



Effect of Y_2O_3 rare earth oxide on the microstructure and growth kinetics of TiO_2 -based ceramic coatings fabricated by micro arc oxidation

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ABSTRACT

This study investigates the influence of Y_2O_3 addition on the microstructure, growth kinetics, and mechanical properties of TiO_2 -based ceramic coatings formed on Ti–6Al–4V alloy through the Micro Arc Oxidation (MAO) process. The experiments were carried out using Ti–6Al–4V plates coated in silicate-based electrolytes, with and without 1 g/L Y_2O_3 , under a constant current density of 5 A/dm² and a voltage of 360 ± 20 V for different treatment times (1, 3, 7, and 10 min). The coatings were evaluated by weight gain measurements, SEM/EDS analyses, X-ray diffraction (XRD), and constant load scratch testing supported by profilometry. Results revealed that Y_2O_3 significantly accelerated coating growth during the initial stages, leading to higher weight gains and denser, less porous morphologies compared to Y_2O_3 -free coatings. XRD analysis indicated amorphous TiO_2 and anatase phases at short durations, while rutile formation became dominant with longer treatments. Kinetic modeling demonstrated that both coatings initially followed a linear reaction-controlled mechanism ($n \approx 1$), with calculated growth constants of 3.44×10^{-4} mg/mm²·s for Ti-MAO and 7.45×10^{-4} mg/mm² s for Ti- Y_2O_3 -MAO. However, after approximately 420 s, the Y_2O_3 coatings exhibited a marked reduction in growth rate due to electrical insulation effects of the thickened ceramic layer, resulting in multi-stage kinetics. Constant load scratch testing showed that Y_2O_3 incorporation reduced groove depth and width by nearly 45%, improving adhesion strength and mechanical durability. These findings confirm that rare-earth oxide addition is an effective strategy for enhancing the performance of MAO coatings on titanium alloys.

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Introduction

Micro arc oxidation (MAO) is a surface modification technique primarily applied to lightweight metals and their alloys. This technology is mainly applied on aluminum (Al), magnesium (Mg), titanium (Ti), and their alloys [1–4]. During the MAO process, the application of high voltage induces micro-plasma discharges on the metal surface, leading to the formation of a thick, hard, and ceramic like oxide layer. The resulting coating exhibits exceptionally high hardness, excellent wear resistance, superior thermal stability and good chemical stability. Additionally, the ceramic layer offers high electrical and thermal properties [2, 3, 5]. After the MAO coating process, surface roughness and micro-porosity increase; however, good adhesion and enhanced surface hardness are achieved. Nevertheless, parameters such as electrolytic composition, additive elements, applied voltage, and coating duration significantly influence the surface properties [2, 6–10].

The coating mechanism is fundamentally based on the generation of localized plasma discharges at the metal surface within the electrolyte, resulting in extremely high local temperatures (ranging from 3000 to 10000 K) and rapid oxidation of the substrate. Through instantaneous melting and solidification cycles, a ceramic-based layer is formed on the metal surface. The energy of the discharges directly affects the coating's microstructure, phase transformations, and the types of ceramic phases formed [7, 11].

The incorporation of additive particles into micro arc oxidation (MAO) coatings is an effective modification strategy that can significantly modify coating properties. During the MAO process, additive particles are introduced into the electrolyte solution and become integrated into the coating structure. These particles improve the microstructure of the coating by reducing porosity, enhancing hardness, and increasing the chemical stability of the oxide layer. In particular, rare-earth metal oxides (such as Y_2O_3 , CeO_2 , La_2O_3 , and Gd_2O_3) are among the most extensively studied additives reported in the literature [1, 12–16].

The use of rare-earth elements in surface treatments has been pursued through various methods to enhance material performance. These elements are typically deposited onto surfaces by electrophoretic deposition techniques [16–18]. However, since electrophoretic deposition is carried out under low voltages, the resulting coatings exhibit limited density and mechanical strength, thereby restricting their

overall performance improvement. Although rare-earth elements demonstrate significant advantages in surface modifications, their utilization within micro arc oxidation (MAO) processes remains relatively limited in the current literature.

Rare-earth oxides are oxides with high chemical stability and thermal resistance. Recent studies have shown that when used in MAO coatings, these oxides enhance the hardness of the coatings, increase the coating density by filling micropores, and improve corrosion resistance. Ma G. et al. [9] demonstrated that the addition of CeO_2 to MAO coatings resulted in increased density and smoothness on the surface of the coating. Yang X. et al. [18] attributed the presence of La_2O_3 and MnO_2 to improved coating compactness and interfacial adhesion in MAO-treated Mg–Li alloys. The use of Y_2O_3 in MAO coatings applied to Mg alloys has been shown to significantly reduce porosity and improve coating integrity [5]. Owing to its high hardness, high melting point (2430 °C), and excellent thermal stability, Y_2O_3 is considered a promising additive for MAO coatings [1, 14, 19].

Although the beneficial effects of rare-earth oxides in MAO coatings have been widely reported for Mg-based alloys, where corrosion protection is a primary concern, their role in MAO coatings on titanium alloys is fundamentally different. Ti–6Al–4V alloy already exhibits excellent intrinsic corrosion resistance; therefore, the motivation for applying rare-earth oxide-modified MAO coatings on Ti–6Al–4V are not corrosion protection alone, but the enhancement of surface-related functional properties such as wear resistance, coating densification, adhesion strength, and resistance to fretting and mechanical damage.

The current study presents a method to improve the surface properties of titanium alloy Ti–6Al–4V. Ti–6Al–4V alloy, known for its high specific strength, excellent corrosion resistance, and biocompatibility, is widely used in aerospace, biomedical, and automotive industries [2, 20–22]. However, its relatively low surface hardness and limited wear resistance can constrain its service life and performance in certain applications.

In practical service conditions, Ti–6Al–4V components used in aerospace fasteners and biomedical implants are frequently subjected to cyclic contact, sliding, and micro-motion, which may result in surface degradation, material loss, and debris generation. Therefore, improving the

tribological performance and mechanical durability of Ti–6Al–4V surfaces is of critical importance for these applications.

Although several studies have reported the incorporation of Y_2O_3 into MAO/PEO coatings on Ti–6Al–4V alloys, most of these works have focused on corrosion behavior and general microstructural observations [1, 5, 15]. In contrast, the present study provides a systematic and quantitative investigation of the growth kinetics and growth mechanism of Y_2O_3 -modified TiO_2 -based MAO coatings under controlled processing conditions. This work demonstrates a multi-stage growth behavior induced by Y_2O_3 addition, which has not been explicitly addressed in previous studies. Furthermore, the use of constant-load scratch testing supported by profilometry enables a direct assessment of coating mechanical integrity and densification effects. Therefore, the novelty of this study presents in clarifying the role of Y_2O_3 in modifying the kinetic evolution, discharge stabilization, and mechanical durability of MAO coatings on Ti6Al4V.

Table 1 Chemical composition (wt%) of Ti6Al4V

Ti	Al	V	Fe	O
Balance	6.3	4.1	0.3	0.2

Experimental details

MAO treatment

Ti–6Al–4V alloy plates with dimensions of 50 mm × 15 mm × 5 mm were used for the coatings. The chemical composition of the alloy is presented in Table 1. The surfaces of the specimens were cleaned with alcohol prior to the coating process. The coatings were applied using a Micro Arc Oxidation (MAO) system operating at a current density of 5 A/dm². Figure 1 represents the schematic illustration of the Micro Arc Oxidation (MAO) system used in this study. The arrows indicate the circulation direction of the electrolyte within the stainless-steel container, ensuring uniform temperature and ion distribution during the MAO process. The cooling system surrounding the electrolyte container is used to dissipate the heat generated by plasma discharges and to maintain a stable electrolyte temperature throughout the coating process. The power supply provides a controlled current density to the titanium substrate (anode), while the stainless-steel container serves as the cathode.

Group 1 samples were coated using a silicate-based electrolyte solution containing Na_2SiO_3 and KOH. For Group 2, 1 g/L Y_2O_3 nano powder (average particle size < 50 nm), dispersed as a suspension in the

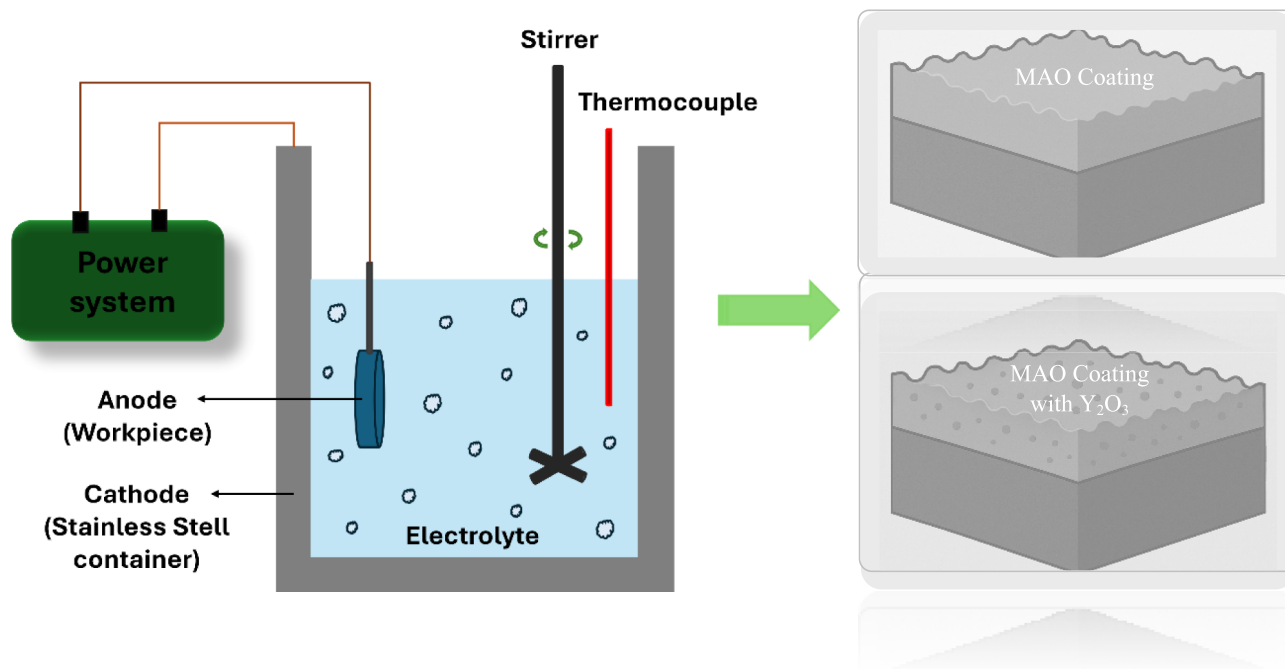


Figure 1 Schematic view of the MAO device and coating.

electrolyte, was added to the same silicate-based electrolyte prior to the coating process. A silicate-based electrolyte was selected in this study due to its well-documented ability to promote the formation of relatively thick, hard, and mechanically stable TiO_2 -based MAO coatings. Coatings for both groups were performed at varying durations (1, 3, 7, and 10 min). The coating parameters and sample codes are provided in Table 2.

