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## The Microstructure and Abrasion Resistance of The Oxide Coatings on Ti-6Al-4V Produced by Plasma Electrolytic Oxidation in A Cheap Electrolyte

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## Abstract

A category of surface treatments, known as plasma electrolyte oxidation, has been employed to enhance the tribological characteristics of titanium and its alloys. The plasma electrolytic oxidation of Ti-6Al-4V alloy has been extensively researched. Energy Dispersive Spectroscopy (EDS), a scanning electron microscope (SEM), and mechanical tests (microhardness and wear) are used to identify the coating properties and morphology, and the phases produced are identified. Findings from an investigation into the TiO<sub>2</sub> layer on the Ti-6Al-4V alloy The findings showed that the MAO method enhanced wear resistance by (69.85)% and allowed for the deposition of layers with thicknesses ranging from 19.08 to 24.6  $\mu$ m and high hardness (420 to 950 HV) in this research, the best results were obtained at the lowest possible cost and using low-cost equipment.

Key Words: Ti6Al4V; Micro-Arc Oxidation; Hardness and Wear Test.

## 1. Introduction

Due to their low density and outstanding corrosion resistance, titanium alloys are ideal for a variety of applications. However, these alloys cannot be used to make mechanical components that are prone to friction because of their tribological characteristics. In actuality, friction—regardless of the materials in contact—increases the seizure phenomenon, particularly for a titanium/titanium contact. Aluminum, titanium, and magnesium are common examples of light metals and valve metals that are commonly anodized utilizing techniques like micro-arc oxidation (MAO) and plasma electrolytic oxidation (PEO). The manufacture of multifunctional coatings is possible thanks to this high voltage technique, which promotes the formation of a thick, ceramic-like oxide [1,2]. This form of coating is being taken into consideration in this situation to increase the required surface. The coatings' potential for substantial porosity raises the possibility of lessening the bonding strength between the oxide layers and the titanium substrate [3]. The coated surface generates pores since it is an electrolyte, which affects how rough the coating is. Both adhesion and roughness affect the wear resistance of MAO coatings..

Micro arc oxidation (MAO) is a promising method for synthesizing the ceramics oxide layers on (aluminum alloys, titanium alloys, Mg) through the discharge sparks at elevated voltages [4]. The micro-arc oxidation method can be considered as a collection of plasma discharging (PD) and anodizing electrolytic oxidation (AEO). In which, the major similarity between anodizing methods and the micro-arc oxidation, is that each of them involve oxidation of substrate using electrolytic baths, and the initially of the (MAO) method is an anodization method [5]. In fact, MAO has been utilized to increase wear rate in a number of studies [6]. Using micro-arc oxidation, a

2. Methodology 2.1.Materials used

composite covering of titanium oxide (TiO<sub>2</sub>) and hexagonal boron nitride (HBN) particles

was produced on the exterior of a Ti6Al4V alloy. The results demonstrate that the inclusion of SiO2 particles improved the MAO coating's anti-friction property[7]. SiO<sub>2</sub> nanoparticles

In order to minimize wear rate and boost micro hardness of Ti6Al4V by MAO coatings, which work to generate a dense, thick, and highly adhesive layer of titanium oxide TiO<sub>2</sub>, a

Ti6Al4V (Grade 5), a titanium alloy that is often used in industry, served as the study's

subject. Its chemical make-up is displayed in Table 1, The alloy was imported from China from the TIANJIN XINJIA ALBERT STEEL TRADE CO.LTD company. The samples were cut with a wire cutter from a 30 mm rod that had a 16 mm diameter and a 5 mm height. The work surface was cleaned with ultrasonic agitation in acetone, rinsed with deionized water,

were created and coated on titanium using the micro arc oxidation (MAO) process.

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and dried in warm air before being successively ground and polished with SiO<sub>2</sub> abrasive sheets ranging from 180 to 3000 grit. Using a Dc power supply and a glass vessel with stirring and ice water chilling, MAO treatment was performed. The counter electrode was a stainless steel plate.

TiO<sub>2</sub> coating was created on the surface of the sample.

## Table 1: Chemical Composition of The Alloy Used in This Research Weight %

Gr-5	Al	V	Fe	С	N	Н	0	Ti
Ti-6Al-4V	4.5-5.9	3.9 -4	≤0.30	$\leq 0.1$	$\leq$ 0.05	≤ 0.015	≤ 0.20	Bal.

