

Article

Predictive Evaluation of Aluminum Profile Corrosion Using Electrochemical Impedance Spectroscopy

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Highlights

What are the main findings?

- Electrochemical impedance spectroscopy tracked corrosion evolution of treated aluminum profiles.
- Corrosion mechanisms were identified using time-dependent equivalent circuit models.
- Surface treatments showed distinct corrosion and impact durability performance.

What are the implications of the main findings?

- EIS enables predictive assessment of coating degradation and corrosion resistance.
- Surface treatment selection should consider both exposure time and service conditions.
- Combined corrosion and impact testing improve durability evaluation of aluminum coatings.

Abstract

This study investigates the time-dependent corrosion behavior of 6060 aluminum profiles subjected to three industrially relevant surface treatments: clear anodizing, black anodizing, and pre-anodizing followed by polyester powder coating during 30 days of immersion in NaCl solution. Electrochemical Impedance Spectroscopy combined with equivalent electrical circuit modeling was used to monitor corrosion evolution and identify the governing degradation mechanisms. Black-anodized specimens exhibited the highest initial corrosion resistance during the first 4 days of immersion, while clear-anodized specimens provided superior protection between 7 and 15 days due to the formation of a more protective inner oxide layer. Both anodized systems showed evidence of repassivation during prolonged exposure through corrosion product accumulation within the porous oxide structure. The pre-anodized/powder-coated system delayed corrosion initiation beyond 15 days, demonstrating the highest long-term barrier performance. Dynamic perpendicular impact loading after corrosion exposure revealed significant differences in mechanical durability: clear-anodized specimens maintained coating integrity up to 15 days, whereas black-anodized specimens experienced complete coating removal, associated with corrosion-induced microcracks and void formation observed by SEM. Overall, clear

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anodizing provided the best balance between corrosion resistance and post-corrosion mechanical durability, while the pre-anodized/powder-coated system offered the greatest long-term corrosion protection, demonstrating that the optimum surface treatment depends on the intended service conditions.

Keywords: aluminum; anodizing; corrosion; equivalent circuits modeling; impact tests

1. Introduction

The corrosion behavior of aluminum profiles is influenced by several factors, including the alloy composition, microstructure, environmental conditions and surface treatments. The type and concentration of alloying elements can impact corrosion susceptibility [1–7]. Characteristics such as grain size, phase distribution and the presence of secondary phases influence the electrochemical stability of aluminum. A coarse grain structure or continuous intermetallic networks may act as preferential sites for corrosion [8–11]. Exposure to aggressive environments, particularly those containing chloride ions along with high humidity and temperature, can dramatically accelerate the corrosion process [12–14]. These conditions damage the protective oxide layer on aluminum, increasing its susceptibility to corrosion. Techniques such as anodizing, pre-anodizing and painting play a critical role in enhancing corrosion resistance. These treatments form protective barrier layers that reduce the interaction between the aluminum surface and the external environment, thereby improving long-term durability [15–18].

Anodizing is one of the most widely employed surface modification techniques for aluminum alloys due to its ability to enhance corrosion resistance, wear performance, and surface durability while maintaining the lightweight characteristics of the substrate. The process involves an electrochemical oxidation treatment in which aluminum acts as the anode in an electrolytic cell, resulting in the formation of a controlled oxide layer consisting of an inner compact barrier region and an outer porous structure. The characteristics of the anodic layer, including thickness, porosity, pore morphology, and sealing condition, depend on parameters such as electrolyte composition, applied current density, voltage, temperature, and treatment time. Post-treatment processes, such as sealing or the application of organic coatings, are commonly employed to reduce electrolyte penetration through the porous structure and further improve the long-term corrosion protection of aluminum components. These surface treatments are extensively applied in architectural, transportation, and aerospace applications where prolonged exposure to aggressive environments is expected.

The prediction of corrosion in aluminum profiles using Electrochemical Impedance Spectroscopy (EIS) techniques is a crucial area of research with significant applications across various industrial sectors, including aerospace, automotive and construction. EIS offers detailed insights into the corrosion mechanisms and the kinetics of electrochemical reactions occurring at the aluminum-electrolyte interface. The technique involves applying a small-amplitude alternating potential signal to an electrochemical system and measuring the resulting current response. Information about the system's impedance can be obtained by studying the correlation between the applied voltage and the resulting current. This impedance reflects the resistance of the material to the flow of alternating current and provides valuable data regarding its electrochemical behavior [19–22]. EIS is a powerful tool to evaluate corrosion resistance, identify the presence of defects or coating degradation and predict the service life of the material [23]. Corrosion of aluminum can take various forms, such as uniform corrosion, pitting corrosion, intergranular corrosion and stress corrosion cracking [2]. Electrochemical Impedance Spectroscopy (EIS) is a

powerful technique that can be used to study these mechanisms. Uniform corrosion is characterized by an even loss of material across the surface [24]. Intergranular corrosion, on the other hand, occurs along the grain boundaries of the metal, and EIS can reveal changes in the surface structure related to this localized degradation [25].

EIS enables the prediction of aluminum profile corrosion through several approaches. By monitoring corrosion resistance using EIS data, it becomes possible to observe changes in the material's resistance over time and gain important knowledge about the corrosion process [23]. Furthermore, EIS plots can be used to develop electrochemical models that simulate the behavior of aluminum under different corrosive environments, providing a deeper understanding of its degradation processes [24]. Finally, this technique can be used to study corrosion inhibitors, helping to evaluate their efficiency and optimize their compositions for better protection [26,27].

Research on predicting corrosion in aluminum profiles using Electrochemical Impedance Spectroscopy (EIS) is continuously evolving, with ongoing efforts to develop new methodologies and techniques that improve both the accuracy and reliability of corrosion predictions. In this context, equivalent electrical circuits (EECs) play a fundamental role in the analysis and interpretation of EIS data [28,29]. By simulating the electrochemical processes occurring at the metal-electrolyte interface, EECs allow for the quantification of important parameters such as solution resistance, charge transfer resistance, double-layer capacitance and diffusion-related elements. These parameters offer valuable information about the nature and extent of corrosion, as well as the effectiveness of protective coatings or oxide layers. Moreover, using EECs enables the comparison of different corrosion states over time or under varying environmental conditions. The use of accurate circuit models enhances the predictive power of EIS, making it a more reliable tool for assessing long-term material performance and guiding the development of more durable aluminum alloys and surface treatments.

In this study, an electrochemical investigation of the corrosion behavior of different surface-treated aluminum samples immersed in 3.5 wt.% NaCl solutions for prolonged periods is conducted using electrochemical impedance spectroscopy (EIS). The aim is to identify representative equivalent circuit models for each system in order to elucidate the corrosion mechanisms and their evolution as a function of immersion time, as well as to evaluate key parameters associated with the corrosion processes and the properties of the passive films.