Before and after coating weights of the specimens were measured using a Radwag analytical balance with a precision of 0.0001 g. The amount of coating deposited as a function of time was recorded. Surface morphology and elemental composition of the coated samples were analyzed using SEM and EDS analyses, performed with JEOL JSM-6060. The crystal structure of the alloys was characterized using X-ray diffraction (XRD, RIGAKU D/MAX/2200) with a wavelength of 1.5418 Å, over a 2θ range of 10–90° using Cu K α radiation.

Growth kinetic

The growth behavior of coatings is mostly evaluated based on the weight gain (ΔW) per unit area. In MAO coatings, the increase in coating thickness is directly related to the amount of oxide phases formed on the surface and can therefore be represented by the weight gain (ΔW) [2, 15, 23, 24]. In the literature, two fundamental kinetic models are commonly used to describe coating growth. The first of these is the linear kinetic model, which assumes that the coating grows linearly with processing time. It is defined by the equation;

$$\Delta W = k \cdot t \quad (1)$$

where, ΔW : weight gain per unit area (mg/mm^2), t : processing time (s), and k : linear growth rate constant ($\text{mg}/\text{mm}^2 \text{ s}$).

The second approach is the parabolic kinetic model, which represents cases where coating growth is limited by ion or atom diffusion. In this model, the growth behavior is expressed by the equation:

$$\Delta W^2 = k_p \cdot t \quad (2)$$

where, k_p is the parabolic rate constant with a unit of $\text{mg}^2/\text{mm}^4 \text{ s}$. This model is particularly applicable to systems where diffusion resistance becomes significant as the processing time increases, leading to a gradual slowdown in the growth rate [2, 15, 24].

Constant load scratch testing

Scratch tests were conducted on the coated samples using a CSM *Tribometer* under a load of 1 N, using a Rounded Conical Rockwell C diamond indenter. The tests were performed at a frequency of 0.16 Hz and a speed of 1 cm/s under ambient conditions. The scratch test parameters are summarized in Table 3. The constant-load configuration was intentionally selected to evaluate intra-coating damage behavior and scratch resistance without penetrating through the coating into the substrate. All scratch tests were carried out using the same load condition to ensure consistency and comparability of the results. The 2D, 3D surface profilometer was used to evaluate the size and

Table 2 MAO coating parameters according to time and electrolyte composition

Sample code		Electrolyte composition	Coating duration (minutes)	Voltage (V)
Group1	S-1	15 g/L Na_2SiO_3 + 5 g/L KOH	1	360 ± 20
	S-3	15 g/L Na_2SiO_3 + 5 g/L KOH	3	360 ± 20
	S-7	15 g/L Na_2SiO_3 + 5 g/L KOH	7	360 ± 20
	S-10	15 g/L Na_2SiO_3 + 5 g/L KOH	10	360 ± 20
Group2	Y-1	15 g/L Na_2SiO_3 + 5 g/L KOH + 1 g/L Y_2O_3	1	360 ± 20
	Y-3	15 g/L Na_2SiO_3 + 5 g/L KOH + 1 g/L Y_2O_3	3	360 ± 20
	Y-7	15 g/L Na_2SiO_3 + 5 g/L KOH + 1 g/L Y_2O_3	7	360 ± 20
	Y-10	15 g/L Na_2SiO_3 + 5 g/L KOH + 1 g/L Y_2O_3	10	360 ± 20

Table 3 Constant load scratch test parameters

Test parameter	Horizontal displacement	Total scratch length	Load
Value	1 cm/s	10 mm	1 N

geometry of the residual scratch patterns, and SEM analyses were performed to obtain high-resolution images of the residual scratch tracks.

Results and discussion

Coating morphology

Figure 2 schematically illustrates the effect of Y_2O_3 addition. The influence of Y_2O_3 on the MAO coating morphology is highly noticeable. The schematics represent stages of micro-discharge activity, oxide formation, particle transport through discharge channels, and coating densification. This figure is intended to provide a conceptual representation of the coating formation mechanism. During the MAO process, plasma discharges occurring on the surface locally generate extremely high temperatures (~ 3000 – 10000 K), which cause the oxidation of the metal substrate and the formation of oxide phases. Inert ceramic additives such as Y_2O_3 , which possess a high melting point (2430°C), become physically embedded in or dispersed throughout the molten oxide matrix on the surface as a result of the thermal effects within these discharge zones [1, 5, 11].

The presence of Y_2O_3 enhances the coating's density and compactness, improves the pore structure, and promotes the formation of a more uniform ceramic phase. However, as the treatment time increases, excessive accumulation of the additive may lead to

structural irregularities and localized melting, highlighting the need for careful optimization of both the processing time and the additive content. Ultimately, the incorporation of Y_2O_3 appears to fill surface pores and increase coating density. A more compact, homogeneous, and less porous structure was observed on the Y_2O_3 added surfaces.

Figure 3 presents the MAO coatings without Y_2O_3 after 1, 3, 7 and 10 min of treatment, respectively. Significant changes in the surface morphology of the coatings were observed depending on the duration of the micro-arc oxidation (MAO) process. At the initial stage of the process (1 min), no continuous and homogeneous ceramic oxide layer characteristic of MAO coatings were observed on the surface. Although slight surface modifications were detected, the overall surface morphology did not exhibit the typical features of a MAO generated ceramic layer, and the metallic character of the substrate was largely preserved. This can be attributed to the insufficient plasma discharges within such a short processing time. When the duration was increased to 3 min, irregular but homogeneously distributed micropores and a thin oxide layer were observed on the surface. At 7 min, the surface morphology changed significantly, exhibiting a denser porous structure and the formation of nodular oxide accumulations. This indicates the transition of the MAO process into an active phase, characterized by the formation of a thick and porous ceramic layer. Finally, after 10 min of treatment, an increase in pore

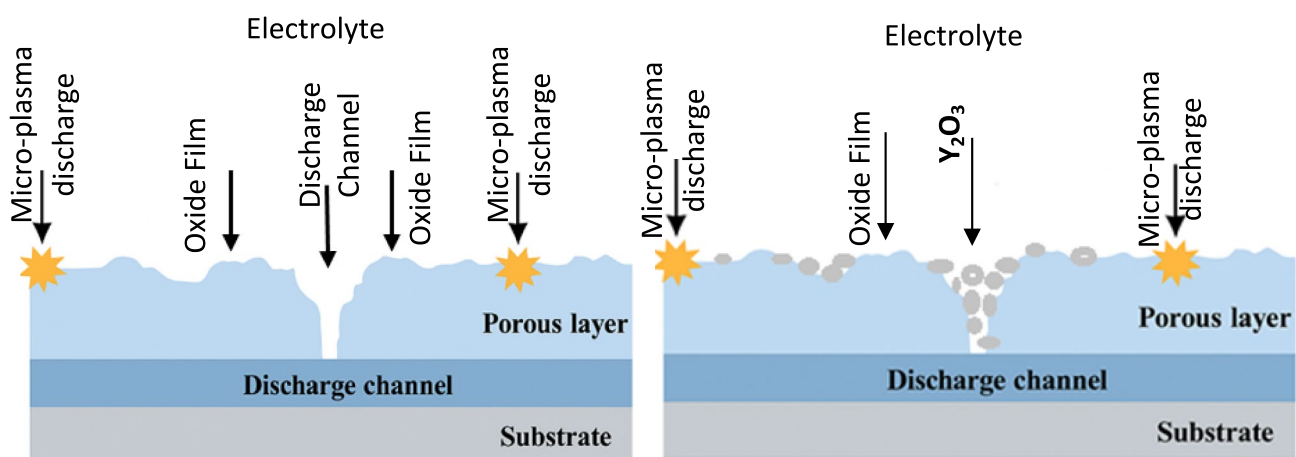


Figure 2 Schematic representation of consecutive stages of Y_2O_3 incorporation into the MAO coating, illustrating micro-discharge activity and coating densification.

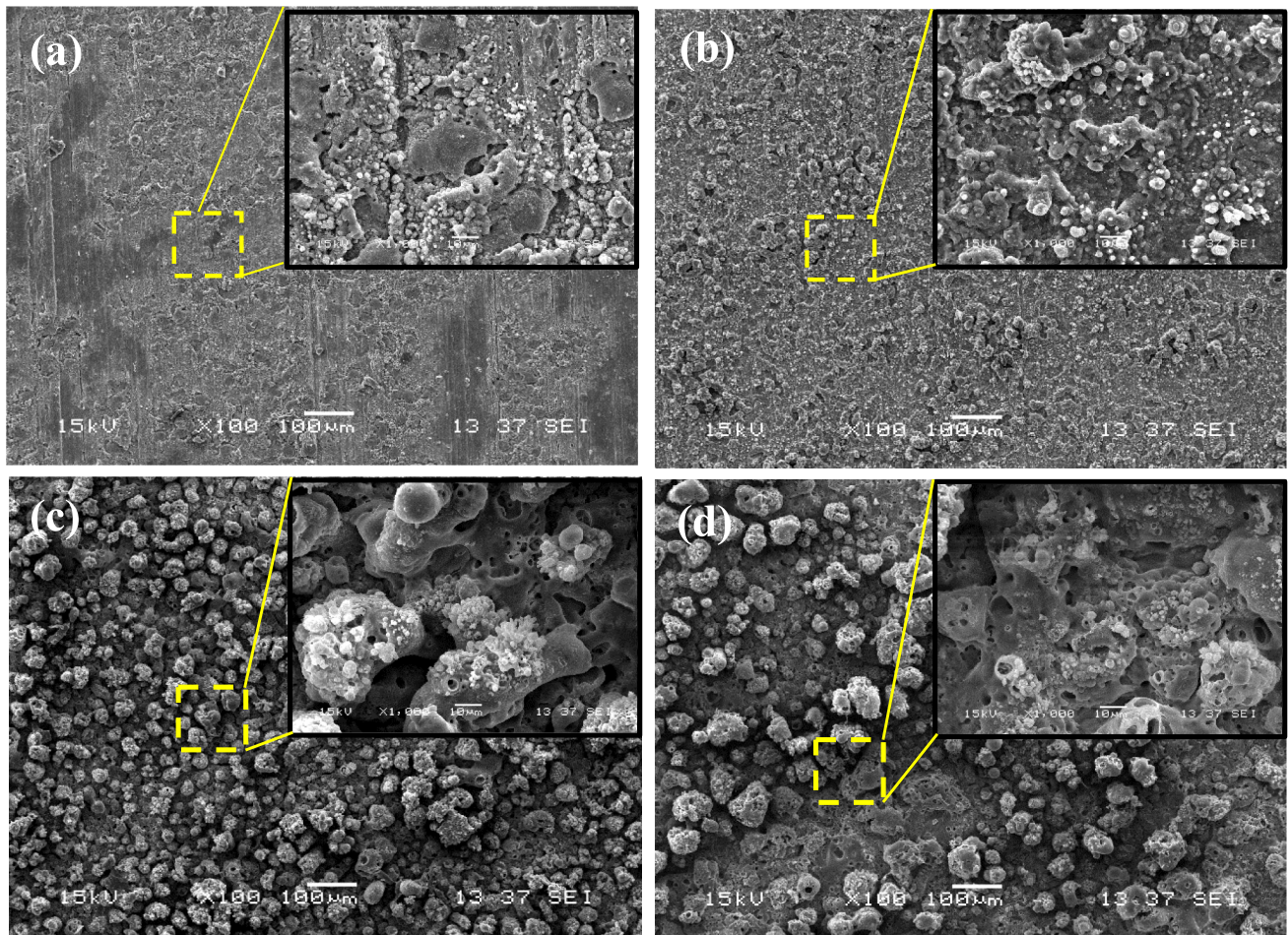


Figure 3 SEM surface morphologies of MAO coatings produced in silicate-based electrolyte without Y_2O_3 at different treatment times: **a** 1 min (S-1), **b** 3 min (S-3), **c** 7 min (S-7), and **d** 10 min (S-10).

