Effect of Ti₃SiC₂ particles addition on friction and oxidation behavior at high temperature of micro-arc oxidation layer on TC4 alloy

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This study involved the preparation of TiO_2 layers enhanced with the Ti_3SiC_2 phase. This was achieved by incorporating Ti₃SiC₂ particles (2-8 g/L) into an Na₂WO₄+Na₂SiO₃+Na₃PO₄ electrolyte system, utilizing the micro-arc oxidation (MAO) technique. The phase composition of the MAO layer was analyzed by X-ray diffractometer, the surface porosity and elemental distribution were assessed via scanning electron microscopy and energy dispersive spectroscopy; the cross-sectional thickness of the MAO layer was measured by metallurgical microscope; and the surface flatness was measured by confocal microscope. The friction behavior of the MAO layer with different Ti₃SiC₂ contents was compared by sliding wear tests; anti-oxidation tests was used to evaluate the oxidation resistance of the layers. The results show: 1) The addition of Ti₃SiC₂ particles with higher concentrations can increase the thickness of MAO layer, improve the existence of numerous "volcanoes" and large-sized pores in the MAO layer and play a certain "pore sealing" effect. 2) When the concentration of Ti_3SiC_2 is greater than 4g/L, the friction and wear behavior of the MAO layer is enhanced; 3) After oxidation at 700°C for 14 h, the MAO layer with higher concentration $(6 \sim 8g/L)$ showed better oxidation resistance. The Ti₃SiC₂ in the layer would work together with the atoms in Ti to block the entry of external oxygen atoms.

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1. Introduction

Ti alloys are extensively utilized across aerospace, military, automotive, and chemical sectors, attributed to their lightweight nature, exceptional specific strength, superior corrosion resistance, and robust resistance to shock and vibration [1-3]. However, its low hardness, poor wear resistance, and poor high-temperature oxidation resistance have again limited its application. Currently, many researchers have extensively explored advanced coating techniques, including chemical and electrochemical conversion, sol-gel processes, electrodeposition, electrophoretic deposition, vapor phase deposition, and thermal spraying, to enhance the corrosion and wear resistance of various alloys/composites [4]. The Micro-arc oxidation (MAO) [5-9] technique can lead to the formation of ceramic layers on the metal surface. The micro-arc oxidation ceramic layer is in situ generated and ensured a strong bond with the substrate. Its structure is defined by a dense inner layer and a loose, porous outer layer. For the MAO layer, the electrolyte composition is

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important in determining its organization and performance. The aluminate system [10,11], silicate system [12-14], phosphate liquid system [15-17], and their hybrid system [18] are commonly used in Ti alloy MAO electrolytes. However, the performance of the film layer synthesized in situ in simple electrolytes could not get a breakthrough, and it has become mainstream to incorporate different particles in the electrolyte [19,20] to enhance the performance of the ceramic layer of micro-arc oxidation. It has been found that crystalline micro/nanoscale surface oxides formed by plasma-assisted deposition can significantly improve wear and corrosion resistance [21-23]. Shokouhfar et al. [24] added SiC and SiO₂ nanoparticles to the electrolyte. They found that including these nanoparticles significantly decreased the microporosity size and surface roughness of the micro-arc oxidation layer. The nanoparticles were effectively integrated into the coating through electrophoretic forces and micro-sparking. Li et al. [25] added Ti₃SiC₂ particles to the electrolyte, and their research demonstrated that with an increase in the concentration of Ti₃SiC₂, there were marked variations in the PECC layers, significantly enhancing the deposition sealing effect. Li et al. [26] investigated the effects of adding ZrO₂ particles to titanium alloys. Their study revealed that as the concentration of nanoparticles increased, the thermal weight gain during heat treatment decreased, reaching only 12% of that of the substrate. However, at excessively high concentrations, film cracking occurred, leading to a slight increase in thermal weight during prolonged heat treatment.

