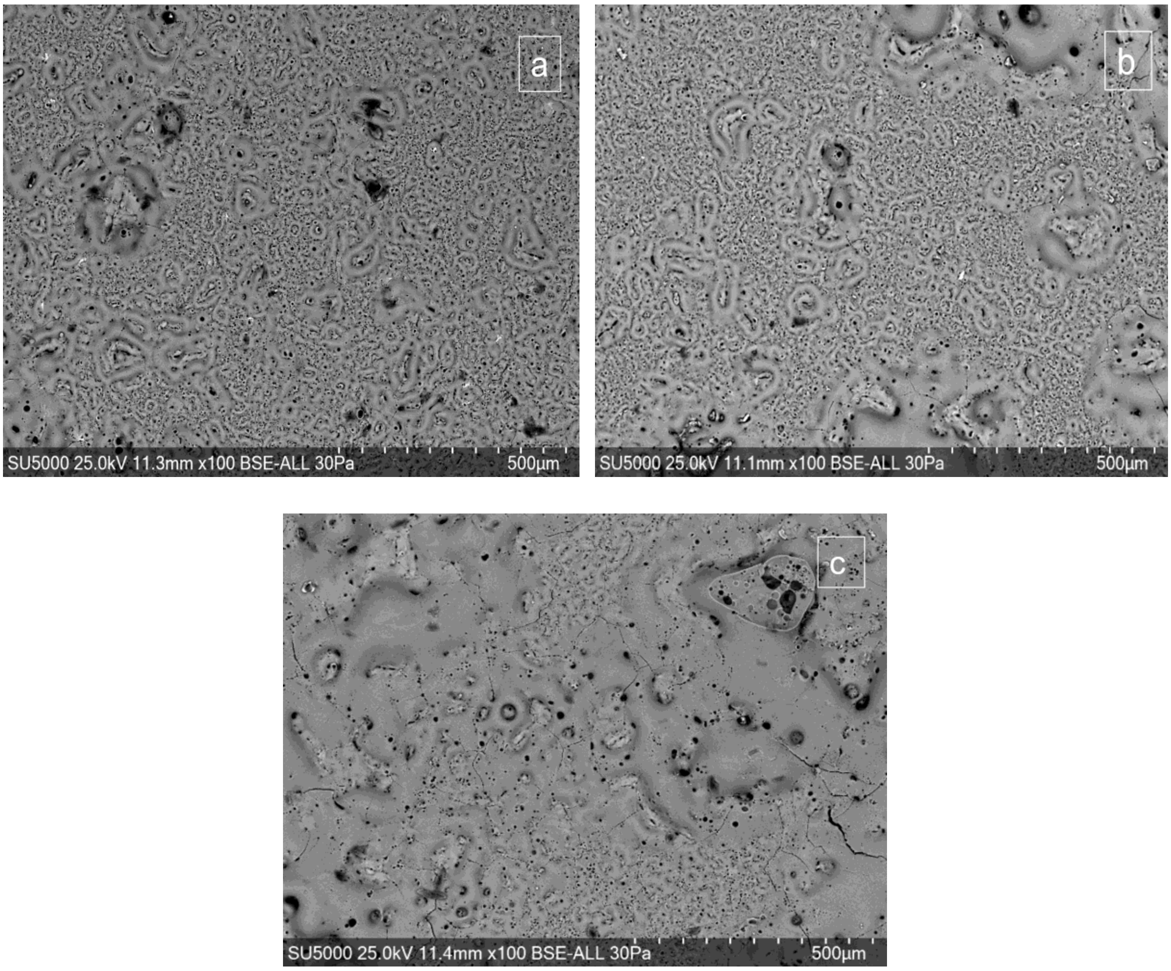

Figure 6. SEM micrographs of oxide layers obtained by PEO on an AA2024 aluminum alloy at treatment durations of: a. 2 minutes, b. 4 minutes, c. 6 minutes

The surface morphology of the layers obtained through PEO reveals a significant amount of porosity, which may be caused by gas trapped in bubbles generated during the conversion process. PEO employs small air bubbles to increase turbulence in the electrolyte, enabling a more homogeneous coating growth. The molten ceramic components rapidly cool upon contact with the electrolyte, while the air and vapors dissolved in the solution become trapped during coating formation [6].

The formation of pores in the resulting coatings is inevitable and depends on the discharge parameters. Pore formation phenomena during oxide layer growth occur after the initial formation of the oxide film. Pores may form rapidly in the early stages, then grow in size, followed by a reduction due to prolonged oxidation time [10].

The layer obtained after 6 minutes of PEO treatment contains a higher proportion of pores and microcracks, formed as a result of localized fusion caused by dielectric breakdown, as also reported in other scientific studies [11–13].