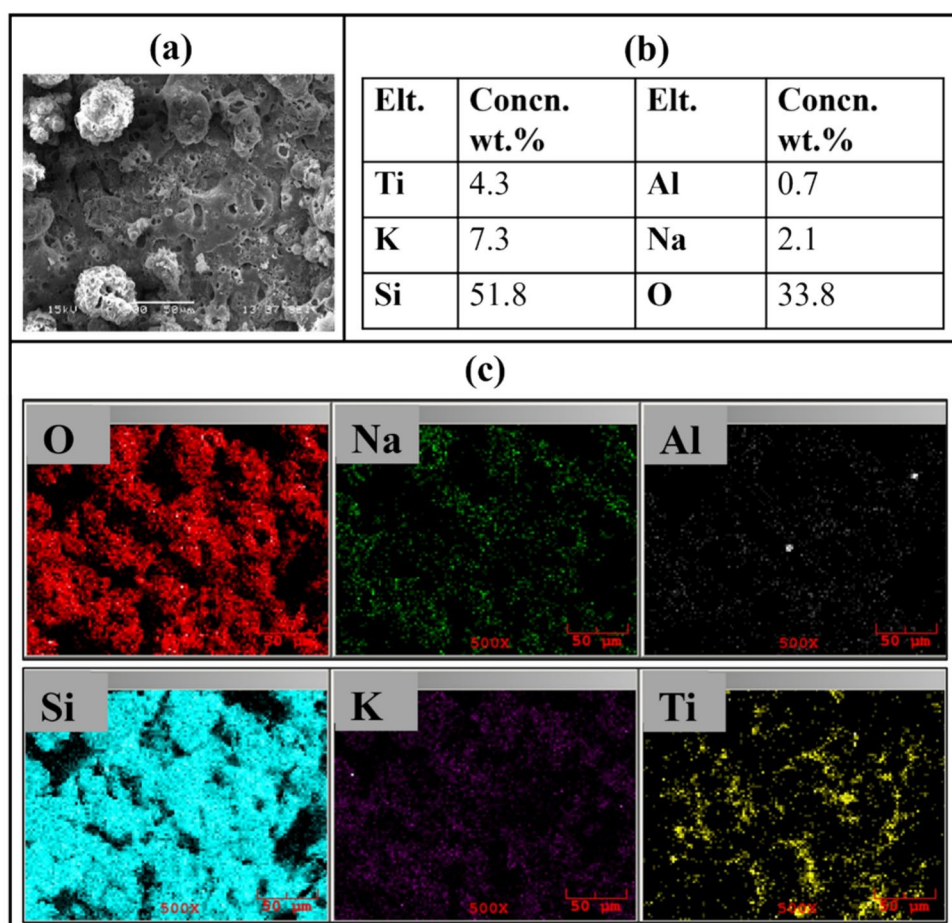
size, surface irregularities, and the presence of re-solidified molten phase residues in certain regions were detected. This suggests that prolonged processing leads to local melting and phase agglomeration on the surface. Overall, increasing the treatment duration enhances the coating thickness and porosity; however, excessively long processing times may negatively affect the structural integrity of the coating.

Figure 4 gives the surface morphology (SEM), elemental composition (EDS), and elemental distribution maps of the MAO coating produced in silicate-based electrolyte without Y_2O_3 . The SEM image reveals a porous structure with nodular features. EDS analysis indicates a Si and O rich coating with minor amounts of Ti, K, Na, and Al. Elemental

mapping confirms the homogeneous distribution of O, Na, Al, K, and Ti throughout the coating surface.

Figure 5 presents the MAO coatings with Y_2O_3 after 1, 3, 7 and 10 min of treatment, respectively. The SEM observations clearly demonstrate the influence of Y_2O_3 incorporation on the micro-arc oxidation (MAO) process. After 1 min of treatment, the surface exhibited a relatively high density of fine particles irregularly distributed, along with the initial formation of pore. This morphology suggests that Y_2O_3 particles rapidly began to adhere to the surface; however, the coating layer had not yet fully developed at this stage. At 3 min, an increase in particle density was observed, and the surface became noticeably rougher. With a treatment duration of 7 min, the effect of Y_2O_3 became more pronounced; thicker, denser, and interwoven nodular structures formed on the surface, while the

Figure 4 **a** SEM image, **b** EDS spectrum, and **c** elemental distribution maps of O, Na, Al, Si, K and Ti for the S-10 MAO coating produced without Y_2O_3 .



porosity decreased, resulting in a more compact coating morphology. These findings indicate that Y_2O_3 promotes pore filling and enhances coating densification. After 10 min of treatment, the surface displayed large nodular features, along with re-solidified molten phase residues and localized agglomerations of nodular structures. Overall, the addition of Y_2O_3 positively contributes to the coating structure by increasing densification, reducing porosity, and supporting the formation of a particle-reinforced ceramic phase. However, excessive processing times may lead to structural irregularities and undesirable accumulation of molten phase residues.

Figure 6 gives the surface morphology (SEM), elemental composition (EDS), and elemental distribution maps of the MAO coating produced in silicate-based electrolyte containing Y_2O_3 . The SEM image shows a porous structure with nodular growths typical of MAO coatings. EDS analysis indicates a Si and O rich coating with notable amounts of Y (8.4 at. %), confirming the incorporation of Y_2O_3 into the oxide

layer, along with Ti, K, and Na in smaller proportions. Elemental mapping illustrates the homogeneous distribution of O, Si, and K across the coating surface, while Ti and Y are dispersed in localized regions. The results demonstrate that Y_2O_3 addition contributed to the densification of the coating by filling micropores and enhancing compositional uniformity.

The cross-sectional morphology was discussed based on the SEM and EDS analyses presented in Fig. 7, which was positioned after this section for clarity. Figure 7 presents cross-sectional SEM images and the corresponding EDS analyses of MAO coatings produced without Y_2O_3 (S-7) and with Y_2O_3 addition (Y-7). The cross-sectional SEM image of the S-7 sample reveals an oxide layer with a thickness of approximately 8–9 μm , exhibiting a markedly heterogeneous and porous morphology. Irregular pore distribution, locally interconnected voids, and re-solidified molten regions are observed throughout the coating thickness. This morphology indicates that the rapid melting solidification cycles induced by intense micro

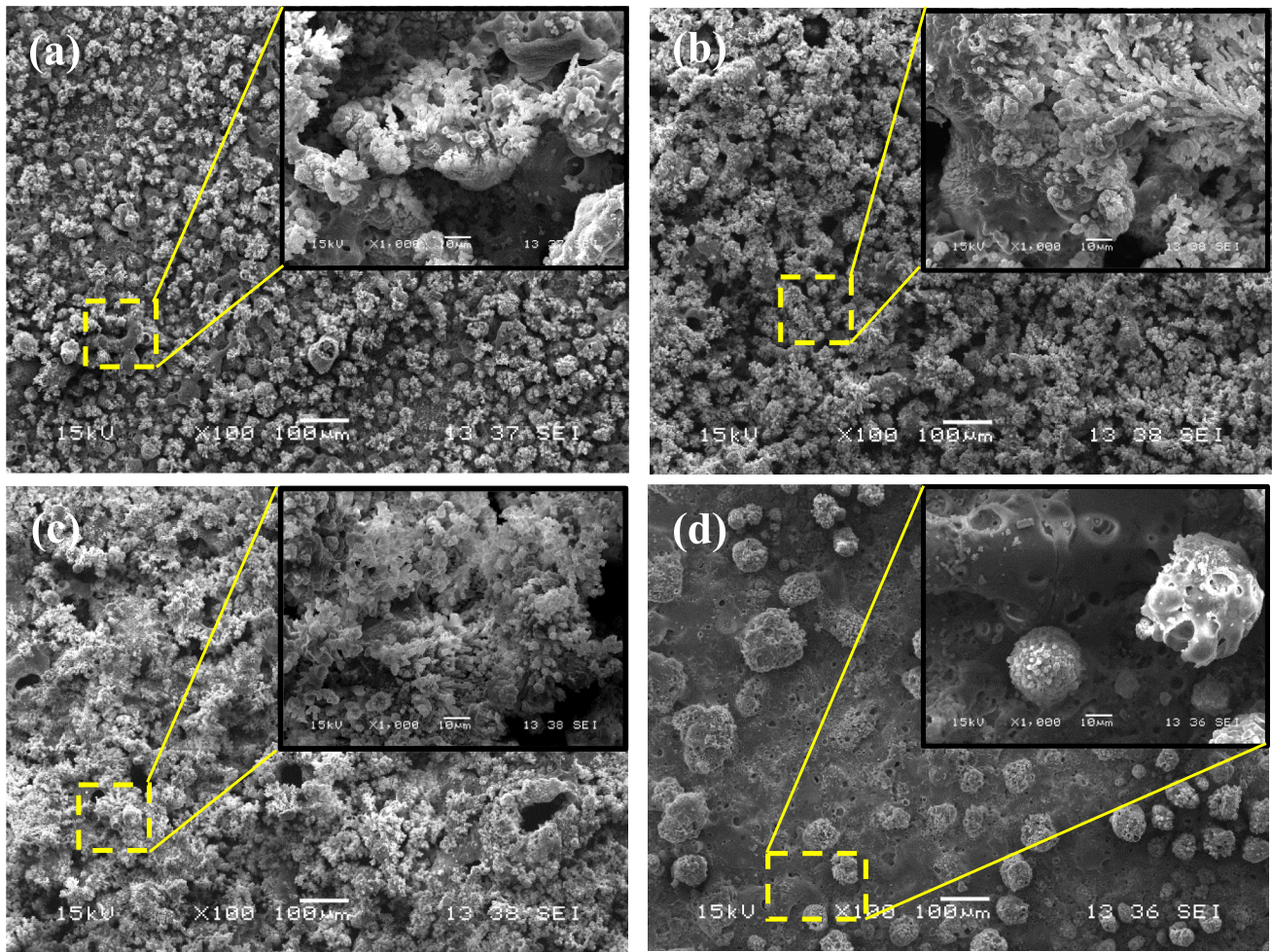


Figure 5 SEM surface morphologies of MAO coatings produced in silicate-based electrolyte with Y_2O_3 addition at different treatment times: **a** 1 min (Y-1), **b** 3 min (Y-3), **c** 7 min (Y-7), and **d** 10 min (Y-10).

discharge activity during the MAO process led to structural discontinuities within the coating.