In this study, varying concentrations of Ti_3SiC_2 particles (0, 2, 4, 6, and 8 g/L) were introduced into the electrolyte as experimental variables. The research focused on examining the microstructure, wear resistance, and high-temperature oxidation resistance of the MAO layer formed at different Ti_3SiC_2 particle concentrations. The objective was to study the impact of Ti_3SiC_2 particle addition on the tribological and oxidation resistance properties of the MAO layer and to determine the optimal concentration range for Ti_3SiC_2 particle incorporation.

2. Experimentation

The substrate was selected as TC4 alloys with a size of φ 25-4 mm. It underwent a sequential polishing process using sandpaper ranging from 80# to 800#, followed by ultrasonic cleaning in acetone for 10 minutes, and was subsequently dried for use as spare parts. The MAO layer was fabricated by a WHD-30 apparatus, operating in constant current mode. The process was conducted with a positive and negative current of 1.5 A at a frequency of 500 Hz for a duration of 15 minutes. The electrolyte solution consisted of 8g/L Na₂WO₄, 6g/L Na₂SiO₃, and 4g/L Na₃PO₄. Particles were incrementally introduced at concentrations of 0, 2, 4, 6, and 8 g/L, serving as experimental variables.

Following micro-arc oxidation treatment, the samples were rinsed with deionized water and allowed to dry naturally for performance testing. A SIGMA-500 field emission scanning electron microscope (SEM) facilitated the examination of pore morphology on specimen surfaces, while porosity in designated areas was quantified via Image J software. Surface scanning with an AZTEC X-Max spectrometer was used to determine the surface element distribution of the samples. Phase identification on sample surfaces was conducted using X-ray diffractometry (XRD) with a D/max-2500/pc model, and diffraction peaks were cross-referenced with standard PDF maps via Jade software. The surface flatness of the samples was observed using a 3CCD confocal microscope. The MAO layer's thickness was assessed utilizing an Axio Scope A1 metallographic microscope. The

HT-100 high-temperature friction and wear tester was employed under conditions of 600° C, a 10N load, Si₃N₄ as the abrasive material, a 4mm wear radius, and a 20-minute duration. Resultant experimental data were graphically represented as line graphs using Origin software.

The cyclic oxidation method was employed to assess the coating's resistance to oxidation at elevated temperatures: the coating sample was subjected to a cyclic oxidation test in a muffle furnace at 700°C, held for 14h, air-cooled to room temperature. The mass of the samples before and after oxidation was weighed separately, the weight gain or loss curves were plotted, and the oxidized samples were analyzed by XRD.

3. Results and analysis

Fig.1 shows the morphology and pore distribution of the MAO layer with different Ti₃SiC₂ contents. It can be seen that by adding different contents of Ti₃SiC₂, there are a large number of "peak" deposits on the surface of the MAO layer and many micropores of various sizes are distributed. Employing Image J software for the assessment of porosity, as shown in Fig.1: f, it can be found that the porosity distribution on the surface of the MAO layer shows a decreasing trend of increasing and decreasing. The best porosity is 17.64% and 16% at 4 g/L and 8 g/L. The enhancement in conductivity, reduction in arc energy, and formation of a denser MAO layer are attributed to the incorporation of Ti₃SiC₂. As Ti₃SiC₂ concentration increases, the prevalence of large-diameter pores also rises. This phenomenon is likely due to excessive Ti₃SiC₂ content, which enhances solution conductivity and accelerates the micro-arc oxidation process. In the initial stages, concentrated arc discharges lead to repeated breakdowns in smaller channels, resulting in large-diameter pores. Concurrently, molten substrate oxides interact with excess Ti₃SiC₂ in the electrolyte through these large micropores, condensing on the membrane surface and causing peak-like oxide deposition. This substantial oxide deposition obstructs surface pores, leading to reduced flatness at 8 g/L, despite achieving the lowest porosity.