## **2.2.preparation of MAO coatings**

The MAO, anodizing, and duplex procedures were utilized in a handmade coating equipment to deposit the ceramic coating. Coating equipment, a cathode, an anode, a ventilation unit, a stirrer, an exterior plastic tank, an electrolyte container, a cooling system, and a power supply unit. This study creates a coating system with high voltage and current requirements. The power supply was a homemade, variable AC-DC unit with variable voltage (0-500v) and variable current density (0-1) mA/cm2. The substrate utilized as the anode (Grade 5) is Ti6Al4V, while the electrode (Cathode) is stainless steel 316L. The solution was placed in a 1-liter container, and the electrodes were submerged vertically. The electrolyte was applied to the electrodes, and the spacing between them was measured where it was 3 cm., which is shown in Fig.1.



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## Fig.1: Images Of The Anodizing Coating Unit (1) Mixer, (2) Coating Container, (3) Cooling Container, (4) Power Supply, (5) Voltage Gauge, (6) Current Gauge.

### Table 2: The levels of MAO parameters

Sample code	Voltage (V)	<b>Operation time (min)</b>		
A(base)		uncoated		
В	300	35		
C	320	50		

## 2.3. Characterizations:

Micrograph observations were used to estimate coating thickness, and a scanning electron microscope (SEM) axia chemi sem thermo scintific company\ Dutch origin, This analysis was done at in Al-Khorah Laboratory/ Baghdad. was used to examine cross-sectional morphologies. Titanium dioxide was confirmed to exist via X-ray diffraction This analysis was done at Ceramic Department College of Material Engineering/Babylon University . A profilometer with interferometric technology was used to measure roughness. On the uncoated and coated specimens, the Vickers microhardness test was performed using a load of 200 g and an indentation period of 25 s. Five measurements are averaged to produce each hardness value. A tribometer with a ball-on-disk (TBR Anton Paar) was used to test the coatings for friction and wear while they were at room temperature. The circular module was used to operate the tribometer, which is shown in Fig. 2. In Table 3, the parameters for circular friction are listed.



### Fig 2. Schematic Diagram For Ball-on-Disk Testing

#### **Table 3 Ball-on-Disk Testing Parameters**

Grade 5 alloy	Ti6Al4V
Tested substrates	Circular friction

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Load N	4
Ball(diameter mm)	6
Frequency /Rotating speed	477
Cycle number	5
Dry conditions	ves

## **3. Results And Disscusion**

## **3.1.**Composition and microstructure

Fig.3.a showed SEM of the surface of Ti6Al4V, EDS analysis clearly showed the presence of Ti, Al, and V shown in Fig.3.b, The MAO coatings that were created in the electrolytes are shown in surface form in Fig.4. The surface of the particle-free MAO coating is shown in Fig.4(a), together with the pan-like structure, and fractures caused by the eruption of melted material from the coating's interior[8]. These pores were created as a result of a breakdown process that involved the release and discharge of gas bubbles, with the former aspect having a considerable impact on pore development the surface morphologies of MAO coatings were examined in order to study their surface microstructures, as shown in Fig. 4. Evidently, every coating demonstrates the characteristic porous surfaces with numerous micropores, which are believed to have been created by the residues of the discharge events in the MAO processes [9].

Various MAO coatings' element composition was examined using the EDS investigation's findings, which are depicted in Fig. 4. Every layer, as can be seen, contains O, Ti, Al, Na, Si, and /or P. Given that the coatings include titanium 6.3 Weight %, silicon 15 Weight% and 45 Weight % or more oxygen, it is likely that Ti, Al, Si, and/or P oxides make up the majority of the coatings. The Na, Si, and/or P elements in the coatings derive from the silicate- and/or phosphate-containing electrolytes, whilst the Ti and Al components are clearly from the Ti alloy substrates. The XRD pattern in Fig. 5 provides additional evidence of how electrolytes affect the development of MAO coatings. SiO<sub>2</sub> particles, TiO<sub>2</sub> coating particles, and TiO<sub>2</sub> /SiO<sub>2</sub> composite coatings are shown in Fig. 4 in the electrolyte made up of Na2SiO<sub>3</sub>=10g/l, (NaPO3)6=8g/l, and NaOH=2g/l particles, respectively, according to their XRD patterns. The majority of the particle-free TiO<sub>2</sub> coating was composed of rutile and anatase TiO<sub>2</sub>[10,11]. It was found that silicon particles in the electrolyte had produced a new phase of S and P when MAO coatings were produced. By demonstrating that the Si particles in the electrolyte were effectively absorbed by the MAO TiO<sub>2</sub> coatings, this result lends credence to the EDS results in Fig. 4a and b. Si particles were deposited into the TiO<sub>2</sub> coating during the MAO process, either filling the coating micro-pores or being trapped by the liquid coating materials, under the impact of electrophoretic, diffusion, and adsorption [12]. Additionally, the XRD pattern demonstrates that the inclusion of Si particles raised the relative peak strength of rutile TiO<sub>2</sub> against anatase  $TiO_2$ . The unstable anatase  $TiO_2$  phase was encouraged to change into the stable rutile TiO<sub>2</sub> phase at higher temperatures[13] because the Si particles in the MAO electrolyte reduced its heat conductivity.