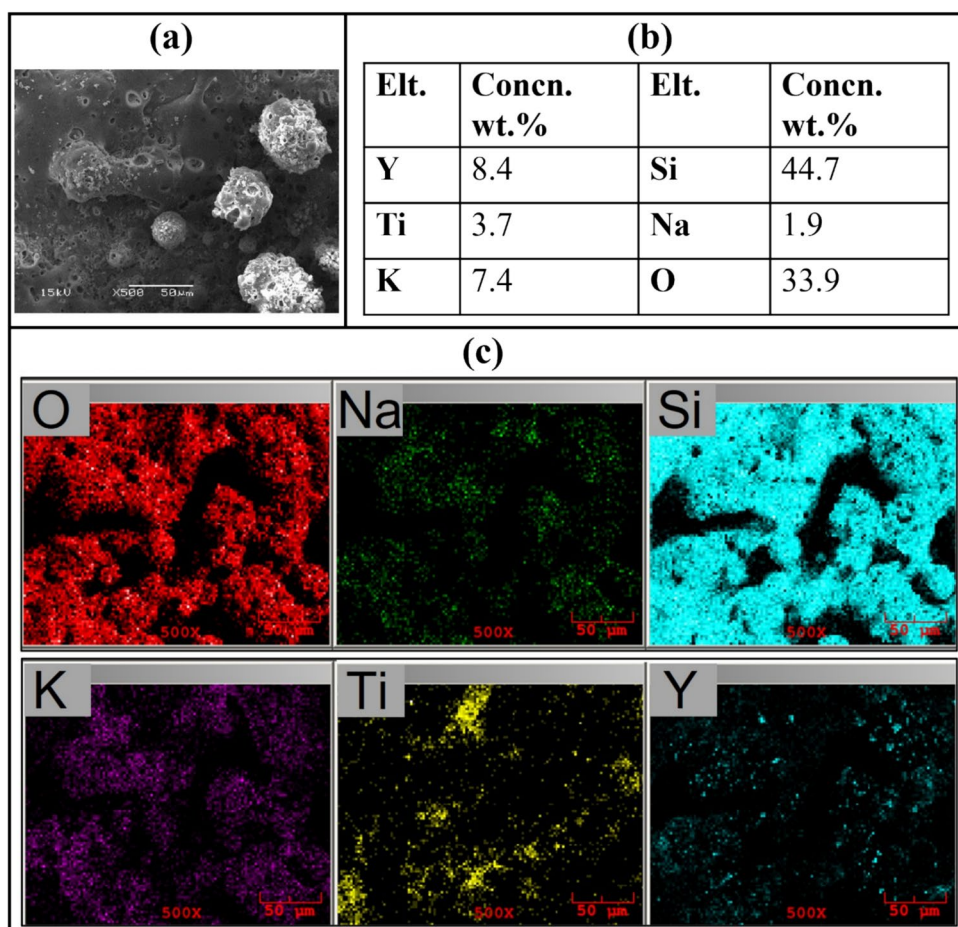
In contrast, the cross-sectional SEM image of the Y-7 sample reveals a more homogeneous, compact, and continuous coating structure. With the incorporation of Y_2O_3 , the coating thickness becomes more uniform, large pores are significantly suppressed, and the interfacial integrity is markedly improved. The smoother and more uniform contact between the coating layer and the substrate indicates that the Y_2O_3 addition stabilizes oxide growth during the MAO process and promotes densification within the coating matrix.

The EDS analyses and spectra confirm that the coatings in both samples are primarily enriched in Ti and O. In the Y-7 sample, the detection of Y within the coating layer indicates the successful incorporation of Y_2O_3 particles into the oxide matrix. The dispersed

distribution of Y throughout the coating suggests that Y_2O_3 is not only accumulated on the surface, but is instead transported into the coating volume through micro discharge channels. This observation is consistent with the particle assisted densification mechanism reported in the literature [25, 26].

Figure 8 presents cross sectional SEM images and the corresponding EDS analyses of coatings formed without Y_2O_3 (S-10) and with Y_2O_3 addition (Y-10) under extended MAO treatment conditions. The cross-sectional SEM image of the S-10 sample indicates that, despite the increased coating thickness, the structure remains markedly heterogeneous, porous, and irregular. Large pores, interconnected voids, and re-solidified molten regions are observed throughout the coating thickness. This morphology suggests that the increased micro discharge energy associated with

Figure 6 **a** SEM image, **b** EDS spectrum, and **c** elemental distribution maps of O, Na, Si, K, Ti and Y for the Y-10 MAO coating.



prolonged MAO treatment enhances structural discontinuities rather than stabilizing coating growth.

The EDS analyses confirm that the S-10 coating is primarily enriched in Ti and O; however, pronounced compositional fluctuations are detected across the coating thickness. This compositional heterogeneity indicates the presence of regions with non-uniform phase distribution and density, which may limit the intra-coating mechanical stability.

In contrast, the cross-sectional SEM image of the Y-10 sample exhibits a more compact, continuous, and structurally integrated coating, despite the extended MAO treatment duration. With the addition of Y_2O_3 , large pores are effectively suppressed, and a finer and more homogeneous microstructure is clearly observed within the coating matrix. The more uniform and more continuous morphology of the coating-substrate interface indicates that the incorporation of Y_2O_3 plays a regulatory role in oxide growth kinetics during the MAO process. The detection of Y in the EDS spectra of the Y-10 sample confirms the successful incorporation

of Y_2O_3 particles into the oxide layer via micro discharge channels. The distribution of Y together with Ti and O throughout the coating matrix suggests that Y_2O_3 does not just form a superficial phase, but instead acts as a structural contributor that promotes coating densification. This homogeneous chemical distribution directly contributes to enhanced intra coating densification and improved microstructural stability.

The EDS analyses and elemental mapping results confirmed the presence of yttrium within the MAO coatings produced with Y_2O_3 addition. The localized distribution of Y signals, particularly in nodular and dense regions, may be Y_2O_3 that is incorporated mainly as physically embedded particles. This observation is consistent with the rough surface morphology of MAO coatings and supports a particle-assisted densification mechanism.

Figure 9 presents the XRD analyses of MAO coated with and without Y_2O_3 samples as a function of increasing MAO processing time. In the titanium sample subjected to MAO treatment for 1 min

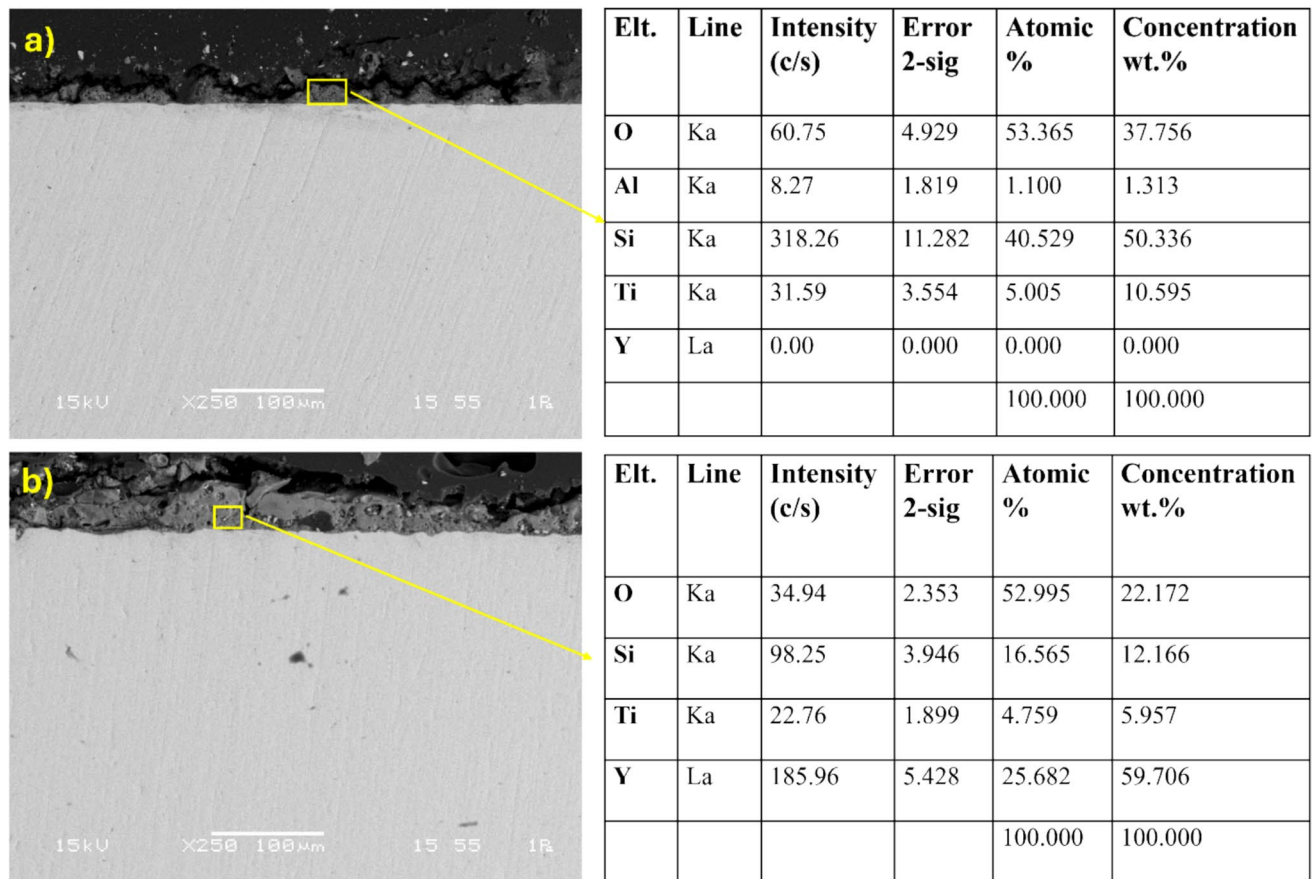


Figure 7 Cross-sectional SEM images and corresponding EDS line analyses of MAO coatings formed at 7 min: **a** S-7 (without Y_2O_3) and **b** Y-7 (with Y_2O_3).

(S-1) without Y_2O_3 addition, the wide peak observed in the $10\text{--}30^\circ$ 2θ range indicates the presence of an amorphous phase. Additionally, diffraction peaks corresponding to α -Ti and anatase (TiO_2) were identified. The presence of α -Ti peaks originating from the substrate suggests that the coating has not yet fully covered the underlying surface or that the coating is relatively thin. With increasing treatment time, partial crystallization becomes evident; however, the surface still remains largely amorphous in nature.

In the S-10 sample, a wide and intense peak is clearly observed in the $10^\circ\text{--}30^\circ$ two theta (2θ) range. This wide peak is characteristic of an amorphous structure and indicates the presence of disordered atomic arrangements on the coating surface rather than well-defined crystalline structures. This phenomenon is attributed to the rapid solidification under the high-temperature and short duration conditions typical of the micro arc oxidation process, which prevents atoms from organizing into a crystalline lattice.