The thicknesses of MAO layers with different Ti_3SiC_2 contents were measured at five locations on the same position and the average values were calculated as shown in Fig.1: g. Results indicated an increase in thickness for all MAO layers following the addition of Ti_3SiC_2 particles. Specifically, the average thickness was 52.99 µm with a Ti_3SiC_2 content of 8 g/L, compared to 28.142 µm at 0 g/L. The Ti_3SiC_2 particles, suspended in the electrolyte, are incorporated into the molten oxides ejected from the oxidized layer of the sample, rapidly cooling and depositing to form the MAO layer. The addition of Ti_3SiC_2 serves a "pore sealing" function, enhancing the flatness and quality of the MAO layers when the content is below 6 g/L. However, contents exceeding 6 g/L negatively impact the MAO layer, as excessive Ti_3SiC_2 particles agglomerate and deposit on the surface, compromising flatness and quality. Furthermore, as electrically conductive ceramic particles, Ti_3SiC_2 alters the electrolyte's conductivity, affecting the breakdown voltage, optimizing energy density, and consequently influencing the MAO surface's flatness.



*Fig. 1. Morphology and pore distribution of MAO layer with different Ti*₃*SiC*₂ *contents: (a)0g/L; (b)2g/L; (c)4g/L; (d)6g/L; (e)8g/L; (f) Porosity; (g) layer thickness.*

Fig.2 shows the surface and 3D topography of the MAO layer with different Ti_3SiC_2 contents. It can be found that the 3D shows a high and low undulating topography when no Ti_3SiC_2 particles are added. This is related to the inherent characteristics of the MAO layer, that is, there are a large number of "volcanoes" and large-size holes in the MAO layer. When Ti_3SiC_2 particles are added to the electrolyte, this characteristic is somewhat improved. When the concentration of Ti_3SiC_2 is 4g/L, the surface of TC4 alloy has the lowest oxide deposition and the highest surface quality. The reason for the quality improvement is twofold. Firstly, Ti_3SiC_2 particles exhibit stability

and remain inert when in contact with other substances with the electrolyte, so they are gradually deposited and solidified in the MAO layer with the reaction, which plays a certain "pore sealing" effect. Secondly, incorporating Ti_3SiC_2 particles into the solution enhances electrical conductivity, optimizes discharge energy density, and significantly improves the quality of the MAO layer. Conversely, at an 8g/L concentration, the TC4 alloy surface experiences maximum oxide deposition and increased roughness due to the higher Ti_3SiC_2 content in the electrolyte. This results from the increased deposition and solidification of Ti_3SiC_2 particles within the MAO layer and the agglomeration phenomenon, where Ti_3SiC_2 deposits in the pores and cracks, affecting the macroscopic morphology.



*Fig. 2. Surface and 3D topography of MAO layer with different Ti*₃*SiC*₂ *contents: (a)0g/L; (b)2g/L; (c)4g/L; (d)6g/L; (e)8g/L.*

Fig.3 shows the XRD composition of the MAO layer with different Ti_3SiC_2 contents, which shows that the MAO layer is mainly composed of Anatase-TiO₂, Rutie -TiO₂, matrix Ti and a small amount of Ti_3SiC_2 . Moreover, with the addition of Ti_3SiC_2 , the diffraction peak of Anatase-TiO₂ at $2\theta=28^{\circ}$ is slightly enhanced, which is due to the increase in Ti_3SiC_2 content leading to the increase in the thickness of the film layer, which makes the film layer decrease in thermal conductivity and promotes the transformation of room temperature stable Anatase-TiO₂ to Rutie-TiO₂. The presence of diffraction peaks in the Ti matrix can be attributed to the loose structure of the surface layer, facilitating ray penetration into the matrix. The addition of Ti_3SiC_2 enhances the layer's thickness and therefore slightly increases the Rutile and Anatase phases. The appearance of Ti_3SiC_2 diffraction peaks in the XRD pattern indicates that the Ti_3SiC_2 ceramic particles enter or remain on the MAO layer. The apparent "bun peak" between $2\theta=20^{\circ} \sim 30^{\circ}$ may be due to the formation of amorphous SiO_2 under the effect of high-temperature discharge.