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Fig. 3 show the (A) Sem Microphotographs (B) The Eds analysis of (Ti6Al4V) for sample A.



Fig. 4. Surface Morphologies (Sem And Eds analysis) of The: A – Titanium Substrate Mao At 300v, B – Titanium Substrate MAO At 320v.

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Fig. 5. Xrd Patterns Of Sio2 And P Particles, TiO<sub>2</sub> Coating Fabricated In The Electrolyte Containing Na2sio3= 10g/L, (Napo3)6=8g/L, And Naoh=2g/L Particles.

## 3.2 MAO Coating Characteristics: Roughness and Thickness:

The qualities of the manufactured MAO coatings on grade 5 (Ti6Al4V) titanium alloy that is readily accessible commercially are listed in Table 4. The thickness and oxide composition of coatings applied on Grade 5 titanium at a voltage of 300V (TiO<sub>2</sub>, alone) were not especially attractive. During the MAO process, micro-arcs were also seen on the substrate surface, albeit very faintly. MAO coatings produced at 320 V frequently have a harder surface and a larger oxide layer than those created at lower voltages (300 V). The results of measuring the surface roughness of the (B) and MAO (C) samples are shown in Table 4. It is clear that after MAO treatment, the surface roughness increased. The surface roughness increased gradually throughout the treatment, which is in agreement with SEM surface e s r S u t

Sample	Thickness	Roughness Ra	Microhardness HV
Nontreatd Ti Grade 5 substrate	0	$\sim 0.1 \ \mu m$	420
MAO coating on (B) sample at 300 V	19.08 µm	$\sim 2.7 \ \mu m$	780
MAO coating on (C) sample at 320 V	24.60 µm	$\sim 3.1 \ \mu m$	950

## Table 4 Thicknesses, Roughness, And Microhardness Hv Of Mao Coatings **Obtained On Grade 5 Titanium Alloys**

Fig. 6 displays high-magnification SEM images of the MAO coatings' surface and crosssectional morphologies. There were numerous micro-cracks, particularly along the discharge channels, on the surface of the TiO<sub>2</sub> coating covered with SiO<sub>2</sub> particles (Fig. 6 a). The crosssectional SEM observation revealed that after piercing the entire coating in the thickness direction, a number of microcracks had begun to spread along the coating/substrate contact. The strength of the coating and its capacity to stick to the substrate would both be negatively

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impacted by these fissures (Fig. 6 a). Below is a list of the major factors that contributed to the development and spread of these microcracks[14]. The coating rapidly cooled as a result of the powerful and intricate MAO reaction, moving electrolyte, and localized instantaneous high temperatures brought on by plasma micro-arc discharges. The titanium substrate was subjected to frequent and strong temperature changes, which significantly aided in the development and spread of cracks since the MAO ceramic covering had a lower coefficient of thermal expansion than the titanium substrate. The MAO coating's brittleness, porous design, and high growing stress all played a part in the fracture development as well. However, as can be shown in Fig. 6b, no substantial microcracks were found on the surface of the composite covering. This is done in order to increase the ceramic coating's fracture toughness by enhancing the high voltages obtained during the PEO process and the prolonged macro-arcing stage during coating growth on the matrix of brittle ceramic materials. Additionally, as current density and frequency decrease, the coatings thicken. The beginning and spread of microcracks were substantially prevented by the coatings' increases [8]. The two sample coatings had a noticeable thickness variation, as can be seen in Figs. 6a and 6b. They had different voltage-time responses, which is what caused this occurrence (Fig. 1) [15]. Fig. 6 shows the (a, b)  $TiO_2$  coating's surface and cross-sectional morphologies.



Fig. 6 shows the section-by-section morphologies of the (A, B) TiO<sub>2</sub> coating at various voltages.