In addition, anatase (TiO_2) and rutile (TiO_2) phases are also detected in the XRD pattern. While anatase is a metastable crystalline form typically formed at lower temperatures, rutile is a more stable phase that develops at higher temperatures [6, 12, 21, 27]. The existence of both phases suggests that the coating consists of a mixture of crystalline and amorphous regions. Furthermore, diffraction signals corresponding to the metallic Ti substrate are also present in the pattern, indicating that reflections from the underlying titanium surface are still detectable.

In the S-10 diffractogram, both a more pronounced amorphous character and the presence of the rutile phase were observed. This indicates that, with increasing treatment time, anatase gradually transforms into rutile. This transformation is determined by higher discharge intensity and elevated local temperatures generated during the MAO process [27–31]. In contrast, in the S-1, S-3 samples, only the anatase phase is present, with no evidence of rutile formation. This

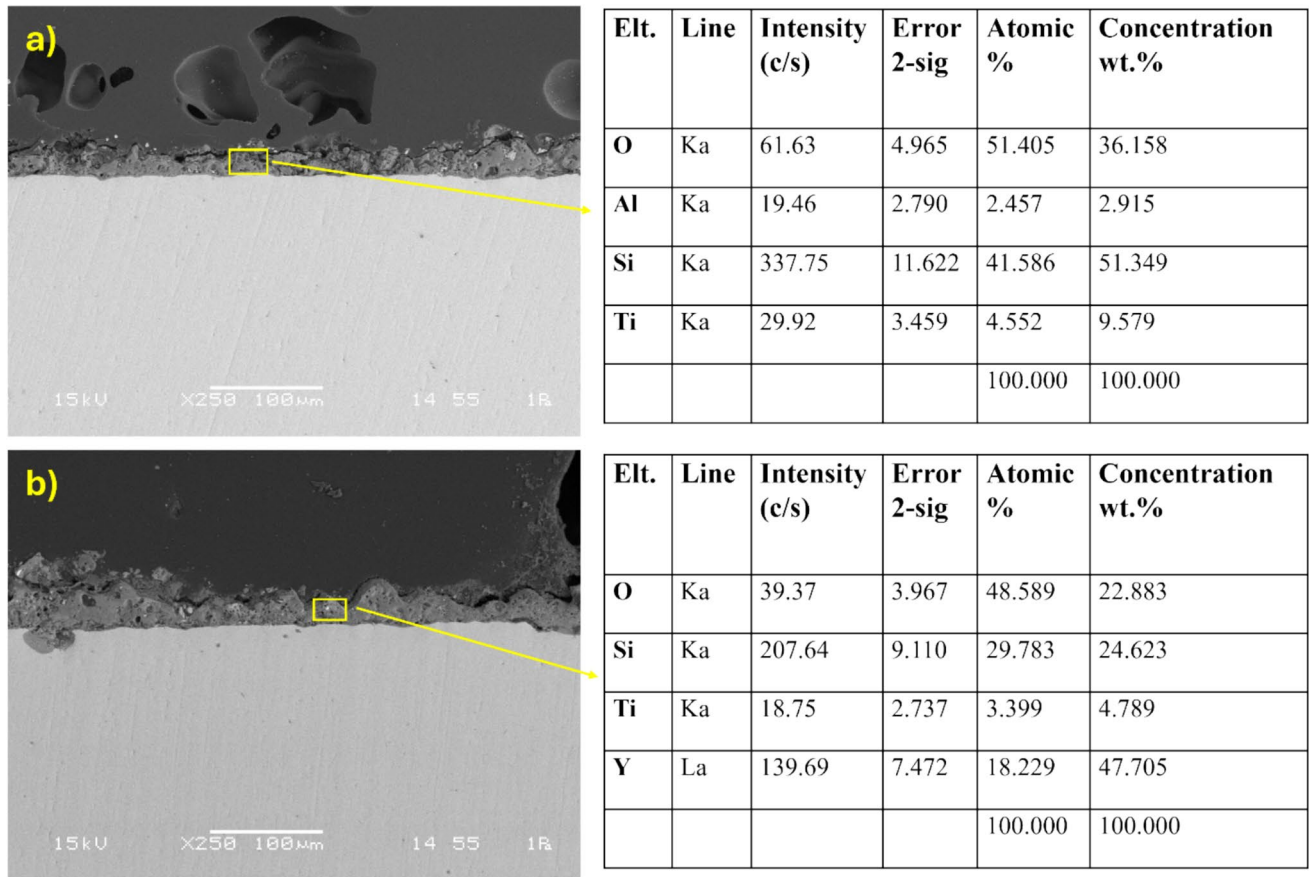


Figure 8 Cross-sectional SEM images and corresponding EDS line analyses of MAO coatings formed at 10 min: **a** S-10 (without Y_2O_3) and **b** Y-10 (with Y_2O_3).

suggests that, at shorter processing durations, the temperature and ionic mobility are insufficient to drive the crystallographic transformation from anatase to rutile.

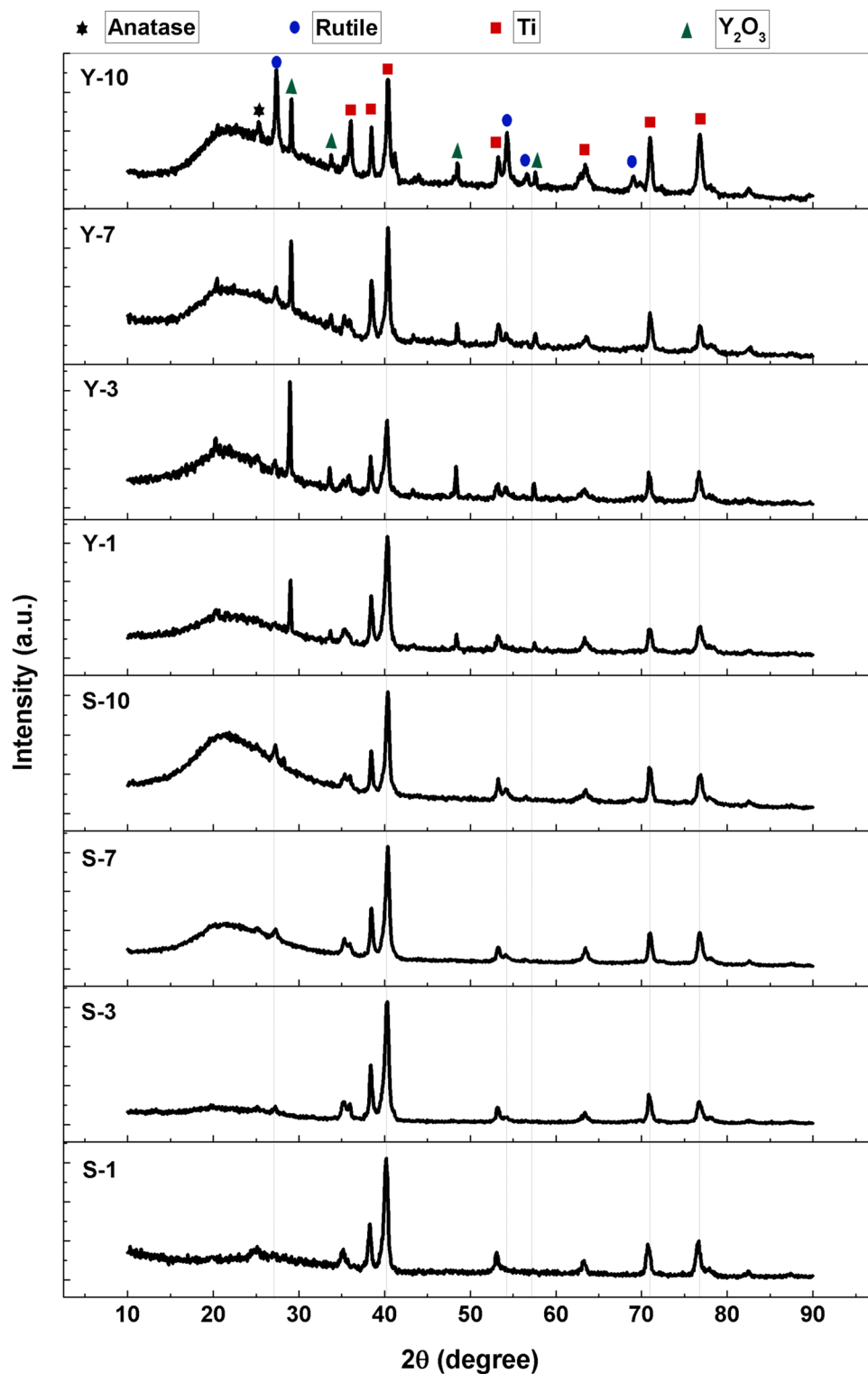
The formation of amorphous TiO_2 occurs due to the high surface temperatures (~ 3000 – 10000 K) and rapid cooling (fast solidification) conditions generated during the micro arc oxidation process. As a result of plasma discharges, localized melting takes place on the surface, and this molten phase solidifies rapidly once the discharge ceases or the process is stopped. During this rapid cooling, atoms do not have sufficient time to arrange into a crystalline structure, leading to the formation of a disordered, amorphous structure instead [32–34].

Especially, high-temperature and short duration MAO processes, although a molten phase may form on the surface momentarily, the cooling rate is extremely high. Thus, the atoms solidify in a disorganized and non-crystalline manner, resulting in the

formation of an amorphous phase. This rapid solidification process does not allow sufficient time for atomic rearrangement. As a result, the atoms solidify in a disordered and irregular manner, leading to the formation of an amorphous structure. Therefore, in high energy and sudden cooling systems in the MAO process, insufficient ion mobility becomes a major obstacle to crystallization, leading to the formation of amorphous oxide phases.

Studies [23, 35–37] have shown that during the MAO process, an amorphous TiO_2 layer forms at the interface between the coating and the substrate, and the growth of this layer is attributed to diffusion based reactions between O^- ions and Ti^{+4} ions. KOH completely dissociates in aqueous solution, producing a high concentration of OH^- (hydroxide anions) and K^+ (potassium cations). During the MAO process, OH^- ions interact with plasma discharges and combine with Ti^{+4} ions on the surface, contributing to the formation of TiO_2 .

Figure 9 XRD patterns of Ti-MAO coatings produced with and without Y_2O_3 at different treatment times. Identified phases include amorphous TiO_2 (at $10\text{--}30^\circ$), anatase TiO_2 (A), rutile TiO_2 (R), and α -Ti substrate peaks (Ti).



In the analysis of the coated samples with Y_2O_3 , both wide peaks characteristic of amorphous structures and sharp peaks corresponding to crystalline phases were observed on the surface. Although Y_2O_3

exhibits low solubility in pure water, it can decompose into ionic form under the high temperature and plasma discharge conditions of the MAO process. Due to its high charge density, Y^{+3} ions tend to become

physically embedded in the negatively charged oxide phases on the surface. This behavior increases the coating density and reduces porosity [5, 14, 32, 33].