The novelty of this work lies in the evaluation of the combined effects of corrosion and wear durability of anodized aluminum samples through dynamic impact loading tests performed on pre-corroded specimens. This approach establishes a more comprehensive framework for durability assessment of aluminum materials exposed to chloride-rich environments, as well as additional real-world factors such as wind-blown particles, dust, and other mechanical impacts that may coexist in practical service conditions. Since corrosion-induced degradation may weaken the protective coating and alter its resistance to mechanical damage, evaluating the impact durability after corrosion provides valuable information regarding the long-term performance and service reliability of the investigated surface treatment systems.

2. Materials and Methods

In this research, three samples made from EN AW 6060 aluminum alloy profiles are analyzed. The chemical composition of the alloy (wt.%) was as follows: Si: 0.3–0.6, Fe: 0.10–0.30, Cu: max. 0.10, Mn: max. 0.10, Mg: 0.35–0.6, Cr: max. 0.05, Zn: max. 0.15, Ti: max. 0.15 with the balance Al. These samples were previously subjected to various surface modification techniques: the first is clear anodizing, the second is black anodizing, and the third is a combination of pre-anodizing followed by painting. These treatments are selected to evaluate their impact on both corrosion resistance and visual appearance. The clear- and black-anodized samples were processed using sulfuric acid anodizing, carried

out in compliance with Qualanod [30] and DIN 17611 [31] standards. The anodizing procedure involved several stages: alkaline degreasing, alkaline etching, desmutting, anodizing itself, electrocoloring, and cold sealing. Therefore, the anodic oxide layers investigated in this study correspond to a sealed anodized condition rather than an unsealed porous anodic layer. Accurate control over these processing parameters ensures a consistent and durable oxide coating, making it suitable for a wide range of applications. The resulting oxide layer is approximately 17 μm thick, consisting of hexagonal columnar structures with nanoporous features. The third treated sample was initially pre-anodized to form a protective oxide layer, approximately 5–8 μm thick, before the application of architectural-grade polyester powder coating. This pre-anodizing step enhances the corrosion resistance of the aluminum surface before powder coating. The combined process was carried out in accordance with Qualicoat specifications. Overall, the three surface treatment approaches, clear anodizing, black anodizing, and pre-anodizing with powder coating, offer a comprehensive comparison of different surface engineering methods for improving the performance and appearance of EN AW 6060 aluminum alloys [15,32–34].

Scanning electron microscopy (SEM) analysis is performed using a Phenom ProX desktop SEM (Thermo Fisher Scientific Waltham, MA, USA) to investigate the surface morphology of the anodized and pre-anodized/powder-coated samples. The SEM images are used to evaluate the morphological characteristics of the anodic layers, including surface homogeneity, pore features, and differences between the investigated surface treatments.

The corrosion behavior of the aluminum samples is evaluated through the electrochemical impedance spectroscopy (EIS) technique. EIS measurements are conducted over a broad frequency range, typically from 100 kHz down to 100 mHz, using an amplitude of 10 mV peak-to-peak. Prior to each test, the Open Circuit Potential (OCP) is monitored by immersing the sample in the test solution until a stable potential is achieved. An OCP stabilization period of 30 min is employed for all the experiments, which is sufficient for the OCP to reach a steady state. All the electrochemical experiments are performed in a 3.5 wt% NaCl solution at ambient temperature (approximately 25 °C), using a Gamry potentiostat. A classic three-electrode arrangement is employed, with the aluminum sample serving as the working electrode, a platinum wire as the counter electrode, and a saturated calomel electrode (SCE) as the reference. The working electrode area exposed to the electrolyte is 1 cm^2 . To track the corrosion progression and stability of the surface modifications over time, measurements are taken at predefined intervals: after 0, 1, 4, 7, 10, 15, 20, 25, and 30 days (where 0 days corresponds to the initial, as-received samples). This approach provided a detailed overview of the electrochemical response over time, allowing evaluation of the long-term effectiveness of the different treatments. All the EIS and impact tests are performed at least three times to ensure reproducibility. The extracted EIS results are normalized to the same surface area to enable accurate comparison between samples. The reported results for both EIS and impact tests represent the average values obtained from the independent experiments.

Perpendicular impact tests are conducted on anodized aluminum specimens, following different durations of exposure to a corrosive environment, for assessing their wear resistance under dynamic loading conditions. The employed impact tester was developed by Impact-BZ in partnership with the Laboratory for Machine Tools and Manufacturing Engineering of Aristotle University of Thessaloniki. A 5 mm diameter ceramic ball repeatedly impacts the specimen under a controllable maximum load. A proportional–integral–derivative (PID) controller regulates the output voltage of a variable transformer, which drives a direct current (DC) motor, to maintain constant impact force peaks throughout the test. In addition, key process parameters, including current, force, temperature, and other relevant variables, are continuously measured and monitored during the experiment. The applied force signal during the impact test is also shown in Figure 1. During

testing, both the applied load and the number of impact cycles are maintained constant at 120 N and 5000 impacts, respectively, in order to ensure consistent comparison among samples. The applied load and number of impacts are carefully chosen to avoid sudden, extensive surface damage while allowing a gradual progression of wear throughout the testing process. The resulting impact imprints are analyzed using 3D measurements obtained by the μ SURF confocal microscope from NANOFOCUS AG (Oberhausen, Germany).

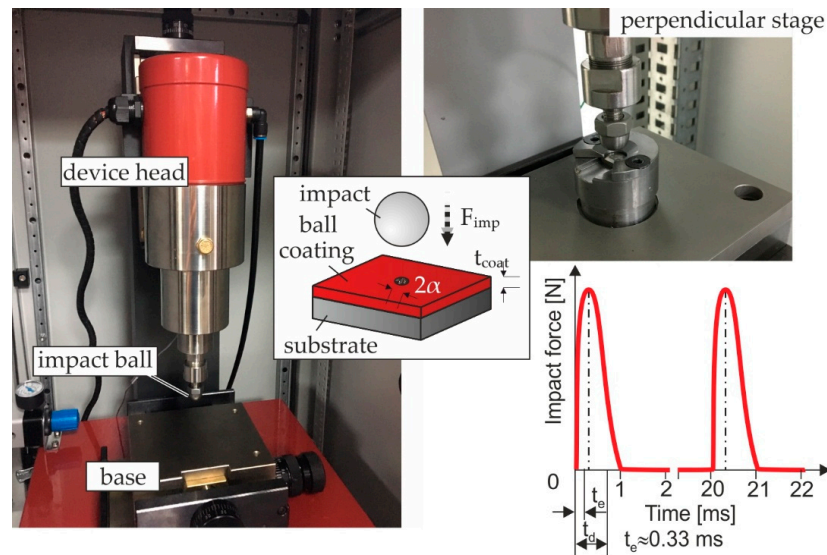


Figure 1. The employed impact tester and the applied force signal.