## **3.3 Dry sliding tests**

Dry conditions have been used for circular ball-on-disk investigations. The friction coefficient value is unaffected by the presence or absence of an MAO coating on top of a titanium Grade 5 surface, regardless of the test setup. The type of contact (non-treated titanium on MAO coating, both MAO-coated surfaces,etc.) had no impact on the seizure phenomenon. As seen in Fig.7, where Fig. 7a shows the uncoated sample with coated samples and Fig. 7b shows the coated samples alone, wear tracks and worn volumes, however, varying quite a bit depending on the types of surfaces in contact. A MAO

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coating applied to one or both sides considerably reduces the wear propensity that balls exhibit, with asymptotic behavior for heterogeneous contact reaching roughly 71000 cycles.. Despite the fact that the worn volume is greater at the maximum speed for both ball and disk surfaces, a comparable wear constraint is seen for the other speeds tested in this work. We also notice in Figure 8 for Samples B and C that the SEM results when conducting the wear test showed that the line was significantly and clearly affected for



Sample B compared to Sample C, which shows through the wear path in the SEM

Fig.7 A the uncoated sample with coated samples and fig.7 B shows the coated samples alone wear curves on balls after circular ball-on-disk tests at a speed of 0,015 m.s-1[16].



fig.8 shows the coated samples B and C with various voltage

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## 3.4 Micro Hardness HV

The  $TiO_2$  layer was virtually entirely densified and shown high Vickers hardness, which is practically ceramic-level. The MAO treatment was found to significantly enhance the tribology performance of the Ti6Al4V alloy. As can be seen in Fig. 8, the results indicated that as the thickness of the outer layer rose, the hardness values increased with the increase in voltage from 300 to 320, which significantly improved the micro hardness value.



Fig.8 : Relationship Between Voltage And The Micro-Hardness of Coatings at Tio<sub>2</sub> By MAO Process.

## **Conclusions:**

The objective of this work was to create a new electrolyte for Ti-6Al-4V alloy PEO coating that would streamline the procedure and save expenses. The electrolyte components were chosen from common and affordable resources to achieve this. Because of the straightforward DC generator, low-cost components, low power, and brief working times, the prices were decreased. The dry sliding behaviour of coated Ti-6Al-4V was evaluated, compared with that on the uncoated alloy, and related to microstructural features and mechanical properties of the coating/substrate system. The coatings' tribological characteristics have been improved. Compared to the uncoated substrate, the PEO-coated material lost very little weight. Rutile, anatase, and non-stoichiometric TiO oxide are all components of the coatings created in this study. The key to the coatings' extraordinary wear resistance is their surface and cross-sectional morphology, which possessed a very solid structure without any cracking.

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البنية المجهرية ومقاومة التآكل لطلاءات الأوكسيد الموجودة على سبيكة (Ti-6Al-4V) الناتجة عن الأكسدة الإلكتروليتية بالبلازما باستخدام إلكتروليت رخيص نوار فاهم <sup>(\*</sup> علي هوبي <sup>\*</sup> سمير حميد <sup>\*</sup> <sup>\*,</sup> كلية هندسة المواد-جامعة بابل –العراق \*aboamna299@gmail.com

### الخلاصة:

تم استخدام فئة من المعالجات السطحية ، تُعرف بأكسدة البلازما بالكهرباء ، لتعزيز الخصائص الترايبولوجية للتيتانيوم وسبائكه. تم إجراء بحث مكثف حول الأكسدة الإلكتروليتية للبلازما لسبائك Ti-6Al-4V. يتم استخدام التحليل الطيفي المشتت للطاقة (EDS) ، والمحبور الإلكتروني الماسح (SEM) ، والاختبارات الميكانيكية (الصلادة الدقيقة والبلى) لتحديد خصائص الطلاء والمورفولوجي ، و يتم تحديد المراحل المنتجة. النتائج من التحقيق في طبقة رTi-5 على سبيكة لتحديد خصائص الطلاء والمورفية والبلى) وسبائكه. تم إجراء بحث مكثف حول الأكسدة الإلكتروليتية للبلازما لسبائك Ti-6Al-4V. يتم استخدام التحليل الطيفي المشتت للطاقة (EDS) ، والمحبهر الإلكتروني الماسح (SEM) ، والاختبارات الميكانيكية (الصلادة الدقيقة والبلى) لتحديد خصائص الطلاء والمورفولوجي ، و يتم تحديد المراحل المنتجة. النتائج من التحقيق في طبقة Ti-5 على سبيكة التحديد خصائص الطلاء والمورفولوجي ، و يتم تحديد المراحل المنتجة. النتائج من التحقيق في طبقة Ti-6Al-4V المتحديد خصائص الطلاء والمورفولوجي ، و يتم تحديد المراحل المنتجة. النتائج من التحقيق في طبقة Ti-6Al-4V التحديد خصائص الطلاء والمورفولوجي ، و يتم تحديد المراحل المنتجة. النتائج من التحقيق في طبقة Ti-6Al-4V المنتجة. النتائج من التحقيق في طبقة Ti-6Al-4V المنتجة الماد الماد

الكلمات الدالة: سبيكة Ti-6AI-4V - صلادة فكرز اختبار البلى- أكسدة القوس الصغير ( MAO )