On the other hand, O^{2-} ions act as the primary oxygen source during coating growth and react with Ti^{4+} ions to form rutile and anatase phases. The amorphous TiO_2 layer formed at the coating/substrate interface is mainly generated through diffusion-based reactions between Ti^{4+} and O^- ions. During the MAO process, the high temperatures generated in the micro-arc regions lead to partial melting of both the metal and electrolyte components and promote intense ionic motion in these zones. In this environment, O^- ions diffuse to the surface and react with Ti^{4+} ions to initially form an amorphous TiO_2 layer. Without sufficient time or energy, crystallization cannot occur [23].

Therefore, at early processing stages or under low additive concentrations, crystal growth remains incomplete, and the resulting TiO_2 phase exhibits amorphous characteristics. The broad peak observed in the $10\text{--}30^\circ 2\theta$ range in the XRD pattern indicates the presence of amorphous TiO_2 . This amorphous character is attributed to rapid cooling, high temperature, and insufficient ionic rearrangement. The presence of Y_2O_3 may disrupt heat and ion distribution within the system, inhibiting crystal growth and thus increasing the proportion of amorphous structure. In addition, the presence of sharper and more intense peaks confirms the simultaneous development of crystalline phases within the system.

Kinetic study and growth mechanism

In the MAO coating process, the formation of the coating occurs through plasma discharges generated under high temperature and high voltage conditions. During this process, the ceramic layer formed on the surface exhibits a complex kinetic mechanism in terms of both physical growth behavior and chemical reactions. In this kinetic study, the MAO coating kinetics on titanium surfaces with different additive conditions (with and without Y_2O_3 addition) were evaluated using weight gain data.

The weight gain of MAO coated with and without Y_2O_3 samples was compared over time, and the coating growth kinetics were modeled based on these experimental data. The results revealed that weight gain increased with processing time in both groups; however, this increase was more pronounced in the Y_2O_3 containing samples. Nevertheless, after the 7th

minute, a decrease in coating was observed. This clearly demonstrates the accelerating effect of Y_2O_3 on coating formation at the initial stages. However, as the ceramic based coating layer gradually accumulates on the surface over time, a decrease in conductivity is observed, leading to a slowdown in the weight gain of the coating (Fig. 10). After 10 min of coating, weight increases of $\sim 0.2 \text{ mg/mm}^2$ and $\sim 0.44 \text{ mg/mm}^2$ were observed for with without Y_2O_3 samples, respectively. The increase in coating mass with the addition of reinforcing elements is consistent with the findings reported by Guo Y.P. et al. [14] and Li H. et al. [5], who also observed similar trends in their studies.

The coating kinetic behavior is evaluated using Eq. (3) [15]:

$$\Delta W^n = k_p \cdot t \quad (3)$$

where; ΔW represents the specific mass change normalized by unit surface area, n is the reaction rate exponent ($n = 1$ for linear kinetics, $n = 2$ for parabolic kinetics), k_p is the kinetic rate constant, t indicates the time [15].

Logarithmic weight gain–logarithmic time ($\log W - \log t$) curves were generated using the mass gain data of Ti-MAO and Ti- Y_2O_3 -MAO coatings (Fig. 10). Regression analyses applied to these curves were used as the basis for determining which kinetic model the coating growth followed. Using these curves, the reaction rate exponent (n) values were calculated. The n values for both curves were found to be approximately 1. Therefore, it was assumed that both coatings exhibit linear behavior, and the subsequent kinetic calculations were performed accordingly. Based on the linear model, $\Delta W - t$ curves were generated from the weight gain data of Ti-MAO and Ti- Y_2O_3 -MAO coatings. To describe the weight gain behavior, the curves with higher adjusted R^2 values were taken into consideration.

The Ti-MAO coating exhibited a high degree of linear conformity in the $\Delta W - t$ graph (Adj. $R^2 \approx 0.9883$), suggesting that the process is reaction-controlled. On the other hand, the Ti- Y_2O_3 -MAO coating showed a lower degree of conformity to the linear model (Adj. $R^2 \approx 0.8654$). This indicates that the presence of Y_2O_3 makes the coating growth mechanism too complex to be explained by a single kinetic model. Y_2O_3 may influence the intensity of plasma discharges by altering the electrical conductivity, and it can also modify the structure of the resulting oxide phases. This can

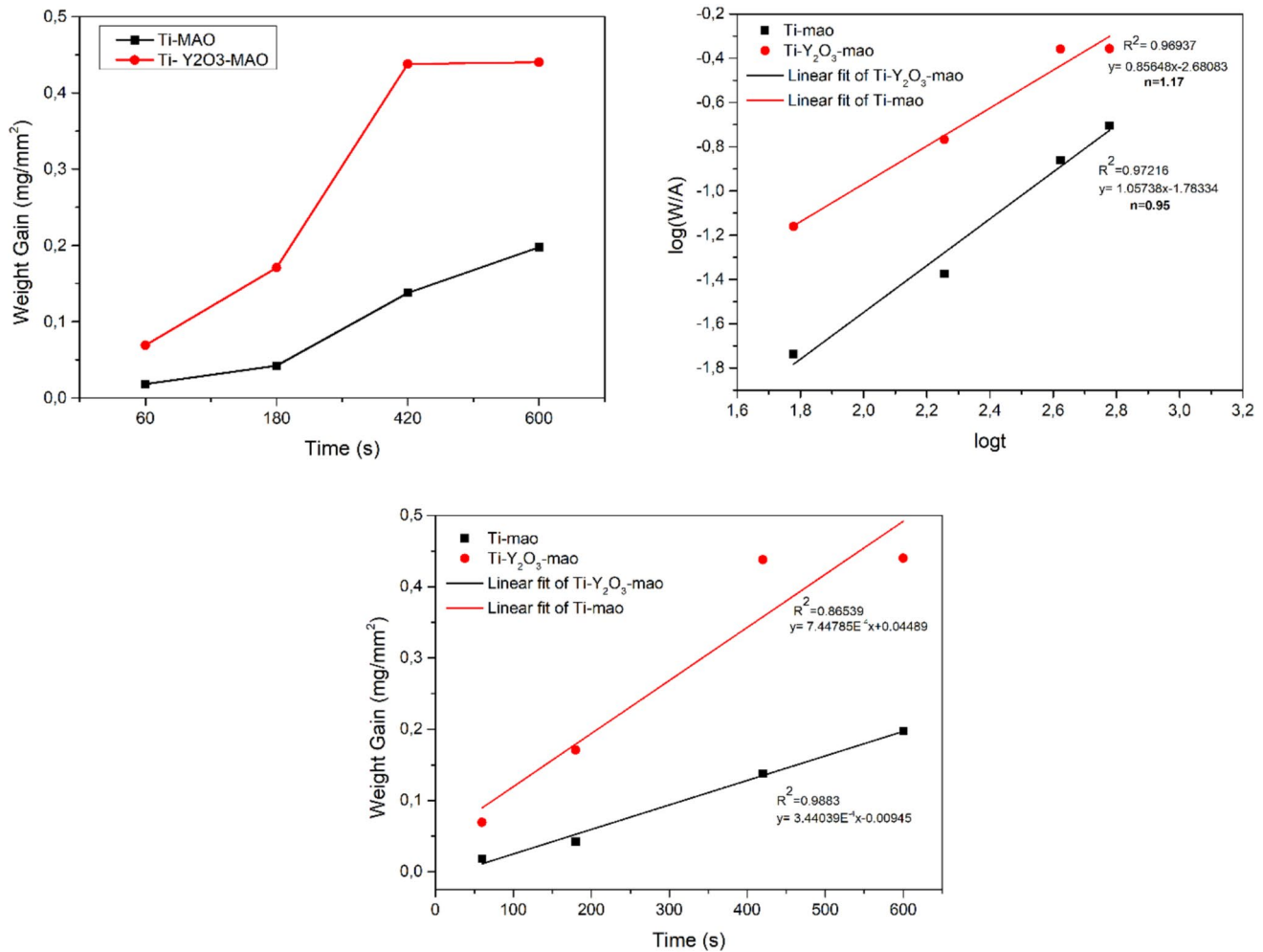


Figure 10 Weight gain, logarithmic transformation, and linear kinetic model fitting of Ti-MAO and Ti-Y₂O₃-MAO coatings as a function of processing time.

transform the growth process into a multi-stage mechanism. Additionally, in samples coated with Y₂O₃ via MAO, the stabilization of weight gain after the 420th second suggests that the thickening coating forms an electrical barrier, which limits discharge activity and subsequently slows down the coating growth.

In the Y₂O₃ coating samples, the correlation was relatively weak ($R^2 \approx 0.8654$), indicating that the coating growth is too complex to be explained by a simple diffusion mechanism. Therefore, the correlation was reevaluated for the initial stage of growth, and a significantly higher value ($R^2 \approx 0.99193$) was obtained. This suggests that the Y₂O₃ containing coating initially follows the linear model, but as time progresses, the additive complicates the coating mechanism, resulting in a multi-stage growth process that cannot be explained solely by reaction or diffusion control. This

complexity can be particularly attributed to factors such as the reduction in electrical conductivity and the limitation of plasma discharge activity as the coating layer becomes thicker over time.

In this context, the kinetics of the Ti-Y₂O₃-MAO coating process cannot be explained only as diffusion controlled or reaction controlled, unlike classical oxidation processes. Numerous variables such as plasma discharge intensity, surface temperature, electrostatic field distribution, and the influence of alloying elements play a role in determining the coating kinetics. It should be noted that real-time current–time or voltage–time evolution curves were not recorded during the MAO process in the present study. The influence of Y₂O₃ addition on plasma discharge behavior and electrical response was discussed indirectly based on coating growth kinetics, weight gain trends, and

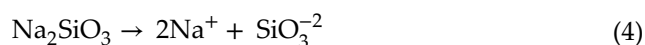
microstructural observations. Although these indirect indicators provided meaningful insight into discharge stabilization and coating evolution, future studies would allow a more direct correlation between additive-induced discharge behavior and coating growth mechanisms.

Table 4 presents the growth rate constants (K_p) calculated based on the weight gain data for both Ti–MAO and Ti–Y₂O₃–MAO coatings. These constants were obtained according to the linear (reaction controlled) kinetic model. According to this model, the K_p value of the Y₂O₃ containing coating (7.44785×10^{-4} mg/mm² s) is significantly higher than that of the Y₂O₃ free coating (3.44039×10^{-4} mg/mm² s), indicating that surface reactions proceed more rapidly in the presence of Y₂O₃. This result shows that the addition of Y₂O₃ significantly enhances the coating growth rate during the MAO process, likely due to its influence on plasma discharge behavior and the formation of a denser ceramic layer.

The addition of Y₂O₃ promotes faster and more compact coating growth by accelerating the formation of oxide phases in the high temperature plasma discharge regions. As mentioned earlier, due to the high melting point of the rare earth oxide, it becomes physically embedded in or dispersed throughout the molten oxide matrix within the discharge zones and on the surface, thereby contributing to the observed weight gain. Therefore, the optimization of parameters such as additive content and processing time is of critical importance for ensuring coating quality and structural integrity. However, as time progresses (after 420 s), it has been observed that in Y₂O₃ containing coatings, the mechanism becomes more complex and the coating growth rate slows down ($K_p \approx 1.16691 \times 10^{-5}$). This can be attributed to the decrease in electrical conductivity and the limitation of plasma discharge activity as the MAO coating layer gradually thickens over time (Fig. 11).