Fig. 3. XRD of MAO layer with different Ti₃SiC₂ contents.



Fig. 4. SEM images of MAO layer with different Ti₃SiC₂ contents: (a)0g/L; (b)2g/L; (c)4g/L; (d)6g/L; (e)8g/L.

The SEM results of MAO layers with different Ti_3SiC_2 contents are shown in Fig.4, indicating that these layers have similar compositional elements. The composition consists mainly of four elements: Ti, O, Si, and Na, with O and Ti being the predominant components in the MAO layer, and Ti being derived from both the matrix and TiO₂. The O element originates from oxide deposits like TiO₂ and SiO₂. The Si element is primarily sourced from Na₂SiO₃ and Ti₃SiC₂ with the electrolyte, while Na is derived from electrolytes such as Na₂SiO₃, Na₃PO₄, Na₂WO₄. Combined with the XRD pattern, the presence of Ti₃SiC₂ is observed within the MAO layer, and it is evident that incorporating Ti₃SiC₂ does not alter the elemental composition of the MAO layer.

The results of the friction test on the MAO layer with different Ti_3SiC_2 contents are shown in Fig.5. It is observed that the friction coefficient of the MAO layer does not change significantly when the content of Ti_3SiC_2 is less, and the friction coefficient of the MAO layer decreased significantly when the content of Ti_3SiC_2 reached 6g/L and reaches the lowest friction coefficient at 8g/L. From Fig.5: (a), the incorporation of Ti_3SiC_2 significantly influences the friction coefficient of the MAO layer. The introduction of Ti_3SiC_2 particles results in a smoother fluctuation curve of the friction coefficient, which decreases progressively with increasing Ti_3SiC_2 content.

After calculating the average friction coefficient and wear volume, the results are shown in Fig.5: (b). The average friction coefficient exhibits a gradual decline, with a peak value of approximately 0.46 at a concentration of 2g/L and a minimum of about 0.393 at 8g/L. This trend aligns with the friction coefficient changes depicted in Fig.5: (a). While the wear volume shows a trend of significant increase and then decrease, the maximum volume wear is about 1.03 mm³ at 0g/L, and the minimum volume wear is only 0.65 mm³ at 8g/L. The combined results indicate that the friction performance of the MAO layer is enhanced by the addition of Ti₃SiC₂, particularly at concentrations exceeding 4g/L, with 8g/L offering optimal wear resistance. This improvement is attributed to the reduction of voids in the film layer and enhancement of surface quality due to the appropriate amount of Ti₃SiC₂ in the electrolyte. Additionally, Ti₃SiC₂ acts as a solid lubricant, owing to its self-lubricating properties and ability to layer easily, thereby contributing to improve friction and wear characteristics.



Fig. 5. Friction curve of MAO layer with different Ti_3SiC_2 contents: (a) average friction coefficient; (b) Average coefficient of friction and Volume wear.

Fig. 6 shows the macroscopic photos of MAO layers with different Ti₃SiC₂ contents after oxidation at 700°C for 14 hours. Most obviously, the coatings with 0g/L and 4g/L Ti₃SiC₂ exhibited localized blackening and prominent black scorch marks, with significant detachment of the loose oxide layer, likely due to severe oxidative transformation at elevated temperatures. Conversely, coatings containing 2 g/L and 8 g/L Ti₃SiC₂ demonstrated superior high-temperature antioxidant properties, maintaining structural integrity with only minor gray-white speckling observed.



Fig. 6. Macroscopic photos of MAO layer with different Ti₃SiC₂ contents after oxidation at 700°C for 14h: (a)0g/L; (b)2g/L; (c)4g/L; (d)6g/L; (e)8g/L.

