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# Study of tribological characteristics of micro-arc calcium phosphate coatings on titanium

[1*]**Mamaeva A.A.**, [1*]**Kenzhegulov A.K.**, [1]**Panichkin A.V.**, [2]**Panigrahi M.**, [1]**Fischer D.E.**

[1] *Institute of Metallurgy and Ore Beneficiation, Satbayev University, Almaty, Kazakhstan*
[2] *Vellore Institute of Technology, Chennai, Tamil Nadu, India*

* *Corresponding author email: a.kenzhegulov@satbayev.university*

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**ABSTRACT**
Tribological characteristics of implants, such as wear resistance and friction coefficient, play a critical role in ensuring their durability and functionality when interacting with surrounding tissues. These parameters influence the implant's ability to withstand mechanical loads and minimize wear throughout its service life. Minimizing friction between the implant and biological tissues not only helps prevent mechanical damage but also reduces the risk of inflammatory reactions, ensuring better biological compatibility. In this study, calcium phosphate coatings were obtained using the micro-arc oxidation method with different duty cycle of current to investigate their tribological characteristics. The coatings deposited on titanium had a structure with volcano-like formations with pores ranging from 66 to 98 micrometers. The thickness of the coatings varied from 74.3 to 100 micrometers depending on the conditions during microarc oxidation. Tribological tests were conducted using a ball-on-flat setup with reciprocating motion. The coatings were subjected to tribological tests against SHX15 steel under normal loads of 5 and 20 N. Depending on the applied load, the friction coefficients of the coatings ranged from 0.029 to 0.034 at 5 N and from 0.9 to 1.26 at 20 N. Analysis of wear parameters and micrographs of worn surfaces indicate that the mode with a pulse current duty cycle of 17.3% during micro-arc oxidation allows for the production of titanium surface coatings with high wear resistance.

***Keywords:*** calcium phosphate coating, micro-arc oxidation, duty cycle, friction coefficient, wear.

***Information about authors:***

| | |
|---|---|
| ***Mamaeva Axaule Alipovna*** | *Associate professor, Candidate of Physical and Mathematical sciences, Leading Researcher, Institute of Metallurgy and Ore Beneficiation, Satbayev University, Almaty, Kazakhstan. Email: ak78@mail.ru* |
| ***Kenzhegulov Aidar Karaulovich.*** | *PhD, Head of Metal science laboratory of the Institute of Metallurgy and Ore Beneficiation, Satbayev University, Almaty, Kazakhstan. Email: a.kenzhegulov@satbayev.university* |
| ***Panichkin Aleksander Vladimirovich*** | *Candidate of Technical sciences, Head of the National Scientific Collective Use Laboratory of the Institute of Metallurgy and Ore Beneficiation, Satbayev University, Almaty, Kazakhstan. Email: a.panichkin@satbayev.university* |
| ***Panigrahi Mrutyunjay*** | *Associate Professor, School of Mechanical Engineering, Vellore Institute of Technology, Chennai, Tamil Nadu, India. Email: mrutyunjayjapan@gmail.com* |
| ***Fischer Dametken Edilovna*** | *Candidate of Chemistry, Acting head of the Rare Scattered Elements Laboratory of the Institute of Metallurgy and Ore Beneficiation, Satbayev University, Almaty, Kazakhstan. Email: damishka3004@gmail.com* |


## Introduction

Titanium and its alloys are widely used in various industries due to their unique combined properties [[1], [2], [3]]. This versatility is attributed to both mechanical (low elastic modulus, low density, corrosion resistance) and biological characteristics (high bioinertness, bioactivity, and biocompatibility) [[4], [5], [6]], making titanium and its alloys a preferred choice, especially in medical applications as implants. However, despite their valuable properties, there are some clinical issues with titanium implants regarding their interaction with the human body and their long-term usage [[7], [8]]. To address such issues, it is necessary to create biocompatible materials or calcium phosphate coatings that promote the formation of a transitional zone between bone and the implant. This area should have a strong connection to the implant material without the risk of rejection, as well as a macro- and microstructure compatible with the organism [[9], [10]].

Currently, there is a wide range of developed methods for depositing calcium phosphate (CaP) coatings on metallic implants, such as plasma spraying [11], micro-arc oxidation [12], detonation gas spraying [13], magnetron sputtering [14], electrochemical deposition [15], sol-gel, and others.