Rate constant (K_p) is closely associated with the efficiency of ion transfer during the MAO process. In

the experimental study, the electrolyte solution consisted of 15 g/L Na₂SiO₃, 5 g/L KOH, and 1 g/L Y₂O₃. Upon dissolution in aqueous standard, these components undergo ionization, generating various cations and anions that contribute to the coating formation during the micro arc oxidation process. As a result of these reactions, sodium silicate (Na₂SiO₃) dissociates into sodium (Na⁺) cations and silicate (SiO₃²⁻) anions. Potassium hydroxide (KOH), being a strong base, completely dissociates in water. This reaction introduces potassium (K⁺) cations and hydroxide (OH⁻) anions into the solution, thereby enhancing its alkaline character. Yttrium oxide (Y₂O₃), on the other hand, exhibits limited solubility under normal conditions; however, under the high temperatures and plasma discharges generated during the MAO process, it may undergo ionic dissociation within the molten oxide phase on the surface. The electrolyte dissociation reactions are given below;



The formation and growth of oxide coatings are strongly influenced by the transformation of oxygen containing anions particularly (O²⁻, OH⁻, and SiO₃²⁻) through the discharge channels. Therefore, a higher K_p value generally reflects more efficient ion movement and accelerated oxide formation. In this context, the significantly higher K_p values observed for Y₂O₃ containing coatings are attributed to enhanced ionic conductivity and more stable discharge behaviour during the early stages of oxidation, facilitating rapid oxide formation. Based on cross-sectional SEM/EDS analyses, yttrium is detected as localized Y rich regions within the coating, indicating that Y₂O₃ derived species are mainly incorporated via physical embedding or encapsulation within the oxide matrix, rather than uniform ionic diffusion. This incorporation contributes to coating densification and improved structural integrity. Accordingly, K_p reflects not only the coating growth rate but also serves as an indirect indicator of transport efficiency within the MAO system. At prolonged treatment times, as the ceramic layer thickens and becomes increasingly insulating, the electrical conductivity of the surface decreases, plasma discharges weaken, and the coating growth rate slows down. As a result, deviation from linear kinetic behavior

Table 4 Linear growth rate constants (K_p) of MAO coatings with and without Y₂O₃ addition

Growth rate	Ti–MAO	Ti–Y ₂ O ₃ –MAO
K_p (mg/mm ² s)	3.44039×10^{-4}	7.44785×10^{-4}
K_p (mg/mm ² s) (Between 420 and 600 s)	Stable	1.16691×10^{-5}

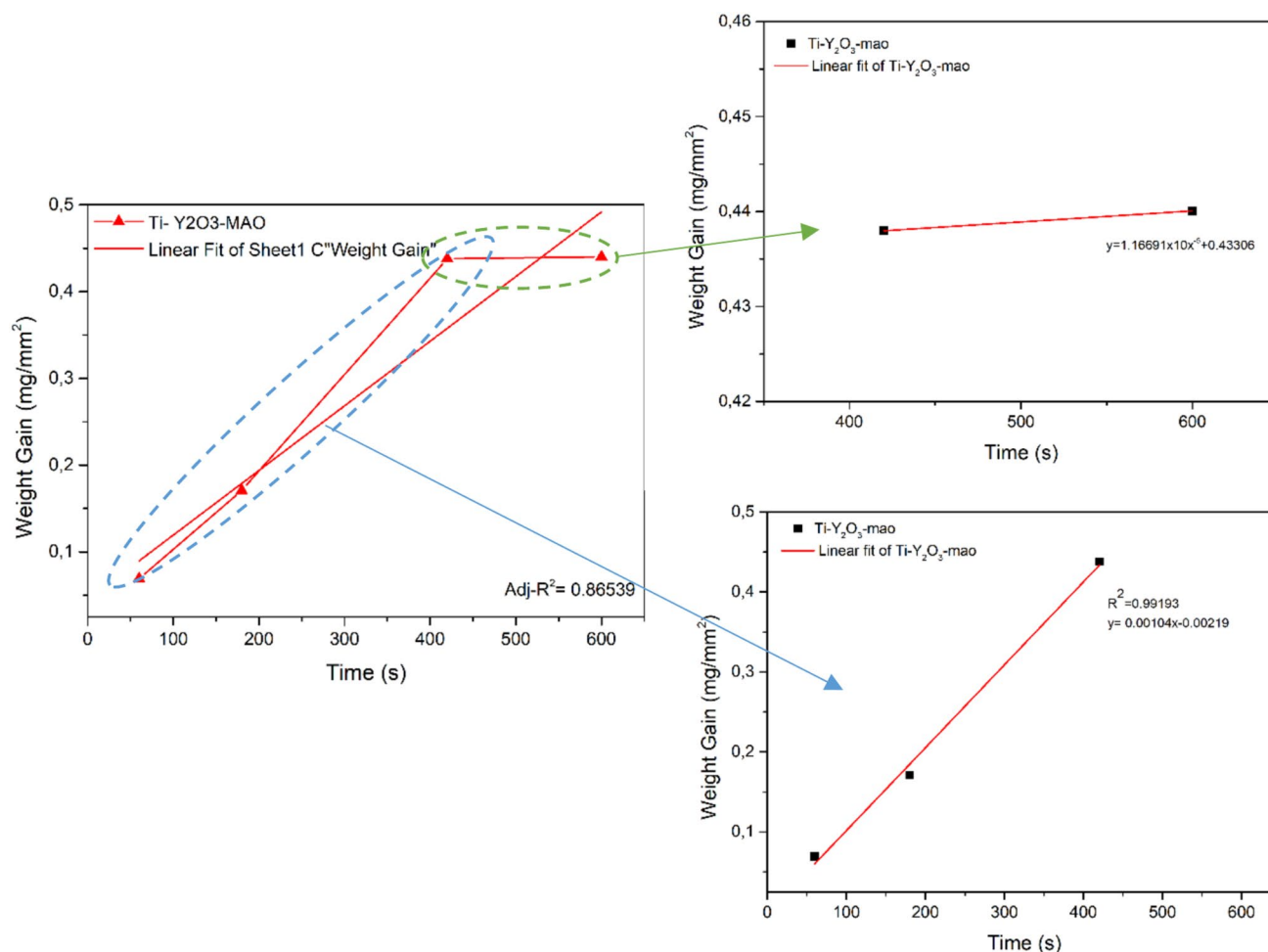


Figure 11 Time-dependent weight gain behavior of Ti-Y₂O₃-MAO coatings: overall linear fit and separate segmental linear regressions for early (0–420 s) and late (420–600 s) stages.

becomes evident, particularly in Y₂O₃ containing coatings [23, 32, 33].

Constant scratch test results

The constant load scratch test provided important insight into the mechanical integrity, coating compactness, and intra-coating damage behavior of the MAO coatings produced with and without Y₂O₃ addition. The constant load approach was deliberately employed to investigate damage evolution within the coating layer without penetrating to the substrate, enabling a direct comparison of scratch resistance and coating densification under identical loading conditions [38, 39]. The SEM images of the scratched surfaces in Fig. 12 revealed distinct differences between the coatings produced with and without Y₂O₃. The

coatings without Y₂O₃ (S-7 and S-10) revealed significant brittle fracture and microcracking along the scratch paths, with evidence of localized delamination. It was seen that micro-voids propagated outward from the scratch path, indicating poor load distribution within the porous oxide structure (Fig. 12a). However, the extended processing time led to increased pore size and the presence of re-solidified molten residues, which acted as weak points under mechanical loading (Fig. 12b). These morphological features led to the formation of larger fragmented regions and more irregular scratch profiles, indicating that the coating exhibited lower resistance to localized mechanical damage and scratch propagation. Wang et al. [40] reported that excessive porosity in MAO coatings compromises their ability to withstand mechanical stresses, thereby reducing adhesion and wear resistance. This

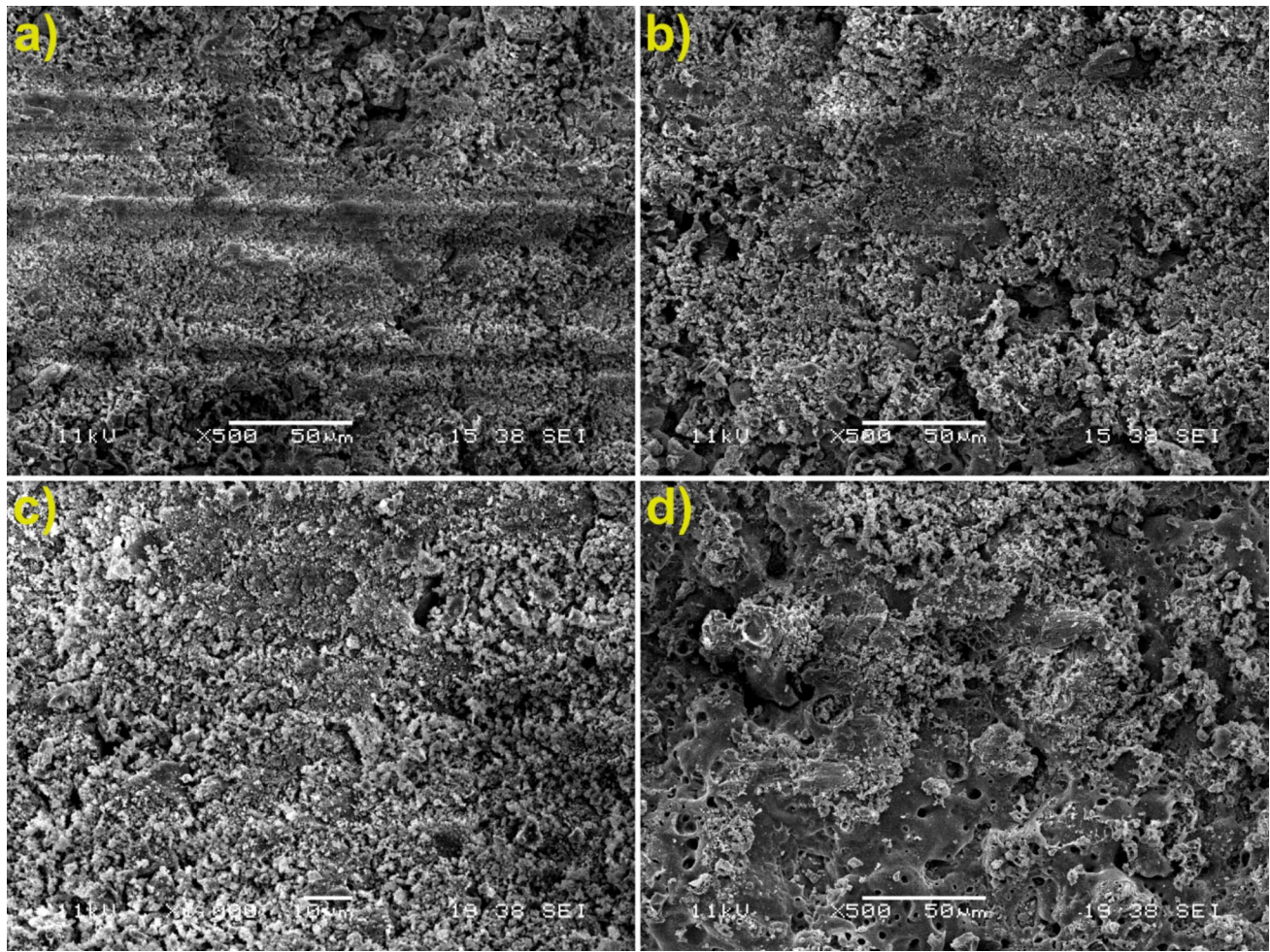


Figure 12 SEM image of the coating surface after the scratch test. **a** S-7, **b** S-10, **c** Y-7 and **d** Y-10.