3. Results and Discussion

3.1. Surface Morphology Observation

Figure 2 presents SEM images of the clear- and black-anodized samples, together with the morphology of the pre-anodized and powder-coated samples. The anodic layer of the clear-anodized sample exhibits a relatively smooth and homogeneous morphology, with limited visible porous and fibrous features, indicating that the post-anodization sealing treatment was effective. The thickness of the clear anodic layer is approximately 13 μm . In contrast, the black-anodized samples display a more pronounced fiber-like oxide morphology, with larger pores extending along the anodization direction. The thickness of the black anodic layer is around 9 μm . Nevertheless, most of these pores do not reach the outer surface of the anodic layer, suggesting that the sealing process was partially successful. However, complete closure of all voids generated during anodization was not achieved. The observed differences in surface morphology indicate that the clear- and black-anodized samples exhibit different characteristics of the outer anodic structure after the industrial sealing treatment. The more compact appearance of the clear-anodized surface suggests a lower exposure of the porous structure to the electrolyte, whereas the more pronounced porous features observed for the black-anodized surface may influence the interaction between the anodic layer and the electrolyte during prolonged immersion. For the pre-anodized and powder-coated samples, high-magnification images reveal that the pre-anodized oxide layer exhibits a predominantly fiber-like morphology, with open porous features extending to the outer surface of the anodic layer. This morphology is consistent with an unsealed pre-anodized condition prior to the application of the powder coating. The thickness of the pre-anodized oxide layer is approximately 7 μm , while the applied powder coating exhibits a thickness close to 80 μm . Finally, a powder coating was deposited on top of the oxide layer to fill and cover the pores formed during anodization,

resulting in a more compact surface morphology. The powder coating appears to conform well to the underlying oxide morphology and provides complete coverage of the anodized surface. No obvious powder coating defects or discontinuities are observed at the magnification examined, indicating satisfactory adhesion and pore filling by the coat layer.

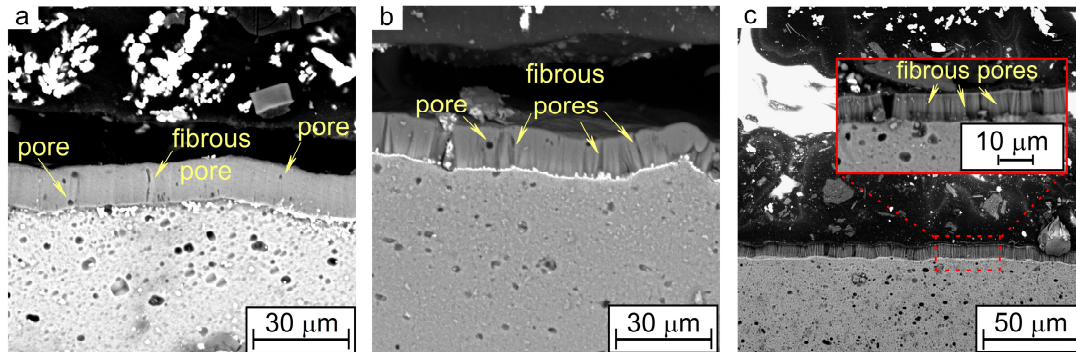


Figure 2. Cross-sectional SEM images showing the microstructural features of (a) clear-anodized, (b) black-anodized, and (c) pre-anodized and powder-coated samples.

3.2. Time-Dependent Evolution of EIS Fitting Parameters for Anodized and Pre-Anodized and Powder-Coated Aluminum Specimens in NaCl Solution

The corrosion performance of aluminum specimens subjected to different surface treatments and immersed in saline solutions is investigated over time using electrochemical impedance spectroscopy (EIS), as shown in Bode and Nyquist diagrams depicted in Figures 3 and 4. The evolution of the corrosion process is interpreted through equivalent electrical circuit (EEC) modeling, with different configurations applied to account for the distinct electrochemical responses observed (Figures 5 and 6). Table 1 presents a summary of the fitted equivalent circuit parameters obtained under all the analyzed conditions for the anodized specimens.

Table 1. Fitted EIS parameters of the proposed equivalent circuits for both clear- and black-anodized specimens, normalized to the exposed surface area.

Days		R_{ct} $10^4 \Omega$	CPE_{dl} $10^{-7} S s^a$	α_{dl}	R_{in} $10^4 \Omega$	CPE_{in} μF	R_{out} $10^4 \Omega$	CPE_{out} μF	W $10^{-6} S s^{1/2}$	L $10^4 H$	R_L $10^4 \Omega$
clear	0	-	-	-	227.8	0.01	0.4	1.6	1.2	-	-
	1	-	-	-	407.4	0.02	0.5	0.01	1.0	-	-
	4	-	-	-	285.6	0.02	2.5	0.003	1.8	-	-
	7	57.6	7.2	0.84	0.2	0.01	4.5	0.06	376	-	-
	10	1.0	40.5	0.8	0.02	0.44	0.3	3.71	-	1.0	0.5
	15	-	-	-	113.2	0.18	0.8	0.002	36.2	-	-
	20	21.8	35.5	0.82	1.1	0.10	0.03	0.002	67.1	-	-
	25	1.4	108.6	1.00	1.8	1.12	0.1	0.001	1399	-	-
	30	0.3	314.1	0.53	0.06	$0.96 \cdot 10^{-4}$	0.1	138.6	-	0.1	0.1
black	0	-	-	-	139.50	0.07	52.00	0.01	0.2	-	-
	1	-	-	-	100.30	0.01	1542	0.44	0.3	-	-
	4	-	-	-	73.76	0.01	1378	0.65	0.2	-	-
	7	1.6	26.6	0.78	0.23	0.01	0.07	0.65	-	0.3	2.1
	10	0.1	492.3	0.82	0.10	10.7	0.01	13.47	-	0.1	0.1
	15	0.4	428.2	0.87	0.02	0.01	0.30	1.71	-	1.1	0.5
	20	1.7	0.1	0.86	37.85	0.20	18.20	0.10	-	4.9	60.2

25	1.1	0.1	0.88	5.65	0.89	0.80	0.39	-	3.2	4.2
30	0.5	7.0	0.90	2.36	0.35	0.50	4.83	-	0.3	1.5

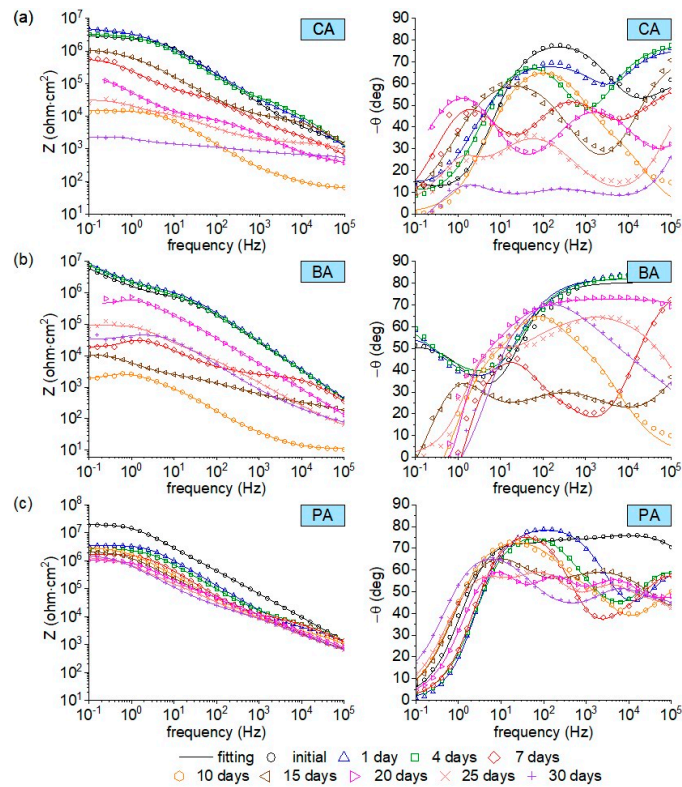


Figure 3. Bode magnitude plots (left), Bode phase plots (right), and equivalent circuit fitting results for (a) clear-anodized (CA), (b) black-anodized (BA), and (c) pre-anodized and powder-coated aluminum (PA) samples after immersion in 3.5 wt.% sodium chloride solution for various immersion times.