The XRD of the MAO layer with different Ti_3SiC_2 contents after oxidation at 700°C for 14 h is depicted in Fig.7. The spectral peaks of the MAO layers after high-temperature oxidation exhibit a general similarity. The primary constituents of the MAO layer include Anatase-TiO₂, Rutile-TiO₂, TiSi₂, SiC, matrix Ti, and a minor fraction of Ti₃SiC2. It is noteworthy that the coating with 4 g/L Ti₃SiC₂ addition showed new phases with spectral peaks at 20=36.88, 42.8, 54.34, and 62.34, which distinguishes it from the other concentrations. A reduction in the intensity of matrix Ti is observed, attributed to the oxidation of residual Ti into rutile-TiO₂, which is more thermally stable, and TiO, which is less stable at elevated temperatures. A portion of Ti3SiC2 is transformed at high temperatures to form SiC and TiSi2. In addition, the absence of prominent "steamed bun peaks" between $2\theta = 20^{\circ} \sim 30^{\circ}$ is attributed to the breakdown of the disordered structure of amorphous SiO₂ at elevated temperatures, leading to the disappearance of the amorphous phase.



Fig. 7. XRD of MAO layer with different Ti₃SiC₂ contents after oxidation at 700°C for 14h.

The relative content of Rutie-TiO₂ after high-temperature oxidation is higher for MAO layers with different Ti_3SiC_2 contents. This is because the residual Ti in the coating is oxidized to Rutie-TiO₂, which is more stable at high temperatures, and part of the Anatase-TiO₂ is transformed at 650°C.

Fig.8 shows the 3D surface topography and cross-section of MAO layers with different Ti_3SiC_2 contents after oxidation at 700°C for 14 h. After high-temperature oxidation, the MAO layers exhibit a notable reduction in three-dimensional waviness, an increase in green areas, a decrease in red bulges, and a filling of depressions. The flatness of these layers is significantly enhanced, particularly with the addition of 8g/L Ti_3SiC_2 , where the roughness Ra is reduced to 0.797 µm. Otherwise, the MAO layer with 4g/L Ti_3SiC_2 maintains a higher roughness Ra of 2.597 µm after oxidation. This is likely due to minimal surface oxide deposition on the TC4 alloy at this concentration, leading to replenished red bumps post-oxidation. The minimal oxide deposition results in residual Ti being oxidized to Rutile-TiO₂, which is stable at high temperatures, alongside unstable TiO. XRD analysis with 4g/L Ti_3SiC_2 corroborates the formation of new phases post-oxidation, contributing to the increased roughness.

From Fig.8:($a_1 \sim e_1$), there are obvious differences in the thickness of the MAO layer after high-temperature oxidation, but the continuity of the coating is not damaged. Especially, the MAO layer's thickness remains relatively unchanged at lower Ti₃SiC₂ concentrations. However, at a concentration of 4g/L, a marked reduction in thickness is observed, with an 8g/L concentration resulting in a 20µm decrease compared to pre-oxidation levels. This phenomenon is attributed to the increased deposition of Ti₃SiC₂ particles within the MAO layer as the electrolyte concentration rises. These particles, encapsulated by in-situ generated TiO₂, undergo a crystalline transformation during high-temperature oxidation, leading to the detachment of Ti₃SiC₂ particles and their conversion into TiSi₂ and SiC. Consequently, this transformation contributes to the reduction in MAO layer thickness and enhances the flatness of MAO layers with varying Ti₃SiC₂ contents post-hightemperature oxidation.

Ti alloys used in high-temperature environments are prone to oxidation and gradual failure, and the smaller the change in oxidation weight gain, the more favorable it is. As shown in Fig. 9, the oxidation weight gain of MAO layers with varying Ti₃SiC₂ contents after cyclic oxidation at 700°C for 14 hours was recorded as -0.00995g/mm², -0.0089g/mm², -0.00266 g/mm², -0.00609 g/mm², and-0.00515 g/mm², respectively. Especially, the sample with 4 g/L Ti₃SiC₂ exhibited the smallest change in weight gain, attributed to reduced oxide deposition on the surface. Despite the MAO layer experiencing peeling and blackening post-high-temperature oxidation, the oxidation of Ti atoms to TiO₂ and TiO accounted for the observed mass difference. During high-temperature oxidation, external oxygen atoms infiltrate the coating's pores, reacting with the residual Ti atoms.