behavior can be attributed to the fact that prolonged MAO treatment led to greater porosity and nodular surface features, which acted as stress concentrators under applied load, thereby reducing scratch resistance. In contrast, the Y_2O_3 -reinforced coatings (Y-7 and Y-10) exhibited shallower, smoother scratch tracks with fewer delamination features. Y_2O_3 addition might lead to denser coatings, which improved coating-substrate adhesion and resistance to crack initiation. As seen in Fig. 12c, it was displayed minor surface damage along the groove edges, while Fig. 12d showed localized accumulation of molten nodular structures that occasionally served as stress concentrators. The Y_2O_3 -containing coatings resisted brittle fracture more effectively than the unmodified coatings. Say et al. [41] attributed to the densification effect of Y_2O_3 particles, which filled micropores and improved coating compactness, consistent with recent studies on rare-earth

oxide incorporation in surface treatments. This behavior is associated with reduced porosity and enhanced bonding provided by Y_2O_3 , effectively increasing resistance to crack initiation and propagation. Furthermore, coating behavior that include scratch test results directly could correlate with the coating morphologies observed before testing. Porous, irregular structures in unmodified coatings acted as stress concentrators, promoting crack initiation under load, whereas Y_2O_3 addition produced dense, compact morphologies that improved load distribution and adhesion. Thus, this effect is ascribed to that the scratch resistance of Y_2O_3 coatings stemmed from their reduced porosity and improved homogeneity.

In Fig. 13, The 2D and 3D profilometer scans provided quantitative information on groove depth and width after scratching. It was detected that the scratch depths reached approximately 7–8 μm in S-7

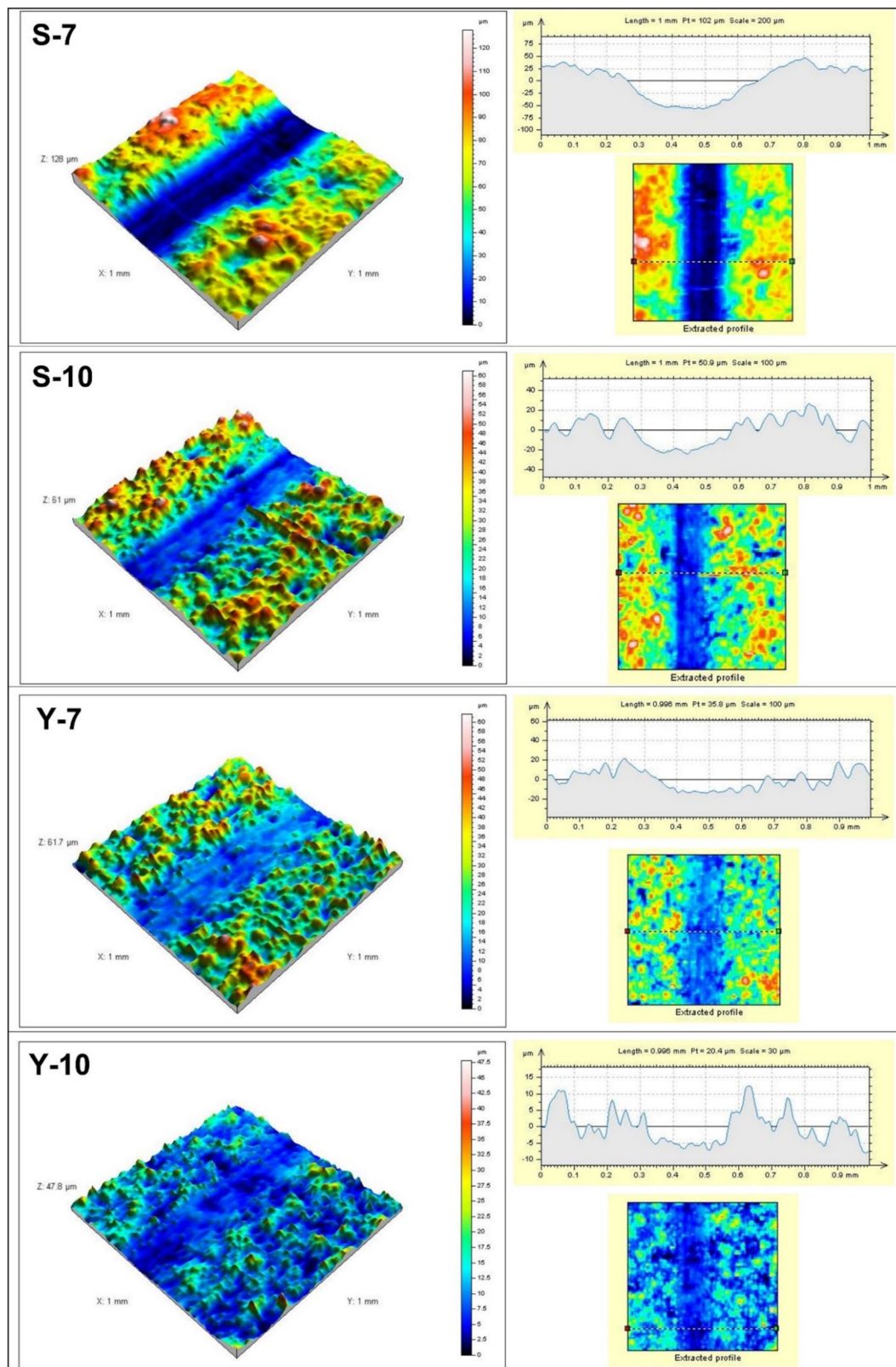


Figure 13 3D and 2D profilometer images of the scratches formed on the surface after the constant load scratch test.

(Fig. 13a) and 8–9 μm in S-10 (Fig. 13b), with groove widths exceeding 70–90 μm . This represented nearly a 45% improvement in scratch resistance with Y_2O_3 addition. The higher depth and width in S-10 were consistent with the weaker, confirming that longer treatment times without additives produced coatings more prone to brittle fracture and material removal under applied load. Otherwise, the scratch depths were markedly reduced, averaging 4–5 μm in Y-7 (Fig. 13c) and ~ 4 μm in Y-10 (Fig. 13d), while groove widths were narrower (40–60 μm). The smoother and shallower profiles indicated stronger adhesion and improved resistance to plastic deformation. The profilometer analysis confirmed the SEM findings that Y_2O_3 addition reduced groove depth by nearly 45% compared to Y_2O_3 -free coatings and demonstrated the clear role of additive-driven densification in improving coating integrity. Similarly, Feng et al. [41] demonstrated that particle-modified MAO coatings exhibited improved friction stability, highlighting the universal benefit of particle reinforcement in MAO processes. Grigoriev et al. [42] revealed that particle-modified MAO coatings on Ti–6Al–4V showed improved wear resistance. Thus, it could be attributed that localized molten residues and nodular agglomerations, consistent with the surface features, might function as stress concentration sites, occasionally initiating coating detachment during scratch testing. Consequently, the incorporation of Y_2O_3 during the MAO process significantly improved the scratch resistance of the coatings by producing denser, more homogeneous structures and reinforcing the oxide matrix. The findings highlighted the importance of both additive selection and process optimization.

The profilometry results corroborate the SEM observations and clearly demonstrate the role of Y_2O_3 -induced densification in improving the mechanical integrity and scratch resistance of the MAO coatings. Although localized molten residues and nodular agglomerations were occasionally observed, these features did not lead to extensive coating failure under constant load conditions. Overall, the incorporation of Y_2O_3 during the MAO process significantly enhanced the ability of the coatings to resist intra coating damage.

Conclusion

In this study, Ti–6Al–4V alloy substrates were successfully coated by the Micro Arc Oxidation (MAO) process in silicate-based electrolytes with and without Y_2O_3 addition. The experimental work, including weight gain analysis, SEM/EDS characterization, XRD phase identification, kinetic modeling, and scratch/profilometry testing, was carried out systematically. The findings can be summarized as follows:

- The incorporation of Y_2O_3 during the MAO process significantly modified the growth mechanism and final properties of TiO_2 -based coatings on Ti–6Al–4V alloy.
- Coatings without Y_2O_3 followed a predominantly reaction-controlled regime with strong conformity to the linear kinetic model (Adj. $R^2 \approx 0.9883$).
- Y_2O_3 containing coatings initially exhibited linear growth behavior but shifted to a multi-stage regime after approximately 420 s due to the insulating effect of the thickened ceramic layer, leading to a considerable reduction in growth rate.
- SEM analyses showed that Y_2O_3 addition reduced porosity, filled micropores, and improved densification, resulting in more compact and homogeneous coating structures.
- XRD results revealed that coatings evolved from amorphous TiO_2 and anatase phases at short durations to rutile at longer treatment times. Y_2O_3 influenced both crystallization and amorphization behaviors.
- The constant load scratch test results showed that the mechanical durability of the MAO coatings on Ti–6Al–4V alloy was strongly influenced by the addition of Y_2O_3 . Coatings without Y_2O_3 displayed brittle fracture and delamination along the scratch tracks, with profilometer measurements indicating deep grooves of about 7–9 μm and wide tracks of 70–90 μm . In contrast, the coatings containing Y_2O_3 exhibited smoother and more continuous scratch tracks with fewer cracks and minimal delamination. The profilometer analysis confirmed shallower grooves of about 4–5 μm and narrower widths of 40–60 μm , corresponding to nearly a 45% improvement in scratch resistance compared to the unmodified coatings. Occasional weak points were observed due to nodular agglomerations and molten residues. The results demonstrated that Y_2O_3 incorporation improved

adhesion strength and resistance to mechanical damage, confirming its effectiveness in producing MAO coatings with enhanced scratch resistance.

- Despite the improvements, prolonged MAO treatment (10 min) led to the formation of nodular agglomerations and localized molten residues, which could act as stress concentrators.

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Author's contribution

Aysun Ayday: Methodology, data curation, investigation, formal analysis, and writing—original draft and supervision. Erhan Duru: Investigation, methodology, data curation, writing—review and editing. Derya Kirsever: Investigation, writing—review and editing and formal analysis. Fatih Doğan: Investigation, writing—review and editing and formal analysis.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval Not applicable.

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