Considering all the advantages and disadvantages of these methods, the micro-arc oxidation (MAO) method has gained widespread popularity in the last decade as a method for applying bioactive CaP coatings to titanium surfaces. Literature [[16], [17]] notes that the MAO method allows for the production of CaP coatings providing high adhesion strength between the substrate and the coating.

Traditionally, CaP coatings have been used to enhance the osseointegration of metallic implants. However, during the implantation procedure, the coatings are usually subjected to significant shear stresses, which can lead to their delamination and wear from the metal surface, deteriorating their functionality [17]. Therefore, studying the tribological and adhesion properties of MAO coatings requires additional attention and evidence. Analysis of tribological characteristics can help better understand the behavior of biomaterials [5]. Furthermore, titanium alloys limit their further application in medicine due to low wear resistance [[18], [19]].

Recently, there has been growing interest in the scientific community in the tribology of CaP coatings, and several studies have been conducted in this direction. For example, Santos A. and colleagues [5] conducted tribocorrosion research on a titanium oxide and calcium phosphate coating. The $Ti/TiO_2/CaP$ coating provided 99.93% protection in static experiments and significantly reduced corrosion during tribocorrosion tests in simulated body fluid. The application of $TiO_2/CaP$ resulted in reduced tribological parameters: friction coefficient (CoF) to 0.25 and wear rate (WR) to 578.45 $\mu m^3/J$. The work [20] showed that the presence of rutile titanium in the composition of CaP coatings obtained by the MAO method is a key factor in improving the tribocorrosion properties of coating surfaces. The authors explain this by the impact softening properties of the nanocomposite gradient structure of the CaP coating. Marques I.D. and colleagues [21] investigated the influence of the Ca/P ratio in the electrolyte on the tribocorrosion properties of biofunctional coatings obtained by the MAO method on a titanium alloy. The study by [22] examined the phase structure, morphology, chemical composition, corrosion mechanism, and tribocorrosion behavior of CaP coatings obtained at different voltages (380, 400, 420 V). The sample processed at 420 V showed the highest wear volume after tribocorrosion. The mechanism of tribocorrosion of samples at 380 and 420 V was mainly determined as the wear effect.

The results of the aforementioned studies are of high quality and provide valuable information in the field of tribology of CaP coatings, bringing us closer to understanding the real wear of implants in the human body. However, to date, there have been no studies on the influence of current parameters in the micro-arc oxidation method on the tribological characteristics of CaP coatings.

The aim of the study was to investigate the morphology, microstructure, and tribological characteristics of calcium-phosphate coatings depending on the pulse current duty cycle during micro-arc oxidation of titanium.

## Experimental part

*Materials*

Plates measuring 10 mm x 5 mm x 1 mm made of VT1-0 grade titanium (equivalent to titanium GRADE 2) were used as substrates. Prior to oxidation, the substrates underwent mechanical processing. The preparation of the titanium surface for coating included cutting, grinding, degreasing with hexane, and rinsing in distilled water. The electrolyte was prepared with the following composition: a 30% aqueous solution of orthophosphoric acid ($H_3PO_4$, thermal grade A) + hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$ - 60 g/L) + calcium carbonate ($CaCO_3$ - 100 g/L). The hydroxyapatite (HA) was sourced from Sigma Aldrich with a dispersity of <5 µm. The electrolyte was poured into an ultrasonic bath, where it was further mixed with an electric stirrer. To ensure chemical reactions were complete, the electrolyte was allowed to stand in the bath for two days.

*Micro-Arc Oxidation (MAO) process*

A micro-arc oxidation setup was assembled for this work, in which a 150 W stainless steel ultrasonic bath was used as the electrolysis bath. A custom-built pulse power source was employed, capable of varying the voltage from 0 to 700 V with a constant pulse duration of 300 µs, allowing for different duty cycles of 0.0102 (35%), 0.0049 (17,3%), and 0.0022 (8,3%) seconds. MAO was conducted in anodic mode at 300 V with a processing duration of 10 minutes. Three series of experiments were conducted in the electrolyte to perform MAO on the surface of titanium samples with varying pulse current duty cycles (modes A, B, C), as schematically shown in Figure 1. The treated samples were labeled according to modes A, B, and C.