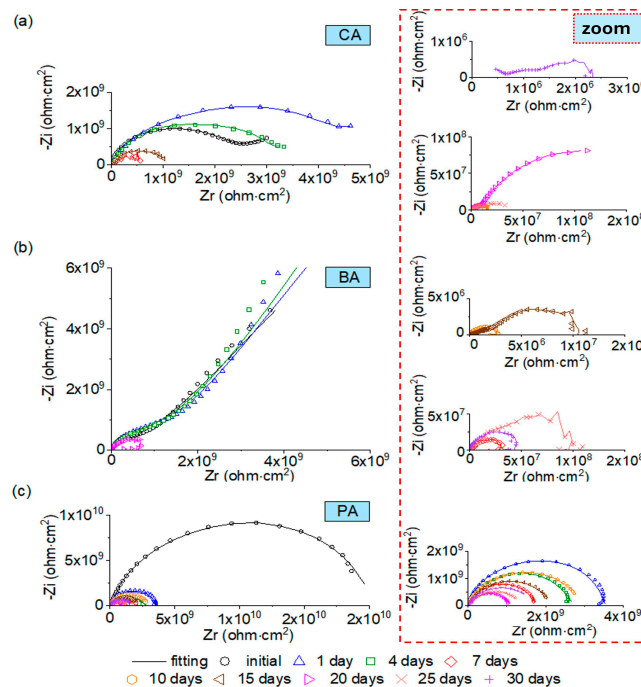


Figure 4. Nyquist plots and fitting data of (a) clear-anodized (CA), (b) black-anodized (BA), and (c) pre-anodized and powder-coated (PA) aluminum samples after immersion in 3.5 wt.% sodium

chloride solution for various immersion times. The right-hand panels display the Nyquist plots using different axis scales (**zoom**) to enhance the visibility of spectra with lower impedance values that would otherwise not be distinguishable in the main plots. In some cases, the y -axis is truncated (not rescaled) to improve readability and optimize figure layout.

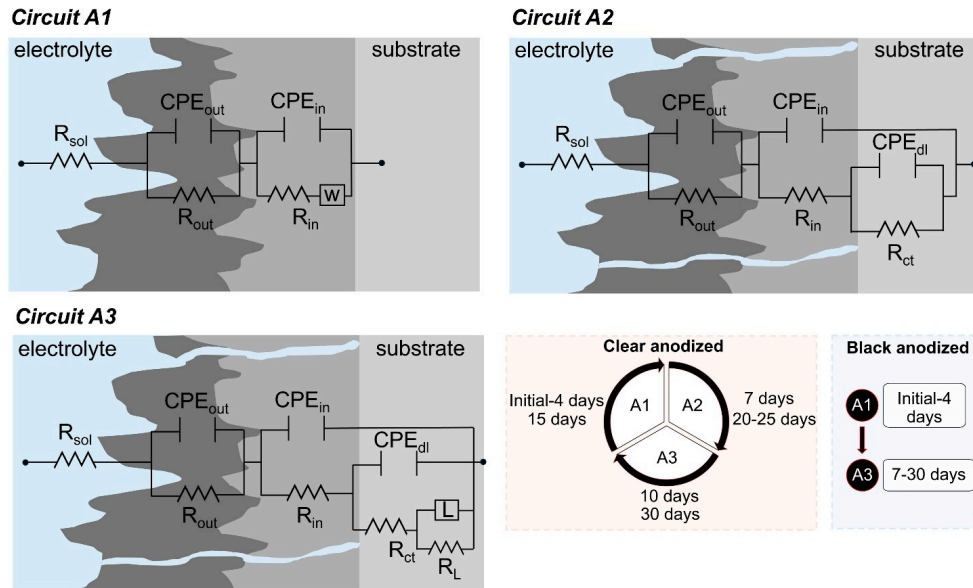


Figure 5. Equivalent circuit models used to represent and analyze the corrosion in clear- and black-anodized aluminum specimens.

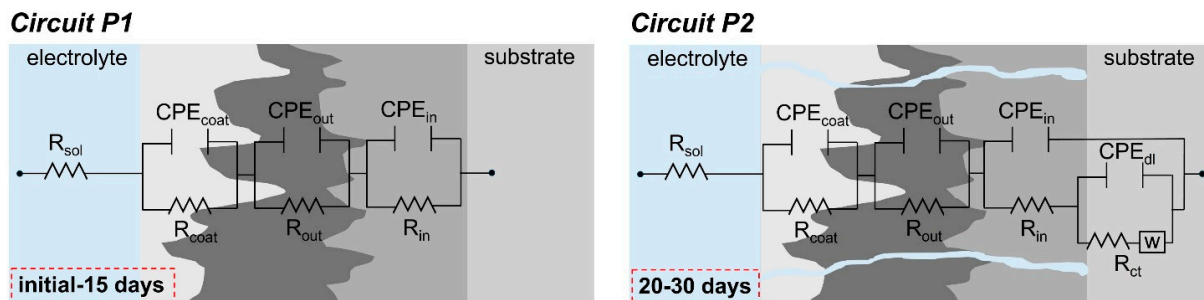


Figure 6. Equivalent circuit models used to represent and analyze the corrosion evolution in pre-anodized and powder-coated aluminum specimens.

For the anodized specimens, three equivalent circuits ($A_1 - A_3$) are used to describe progressive oxide layer degradation. Circuit A_1 comprises two time constants associated with the compact inner barrier layer ($R_{in}|CPE_{in}$) and the porous outer layer ($R_{out}|CPE_{out}$). Circuit A_2 includes an additional contribution from active corrosion processes, represented by ($R_{ct}|CPE_{dl}|W$), accounting for charge transfer and diffusion-controlled behavior. Circuit A_3 further incorporates an inductive element ($L|R_L$), related to adsorption–desorption phenomena and metastable pitting activity.