The incorporation of Ti_3SiC_2 into the coating significantly hinders the interaction between oxygen and titanium. The presence of external oxygen atoms in the Ti atoms, along with Ti_3SiC_2 interception and micro-arc oxidation coating, collectively prevents oxygen from reaching the TC4 substrate surface, thereby enhancing the anti-temperature oxidation performance of titanium alloys. From Fig.1: g, it can be seen that the thickness of all MAO layers is improved after the addition of Ti_3SiC_2 particles, and with the increase of Ti_3SiC_2 concentration, the thickness of the film layer is greater. Therefore, the higher the concentration of Ti_3SiC_2 in the electrolyte, the stronger the blocking effect of the film layer on the diffusion of oxygen to the substrate and the stronger the protective effect on the titanium alloy substrate. It can be seen that the addition of different contents of Ti_3SiC_2 can improve the anti-temperature oxidation performance of MAO layers.



Fig. 8. The surface 3D topography and cross-section of MAO laye with different Ti_3SiC_2 contents after oxidation at 700 °C for 14 h: (a)(a₁)0g/L; (b)(b₁)2g/L; (c)(c₁)4g/L; (d)(d₁)6g/L; (e)(e₁)8g/L.



Fig. 9. Weight gain of MAO layer with different Ti₃SiC₂ contents after cyclic oxidation at 700°C for 14h.

4. Conclusion

The study involved the preparation of TiO_2 metal oxide ceramic coatings, enhanced with the Ti_3SiC_2 phase, on TC4 titanium alloy surfaces using the micro-arc oxidation technique. The investigation focused on the impact of varying Ti_3SiC_2 particle concentrations—specifically 0, 2, 4, 6, and 8 g/L—within the electrolyte on the microstructure, tribological characteristics, and high-temperature oxidation properties of the resulting MAO layer.

The findings indicate that the Ti_3SiC_2 concentration in the electrolyte significantly influences the porosity and thickness of the MAO layers. Initially, an increase in Ti_3SiC_2 content reduces surface pores and cracks, but further increases lead to their proliferation. At a concentration of 4g/L, the surface exhibits uniformity and density with minimal micro-pores and cracks. Conversely, at 8g/L, there is a substantial accumulation of Ti_3SiC_2 particles, resulting in poor film flatness. The incorporation of Ti_3SiC_2 particles consistently enhances the thickness of the MAO layers, with an average thickness of 52.99 µm observed at an 8g/L concentration.

The concentration of Ti_3SiC_2 in the electrolyte has little effect on the physical phase composition of the MAO layers. Under different Ti_3SiC_2 content conditions, the MAO layer is mainly composed of Rutile-TiO₂ and Anatase-TiO₂ with a small amount of Ti_3SiC_2 phase. The wear resistance of the MAO layers is significantly affected by the incorporation of Ti_3SiC_2 particles, as evidenced by a reduction in the friction coefficient and wear volume. Optimal wear resistance is observed at a concentration of 8g/L.

After high-temperature oxidation, the MAO layer exhibits a notable variation in thickness, while maintaining coating continuity. The process induces crystalline transformation in TiO_2 and alters Ti_3SiC_2 particles in the MAO layer's outer region, leading to a thinner MAO layer and enhanced flatness. The synergy of Ti atoms, Ti_3SiC_2 interception, and the micro-arc oxidation coating's oxygen barrier prevents external oxygen from oxidizing the TC4 substrate, thereby enhancing the titanium alloy's resistance to high-temperature oxidation. By comparing the surface morphology, phase composition, wear resistance, and high-temperature oxidation resistance of Ti_3SiC_2 particles with different additions, the optimum additions of Ti_3SiC_2 particles under laboratory conditions were 6-8 g/L.

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