Chemical Composition

Figure 7 presents the EDX spectra for coatings obtained at 350 V and treatment durations of 2, 4, and 6 minutes.

It can be observed that the coatings are composed of Al, O, Si, and Na, in agreement with the chemical composition of the substrate (AA2024 aluminum alloy) and the electrolyte solution, which contains silicates. Undesirable components generally originate from the decomposition of the aqueous electrolyte.

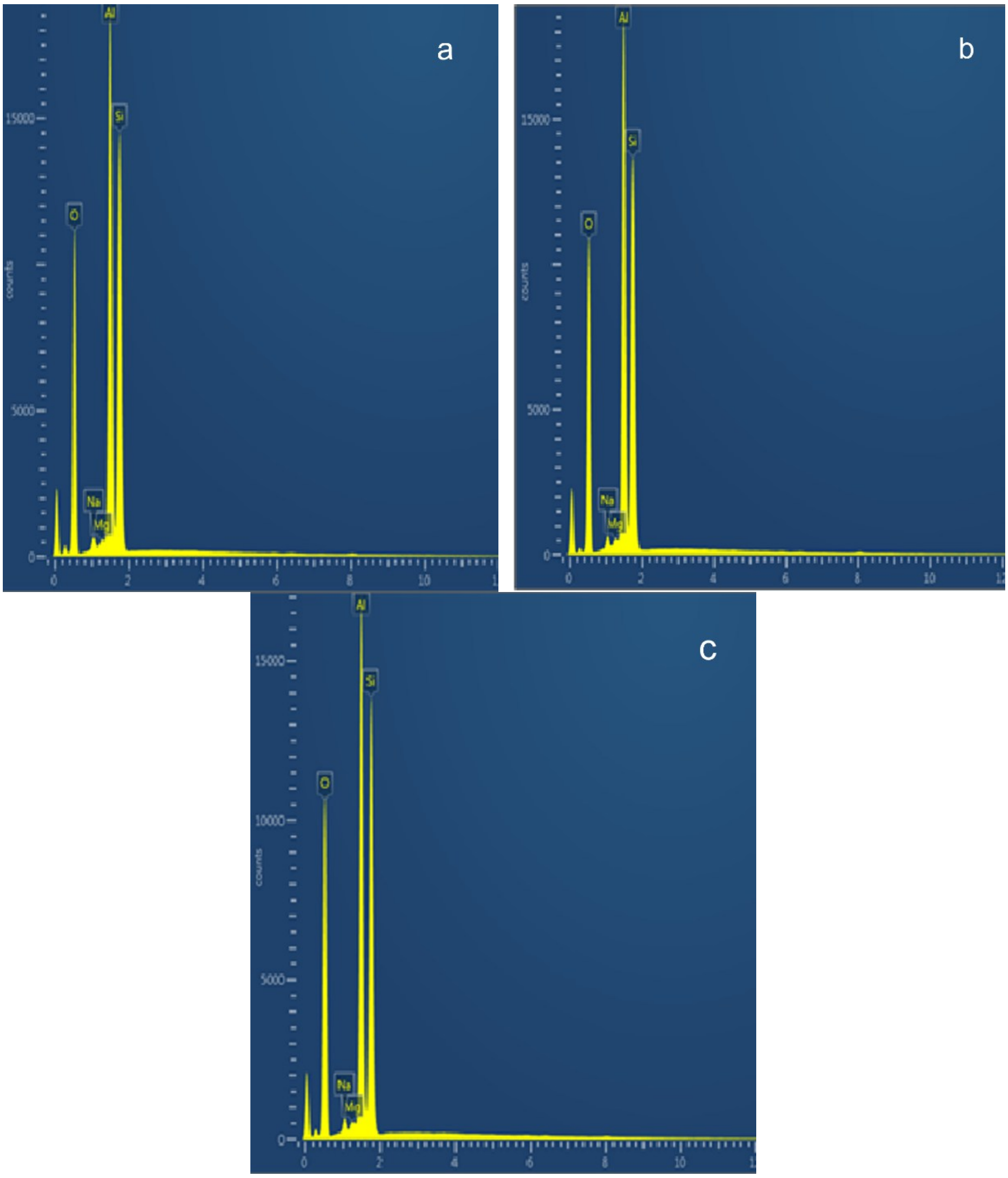

Figure 7. EDX spectra of PEO coatings obtained at 350 V for different treatment durations: (a) 2 min, (b) 4 min, (c) 6 min

The relative content of Al, O, Si, and Na on the surface of the PEO-treated samples is presented in Table 2.