To allow meaningful comparison of the capacitive response across immersion times, the CPE parameters (Q, n) obtained from the equivalent circuit fittings are converted into an effective capacitance (C_{eff} , $\mu F/cm^2$) using the Hsu–Mansfeld (Equation (1)) [35].

$$C_{eff} = Q^{1/\alpha} \cdot R^{(1-\alpha)/\alpha} \quad (1)$$

where R is the resistance associated in parallel with each CPE (R_{in} for the inner barrier oxide, R_{out} for the outer porous oxide, and R_{coat} for the powder coating). Expressing the

results as C_{eff} in $\mu\text{F}/\text{cm}^2$ rather than raw Q values allows direct, quantitative comparison of the capacitive response across samples and exposure times, independent of the fitted n exponent. Therefore, the capacitance values reported in Tables 1 and 2 correspond to the calculated effective capacitances.

Table 2. Fitted EIS parameters of the proposed equivalent circuits for the pre-anodized and powder-coated specimens, normalized to the exposed surface area.

	R_{ct}	CPE_{dl}		R_{in}	CPE_{in}	R_{out}	CPE_{out}	R_{coat}	CPE_{coat}	W
	$10^4 \Omega$	10^{-7} S s^a	α_{dl}	$10^4 \Omega$	μF	$10^4 \Omega$	μF	$10^4 \Omega$	μF	$10^{-4} \text{ S s}^{1/2}$
0	-	-	-	0.8	0.019	22.5	0.016	1999.0	0.009	-
1 day	-	-	-	0.3	0.02	0.3	0.003	49.8	0.064	-
4 days	-	-	-	0.7	0.01	0.3	0.003	267.3	0.021	-
7 days	-	-	-	0.8	0.005	0.1	0.004	174.2	2.116	-
10 days	-	-	-	0.3	0.002	0.4	0.045	296.8	0.08	-
15 days	-	-	-	0.02	0.004	3.4	0.036	215.6	0.096	-
20 days	94.2	1.0	0.91	0.1	0.024	0.8	0.015	0.1	0.006	1.1
25 days	129.9	2.1	0.85	0.1	0.007	4.8	0.133	0.6	0.034	1.0
30 days	1.1	6.1	0.69	0.9	0.013	144.6	0.255	0.1	0.011	6922.0

For the clear-anodized specimens, the response during the first 4 days is well described by A_1 , indicating an intact barrier and absence of active corrosion. The increase in R_{in} and R_{out} suggests partial pore blocking and densification of the oxide layer [36,37]. After 7 days, the spectra are fitted by A_2 , marking the onset of corrosion activity, evidenced by decreased R_{in} and increased CPE_{in} , along with changes in the outer layer consistent with oxide hydration and chloride ingress. After 10 days, A_3 is required, indicating significant degradation of both layers and initiation of localized corrosion, as reflected by pronounced reductions in R_{in} and R_{out} and increased interfacial heterogeneity.

At 15 days, active corrosion is temporarily suppressed, consistent with the formation of corrosion products acting as a blocking layer, as indicated by increased resistive and decreased capacitive responses. From 20 to 25 days, film degradation resumes with reactivation of the corrosion branch, while at 30 days, localized corrosion is re-established due to breakdown of this protective layer.

For the black-anodized specimens, A_1 describes the first 4 days, indicating no active corrosion. A decrease in R_{in} and an increase in R_{out} suggest inner-layer degradation accompanied by partial sealing of the outer porous structure. From 7 to 30 days, A_3 is required, indicating extensive oxide breakdown and electrolyte penetration to the substrate, resulting in localized corrosion. By day 10, a marked drop in R_{ct} and an increase in CPE_{dl} confirm severe loss of corrosion resistance. Between 15 and 30 days, partial stabilization occurs due to corrosion product accumulation within pores, but this effect is transient. From 25 to 30 days, renewed destabilization leads to reactivation of localized corrosion, reflected in persistently low resistive parameters.

The comparative evaluation of the black- and clear-anodized specimens reveals a time-dependent evolution of their corrosion resistance during immersion in saline solution. Up to 4 days of immersion, the black-anodized specimens exhibit superior corrosion resistance, characterized by significantly higher R_{out} values despite comparatively lower R_{in} . This behavior suggests that the outer porous oxide layer formed during black anodizing acts as a more effective barrier against electrolyte ingress, likely due to differences in oxide morphology, pore structure, or corrosion products incorporation within the anodic film.

However, from 7 to 15 days of immersion, the black-anodized specimens exhibit substantially lower corrosion resistance compared to the clear-anodized counterparts. Equivalent circuit analysis reveals markedly lower R_{out} and R_{in} values, indicating progressive

degradation of both oxide sublayers. These findings suggest that, once the electrolyte penetrates the outer porous layer, the lower resistance of the inner barrier oxide in the black-anodized specimens facilitates ionic transport toward the aluminum substrate, thereby accelerating localized corrosion processes. In contrast, the higher R_{in} values observed for the clear-anodized specimens indicate a more stable inner barrier layer capable of more effectively restricting chloride ion transport.

From 20 days onward, the black-anodized specimens again show improved corrosion resistance relative to the clear-anodized samples, as evidenced by increased R_{in} , R_{out} and R_{ct} values. This behavior suggests enhanced repassivation capability and partial restoration of the protective properties of the oxide film. The increase in resistive parameters is likely associated with the accumulation of corrosion products within pores and oxide layer defects, resulting in partial sealing of active sites and suppression of charge-transfer reactions.

For the pre-anodized and powder-coated samples, the equivalent circuits used to fit the EIS data are identical to those applied to the anodized specimens, with the addition of an extra branch representing the outer powder coating ($R_{coat}|CPE_{coat}$), as shown in Figure 6. Table 2 summarizes the parameters obtained from fitting the EIS spectra using these equivalent circuits.

For the pre-anodized and powder-coated specimens, the EIS response is satisfactorily described by the equivalent circuit P_1 up to 15 days of immersion, indicating that no significant electrochemical activity associated with corrosion is detectable during this period of exposure to the saline environment. The resistance of the powder coating layer is markedly higher than that of the underlying oxide layers, highlighting the dominant barrier effect of the powder coating and its contribution to the overall corrosion protection of the system.

Following one day of immersion, a decrease in the resistance values of all the protective layers is observed, with the most pronounced reduction occurring in the powder coating. This behavior is attributed to the adsorption and diffusion of water molecules into the polymeric matrix, which constitutes the initial stage of powder coating degradation. Water uptake increases the dielectric constant of the coating and facilitates the transport of ionic species through the coating network, resulting in a reduction in its barrier properties [38]. As electrolyte penetration progresses, corrosive species gain access to the anodic oxide layers, leading to a decrease in the resistances of both the outer and inner oxide layers due to electrolyte ingress through the porous anodic structure.

After 20 days of immersion, the EIS data are well represented by circuit P_2 , signifying that the electrolyte reaches the metal substrate and metal dissolution is ongoing. At this stage, the powder coating layer exhibits a substantial decrease in resistance, indicating a significant loss of its barrier performance, and the powder coating can no longer be considered the primary protective component of the system. Consequently, corrosion resistance is predominantly governed by the anodic oxide layers. Despite the degradation of the powder coating, both the outer and inner oxide layers maintain relatively high resistance values, suggesting that they continue to provide effective protection by limiting electrolyte transport toward the metallic substrate and suppressing the development of corrosion processes.

The impedance modulus values at low frequency ($|Z|$ at 0.01 Hz) were further compared to evaluate the overall protective performance of the clear- and black-anodized specimens during prolonged immersion (Figure 7a) [39]. Both anodizing systems exhibited high initial impedance values, confirming the effective barrier properties of the sealed anodic layers [40]. However, the black-anodized specimens showed higher initial $|Z|$ values, reaching $8.17 \times 10^6 \Omega \cdot \text{cm}^2$ after 1 day of immersion, compared with $4.74 \times 10^6 \Omega \cdot \text{cm}^2$ for the clear-anodized samples. This behavior indicates a superior initial resistance against electrolyte penetration, likely associated with differences in the structure and properties of the anodic layer.

Nevertheless, after prolonged exposure, the impedance response of the black-anodized specimens decreased significantly, reaching values of $19.1 \times 10^4 \Omega \cdot \text{cm}^2$ and 1.98×10^3

$\Omega\cdot\text{cm}^2$ after 7 and 10 days, respectively. This decrease suggests that electrolyte access to the inner anodic regions progressively modified the electrochemical response of the coating. In contrast, the clear-anodized specimens exhibited a more gradual reduction in impedance, with a decrease from $3.36 \times 10^6 \Omega\cdot\text{cm}^2$ at 4 days to $1.33 \times 10^4 \Omega\cdot\text{cm}^2$ at 10 days. Moreover, the subsequent increase in $|Z|$ observed for both systems, particularly for the black-anodized specimens after 20 days ($8.31 \times 10^5 \Omega\cdot\text{cm}^2$), suggests the occurrence of partial repassivation phenomena during prolonged immersion.

Overall, the low-frequency impedance analysis confirms that the corrosion protection behavior of the anodized systems is not governed solely by the initial barrier properties but also by the evolution of the electrochemical processes occurring during immersion. The black-anodized samples provided enhanced initial protection, whereas the clear-anodized specimens exhibited a more stable long-term response after electrolyte interaction with the barrier oxide layer. Also, the black-anodized specimens exhibited a greater tendency toward repassivation during prolonged exposure.

The impedance modulus values at 0.01 Hz were also compared between the clear-, black-anodized, and pre-anodized and painted specimens (Figure 7b). The pre-anodized and painted system exhibited higher $|Z|$ values at the initial stage and during the prolonged immersion period, with an initial value of $1.83 \times 10^7 \Omega\cdot\text{cm}^2$ and maintaining values in the order of $10^6 \Omega\cdot\text{cm}^2$ after 30 days, confirming its enhanced barrier properties. In contrast, the clear- and black-anodized specimens showed a more pronounced variation in the impedance response with immersion time, indicating the progressive interaction of the electrolyte with the anodic structure. Although both anodized systems exhibited a decrease in $|Z|$ during exposure, the pre-anodized and painted condition provided a more stable and higher long-term corrosion resistance due to the additional barrier effect of the organic coating.

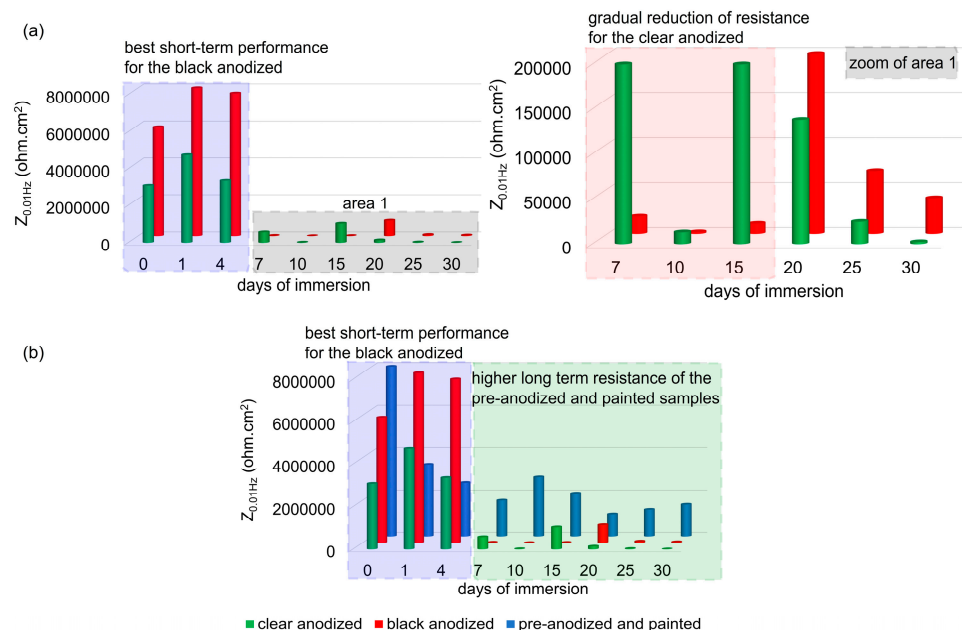


Figure 7. Evolution of the low-frequency impedance modulus (Z at 0.01 Hz) during immersion in 3.5 wt.% NaCl solution for (a) clear- and black-anodized specimens and (b) all the investigated surface treatment systems.

Figure 8 presents the variation in phase angle (θ , degrees) at a frequency of 10^5 Hz as a function of immersion time for clear-anodized, black-anodized, pre-anodized, and powder-coated samples. It also presents surface topography images of the three samples.

As reported by Mahdavian and Attar [41], the phase angle (θ) at high frequencies is a sensitive indicator of coating integrity and barrier performance. Phase angle values approaching -90° are characteristic of an intact coating exhibiting nearly ideal capacitive behavior, whereas deviations from this value indicate deterioration of the coating due to water uptake and electrolyte ingress. Based on this criterion, the as-received black-anodized, pre-anodized and powder-coated specimens exhibit superior protective performance, with phase angle values approaching -80° to -90° at 100 kHz. In contrast, the clear-anodized specimens display a significantly lower initial phase angle (approximately -60°), indicating a less pronounced capacitive response and a lower barrier efficiency. This observation suggests that the clear-anodized layer provided inferior corrosion protection compared with the black-anodized and powder-coated systems.