Table 2. Relative surface content of Al, O, Si, and Na in the PEO coatings

Sample	**Relative content**			
	Al (at.%)	**O (at.%)**	**Si (at.%)**	**Na(at.%)**
350V, 2 min	24.31	49,60	24,88	0,91
350V, 4 min	24.89	49,47	24,30	1,00
350V, 6 min	23.47	50,15	25,08	0,98

The chemical composition analysis indicates the presence of key components for oxide formation (Al and O), as well as elements originating from the electrolyte (Na and Si). It can be observed that the distribution of Al and O across each coating was relatively uniform.

At the grain boundaries, numerous small pores are uniformly distributed, formed as a result of gas migration during plasma discharge. The coating formation mechanism of PEO on aluminum alloys has been described in prior publications [14–15].

Tribological Behavior

The tribological behavior of the coatings obtained by Plasma Electrolytic Oxidation (PEO) was evaluated by monitoring the arithmetic surface roughness, Ra [µm], measured before and after applying 500, 1000, and 2000 loading cycles in a plane-on-cylinder configuration.

The resulting values are presented in Table 3.

Table 3. Evolution of surface roughness (Ra) with increasing number of wear cycles

Sample no.	Duration [min]	Initial Ra[µm]	Ra_500c [µm]	Ra_1000c [µm]	Ra_2000c [µm]
1.	2	5,79	4,15	3,05	2,33
2.	4	5,86	4,37	4,11	2,87
3.	6	8,08	5,12	4,03	2,95

The results highlight a progressive decrease in surface roughness for all three samples as the number of wear cycles increases. This behavior contrasts with that commonly observed in untreated metallic materials, where roughness tends to increase due to the onset of abrasive wear.

In the case of the PEO-treated samples, this phenomenon can be explained by a progressive compaction and localized smoothing of the ceramic layer, caused by repeated friction on an abrasive surface with controlled grain size. Under the applied load, the prominent asperities of the oxide layer are gradually worn down without compromising the overall integrity of the coating, resulting in a decrease in Ra.

Sample 1, treated for 2 minutes, shows the greatest Ra variation, from 5.79 µm to 2.33 µm, suggesting a more porous and less durable surface, with a pronounced tendency for mechanical smoothing.

Sample 2, treated for 4 minutes, exhibits a slower decrease in roughness, and the values recorded at 1000 and 2000 cycles are higher than those of sample 1, indicating better stability of the oxide microstructure.

Sample 3, treated for 6 minutes, starts with the highest initial roughness (8.08 µm), reflecting a more developed PEO morphology. However, after 2000 cycles, the Ra drops to 2.95 µm, similar to the other two samples, suggesting a thicker coating with higher resistance to disintegration, though still subject to significant smoothing over time.

Interpretation of Wear Behavior as a Function of PEO Duration

The data indicate a clear correlation between oxidation time and tribological performance:

- Short time (2 min): thinner layer, accelerated wear, and steep Ra reduction;
- Medium time (4 min): more stable behavior with a controlled decrease in roughness;
- Long time (6 min): high initial roughness but superior mechanical resilience and balanced cyclic behavior.

This evolution is consistent with findings in the literature, which report that longer PEO durations lead to the formation of thicker, more consolidated layers with improved adhesion—capable of withstanding repeated tribological loading without major degradation.

Structure–Performance Correlation

The treatment duration in the Plasma Electrolytic Oxidation (PEO) process has a direct impact on the structure of the ceramic coating and, consequently, on its performance under wear conditions.

The results of this study indicate that longer treatment times improve wear resistance, especially under cyclic friction conditions.

Influence of PEO Duration on Wear Resistance

Samples treated for longer durations (4 and 6 minutes) exhibited superior tribological performance compared to the sample treated for only 2 minutes. Although all samples showed a decrease in Ra after wear testing, this reduction was more gradual and balanced for longer treatment durations, suggesting enhanced microstructural stability.

The sample treated for 6 minutes, despite having the highest initial roughness, showed remarkable resistance to material loss and compaction, maintaining a roughness similar to the other samples after

2000 cycles. This behavior indicates a more durable coating capable of withstanding repeated tribological stress without significant degradation.

Possible Explanations: Layer Density, Composition, and Adhesion

The observed differences in behavior can be explained by several factors linked to the structural evolution of the oxide layer with increasing PEO duration:

- Layer density and thickness: Longer treatment times allow for the formation of thicker, more compact layers that provide a more uniform and effective mechanical barrier against wear. This is confirmed by SEM micrographs showing a more extensive porous structure in the 6-minute sample.
- Chemical composition: EDX spectra indicate a consistent presence of oxygen and aluminum in all coatings; however, longer reaction times may enable more complete diffusion of electrolyte-derived elements (Na, Si) into the oxide layer, promoting the formation of more stable ceramic compounds.
- Coating–substrate adhesion: In PEO treatments, the process duration influences the degree of interpenetration between the substrate and oxide layer due to repeated micro-discharges. This leads to better adhesion of the coating, reducing the risk of delamination under mechanical load.

Overall, the experimental data and morphological/chemical characterizations converge toward the conclusion that a 6-minute PEO treatment provides the optimal balance between coating development, adhesion, chemical stability, and tribological behavior—making it the most effective choice for applications involving cyclic mechanical loads.

CONCLUSIONS

This study demonstrated the significant influence of treatment duration in the Plasma Electrolytic Oxidation (PEO) process on the tribological performance of ceramic coatings on aluminum alloys.
Increasing the treatment time led to the formation of more uniform and morphologically porous coatings with stable chemical composition, as confirmed by SEM and EDX analyses.
The tribological tests revealed that samples treated for longer durations exhibited superior wear resistance, evidenced by smaller variations in surface roughness after repeated loading cycles.
The coating produced with a 6-minute treatment offered the best balance between thickness, adhesion, and wear behavior.
These findings are relevant for the development of high-performance functional surfaces in industrial applications where repeated mechanical stresses demand elevated wear resistance.

ACKNOWLEDGEMENTS

The authors thank Negrea Denis for his kindness and assistance in the characterization of the samples, through SEM and EDS characterization techniques.

REFERENCES

[1]. B. Kasalica, M. Petković-Benazzouz, M. Sarvan, I. Belča, B. Maksimović, B. Misailović, Z. Popović, Mechanisms of plasma electrolytic oxidation of aluminum at the multi-hour timescales, Faculty of Physics, University of Belgrade, Belgrade, 2020

[2] A. Melhem, Oxydation par électrolytique: influence des paraétres du procédé sur le component des micro-décharges et conséquences sur les couches d`oxydes, Institut National Polytechnique de Lorraine, 2011

[3] M. S. Kim, J. J. Ryu, Y. M. Sung, Electrochem. Commun, vol. 9, pp. 1886, 2007

[4] B. Rajasekaran, S. G. S. Raman, S. V. Joshi, G. Sundararajan, Int. J. Fatigue, vol. 30, pp. 1259, 2008

[5] J. He, Q. Z. Cai, Y. G. Ji, H. H. Luo, D. J. Li, B. Yu, J. Alloys Compd., vol. 482, pp. 476, 2009

[6]. M. Treviño, N. F. Garza-Montes-de-Oca, A. Pérez, M. A. L. Hernández-Rodríguez, A. Juárez, & R. Colás, Wear of an aluminium alloy coated by plasma electrolytic oxidation, Surface & Coatings Technology, vol. 206(8), pp. 2213–2219, 2011
[7] A.L. Yerokhin, X.Y. Nie, Leyland, A. Matthews, S.J. Dowey, Plasma electrolysis for surface engineering, Surface & Coatings Technology, vol. 122(2−3), pp. 73−93, 1999
[8] J.-X. Han, Y.-I. Cheng, W-Bin Tu, T-yan Zhan, Y.-liang Cheng, The black and white coatings on Ti−6Al−4V alloy or pure titanium by plasma electrolytic oxidation in concentrated silicate electrolyte, Applied Surface Science, vol. 428(15), pp. 684−697, 2018
[9] C.C. Tseng, J.L. Lee, T.H. Kuo, S.N. Kuo S N, K.H. Tseng, The influence of sodium tungstate concentration and anodizing conditions on microarc oxidation (MAO) coatings for aluminum alloy, Surface & Coatings Technology, vol. 206(16), pp. 3437−3443, 2012
[10] PY Hou, Y. Niu, C. Van Lienden, Analysis of Pore Formation at Oxide- Alloy Interfaces, part I : Experimental Results on FeAl, Oxidation of Metals, vol. 59, pp41-6, 2003
[11] J.A. Curran, T.W. Clyne, Acta Mater., vol. 54, pp. 1985, 2006
[12] J.A. Curran, T.W. Clyne, Surf. Coat. Technol., vol. 199, pp/ 168, 2005
[13] J.A. Curran, T.W. Clyne, Surf. Coat. Technol., vol. 199, pp. 177, 2005
[14] F. Simchen, M. Sieber, A. Kopp, T. Lampke, Introduction to Plasma Electrolytic Oxidation-An Overview of the Process and Applications. Coatings, vol.10, pp. 628, 2020
[15] S. Sikdar, P.V. Menezes, R. Maccione, T. Jacob, P.L. Menezes, Plasma Electrolytic Oxidation (PEO) Process-Processing, Properties, and Applications. Nanomaterials, vol. 11, pp. 1375, 2021