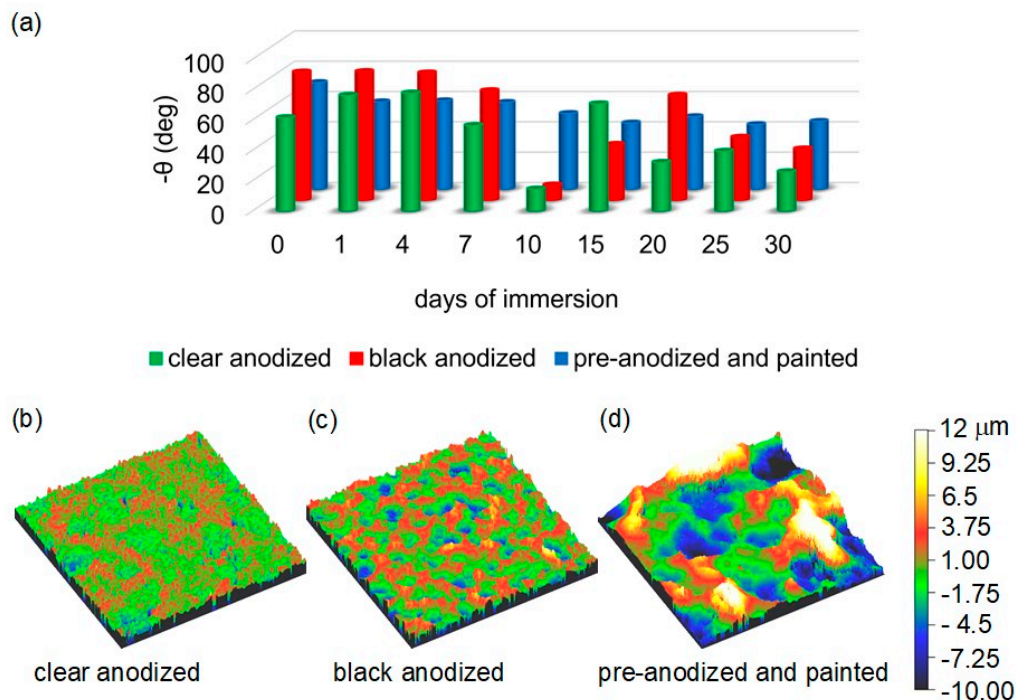


Figure 8. (a) Variation in phase angle (θ) as a function of immersion time at a frequency of 10^5 Hz for both clear- and black-anodized, and pre-anodized and powder-coated samples, together with (b–d) surface topography images corresponding to the respective samples.

As the immersion time in the saline solution increases, variations in the phase angle measured at 100 kHz are observed for both the clear- and black-anodized specimens, indicating continuous changes in the electrochemical characteristics of the anodic oxide layers. These variations are associated with the simultaneous action of degradation processes, promoted by electrolyte ingress through the porous oxide structure, and processes that may partially restore the barrier properties of the films, such as pore blocking by hydrated oxide species or corrosion products.

For the clear-anodized specimens, the phase angle progressively increases from approximately -60° to values close to -10° after 30 days of immersion. This substantial decrease in the absolute value of θ reflects a gradual loss of capacitive behavior and a significant deterioration of the protective properties of the anodic oxide film. The observed trend suggests progressive electrolyte penetration into the porous structure and a consequent reduction in the barrier effectiveness of the anodic layer.

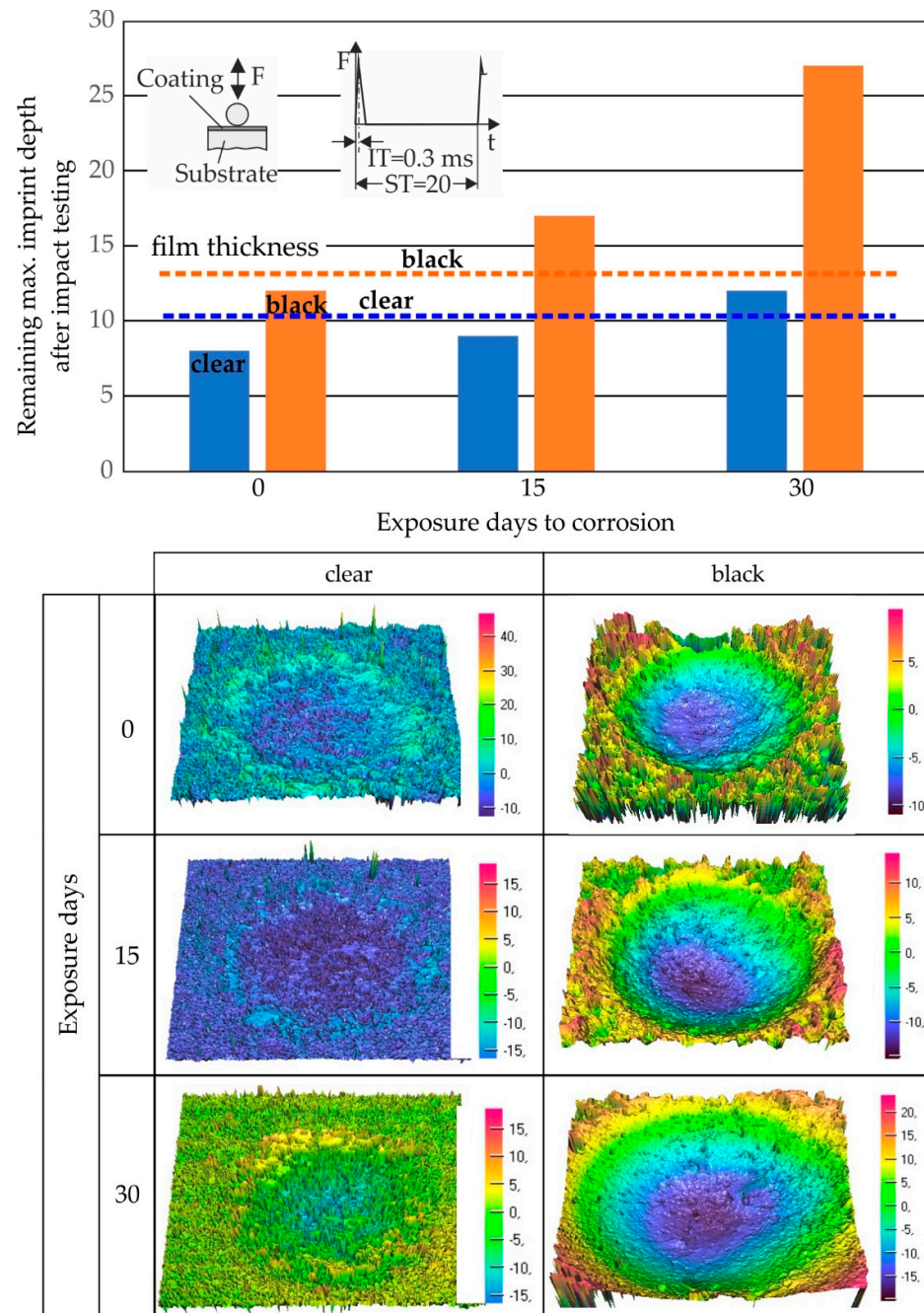
In the case of the black-anodized specimens, a marked increase in the phase angle is observed between 7 and 15 days of immersion, indicating a temporary deterioration of the oxide film. However, beyond 20 days of exposure, the phase angle decreases again, reaching values close to -50° after 30 days. This behavior suggests a partial restoration of the barrier properties of the anodic layer, resulting in a more capacitive response compared to that observed for the clear-anodized specimens.

For the pre-anodized and powder-coated specimens, a pronounced decrease in phase angle is apparent during the first day of immersion, from approximately -80° to -45° . This behavior is attributed to rapid water uptake by the organic coating and the consequent reduction in its barrier properties. Following this initial decline, only minor variations in phase angle are recorded, with values stabilizing around -50° to -55° throughout the remaining immersion period. The relatively stable response indicates that, despite the degradation of the powder coating, the underlying anodic oxide layers continue to provide effective protection by restricting electrolyte transport towards the metallic substrate and delaying the onset of significant corrosion activity.

In general, after 10 days of immersion, the phase angle exhibits its maximum deviation from -90° across all the studied cases, except for the clear-anodized samples, which show an even greater deviation after 25 days of immersion. The surface topography images reveal clear differences in surface morphology among the samples studied. The clear-anodized specimens exhibit a more uniform and homogeneously distributed oxide layer with relatively smooth surface features, indicating a more compact oxide structure. In contrast, the black-anodized samples show a more heterogeneous and rough surface morphology, with pronounced localized irregularities, suggesting increased surface non-uniformity and possible defect sites. The pre-anodized and powder-coated samples present an intermediate behavior, where the coating partially masks the underlying surface roughness but still presents localized inhomogeneities.

3.3. Impact Tests on As-Received and NaCl-Exposed Anodized and Pre-Anodized and Powder-Coated Aluminum Specimens

The upper part of Figure 9 illustrates the progressive degradation of the anodized coatings as a function of corrosion exposure time. Specifically, the residual maximum imprint depth is presented as a quantitative indicator of anodized coating failure. An increase in imprint depth reflects reduced resistance to repeated mechanical loading and, consequently, a deterioration in the structural integrity of the anodized coating. In the case of the clear-anodized specimens, up to 15 days of exposure, the residual imprint depth remains lower than the anodized coating thickness, indicating that the coating is still intact. However, after 30 days, the anodized coating is completely removed. In contrast, the black-anodized specimens exhibit significantly larger maximum imprint depths. After 15 days of corrosion exposure, the anodized coating is already fully removed under the same testing conditions. Overall, the impact resistance of the black-anodized coating is considerably lower than that of the clear-anodized coating.



Indenter ball: K05, $d=2.5$ mm, Impact force=120N, Number of impacts=5000

Figure 9. Remaining max. imprint depths after impact of clear- and black-anodized aluminum at different durations of exposure to a corrosive environment and representative surface profiles of the impact imprints, obtained using white-light confocal microscopy, of the examined cases.

The lower part of Figure 9 presents representative surface profiles of the impact imprints, obtained using white-light confocal microscopy. These scans provide detailed insight into the morphology and depth distribution of the deformation induced by cyclic impacts. Owing to the perpendicular loading applied during the impact tests, the thin coating is compressed against the substrate, resulting in the development of high contact stresses. When these stresses exceed the critical fatigue strength of the anodized coating, damage initiates, leading to coating failure. The evolution of anodized coating damage versus the number of impacts is more intensive on the black-anodized aluminum. Moreover, the results highlight

the critical influence of corrosion-induced microstructural damage on the impact wear performance of the anodized coatings, with the clear-anodized coating showing enhanced durability compared to the black-anodized coating under the examined conditions.

To explain the differences in impact behavior between the clear- and black-anodized aluminum coatings, their microstructures were examined using SEM. As shown in Figure 10, the black-anodized coating is characterized by the presence of microcracks and voids within its structure. These defects act as stress concentrators, promoting crack initiation and propagation under repeated dynamic loading. Consequently, the black-anodized coating exhibits reduced resistance to impact fatigue, leading to earlier coating failure and more rapid wear progression compared with the clear-anodized coating, which possesses a denser and more homogeneous microstructure.

Finally, it should be noted that the pre-anodized aluminum with a powder coating cannot be reliably evaluated using the applied impact testing methodology. Owing to its inherently loose and weakly bonded structure, the powder coating undergoes severe and immediate degradation under dynamic loading conditions. As a result, extensive material detachment and powder coating failure occur even at the early stages of testing, preventing the acquisition of meaningful or comparable imprint depth data.

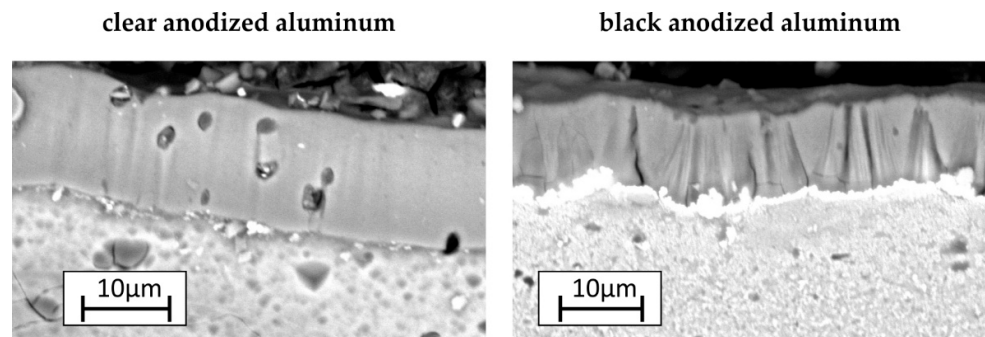


Figure 10. SEM micrographs of the examined clear- and black-anodized coatings.

4. Conclusions

This work investigates the time-dependent corrosion mechanisms governing different types of surface-treated aluminum samples immersed for prolonged periods in 3.5 wt.% NaCl solutions. In addition, the study evaluates, for the first time, the combined effects of corrosion and wear on the durability of anodized aluminum samples through dynamic impact loading tests.

In terms of corrosion, the clear- and black-anodized specimens exhibited different degradation pathways. Black anodizing provided superior initial protection during the first days of immersion, indicating a more effective resistance against early electrolyte penetration. However, its protective performance decreased significantly after prolonged exposure, suggesting progressive modification of the anodic structure and increased accessibility of the underlying regions. Clear anodizing exhibited a more stable corrosion response during intermediate immersion times, associated with a more persistent barrier oxide contribution and partial recovery of impedance during prolonged exposure, likely associated with temporary pore blocking and/or repassivation phenomena.

The pre-anodized and powder-coated system exhibited the highest resistance against corrosion initiation among the investigated treatments, as the organic coating acted as the primary barrier during the first 15 days of immersion. After longer exposure, the onset of electrochemical activity indicated progressive degradation of the protective system and electrolyte interaction with the underlying anodic layer.

Clear-anodized specimens retain coating integrity up to 15 days of exposure, as the residual imprint depth remains below the anodized coating thickness, but complete coating removal occurs after 30 days. In contrast, black-anodized specimens show higher imprint depths and complete coating loss already after 15 days under the same conditions, indicating significantly lower impact resistance compared to clear-anodized coatings. The pre-anodized aluminum with powder coating cannot be reliably evaluated by the applied impact testing method, as its weakly bonded structure leads to immediate degradation and extensive material detachment under dynamic loading, preventing meaningful imprint depth measurements.

Overall, it should be noted that when corrosive environments are combined with wear-inducing factors, the durability performance of anodized coatings must be evaluated under coupled degradation mechanisms, as single-condition assessments may not adequately represent in-service behavior.

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