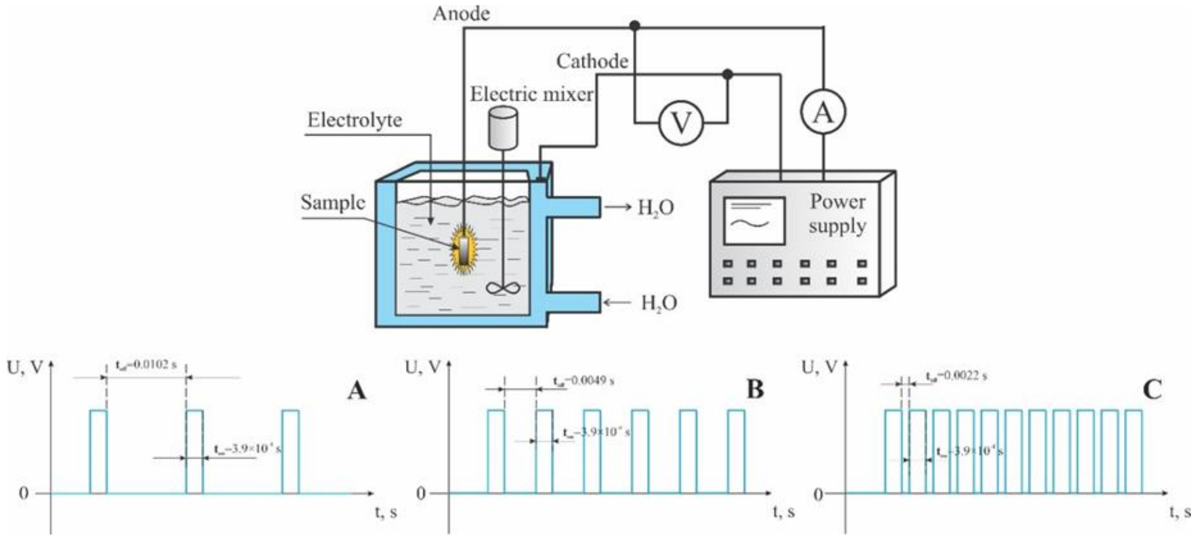


**Figure 1** - Schematic diagram of MAO and characteristics of the electric pulse current applied in different coating synthesis modes

*Methods for Studying CaP Coatings*

Titanium samples after MAO were examined using scanning electron microscopy (SEM) and profilometry, and they were subjected to tribological testing under dry friction conditions at room temperature. Surface morphology and elemental composition analysis, as well as thickness measurements, were carried out using a scanning electron microscope model Phenom ProX with EDS (Phenom, Netherlands) at various magnifications. Surface roughness was measured with a profilometer, the Diavite DH-5 (DIAVITE AG, Switzerland). Each sample was measured 5 times, with the average roughness result from the five measurements reported. The measurement length was 4 mm.

Tribological tests were conducted on a custom-designed setup to determine the friction coefficient (CoF) of samples through reciprocating motion. The tribological test conditions were close to the DIN 51834-1:2010 standard. The setup consisted of a holder for the counter-body with a load, a sample displacement mechanism, an electronic control unit, and a laptop. A 4 mm diameter steel ball made of SHX15 steel was used as the counter-body. Test parameters included: ball displacement speed of 26 mm/sec, friction track length of 5 mm, total friction path length of 5 m, 500 cycles, and loads of 5 and 20 N.

## Results and Discussion

*Morphology, thickness, composition, and roughness of the coating*

CaP coatings exhibit different surface morphologies depending on the duty cycle of the MAO pulse current. Figure 2 shows SEM images of the surface morphology and thickness of the CaP coating obtained at 300 V across all pulse current modes: A, B, and C. In modes A and B, the coating forms a porous structure with spherical aggregates and volcano-like structures with pores. The sizes of spherical aggregates in mode A are 98 ± 7.8 µm, while in mode B they are 66 ± 5.5 µm. Changing the duty cycle to mode C results in not only spherical structures but also walls and fragments thereof. The average size of these structures is 66.5 ± 5.1 µm. Notably, the porous structures (with pore sizes ranging from 15 to 50 µm) promote nutrient circulation in biomedical implant materials [23]. The thickness of the coatings increases from 74.3 µm to 100 µm as the pulse duty cycle decreases, likely due to a greater number of pulses (from A to C), leading to thicker CaP coatings in MAO.

The elemental composition of the CaP coating shows the presence of Ca, P, O, and Ti, as shown in Table 1. All samples exhibit a significant increase in oxygen content, which is a characteristic of the MAO method. The Ca/P ratio is a key indicator of bioactivity. Given this parameter, the coatings produced are likely related to bioresorbable phases. These could include CaP coatings with a lower Ca/P ratio (Ca/P = 1.67) compared to HA [24]. As the duty cycle decreases (from A to C), the Ca/P ratio increases from 0.3 to 0.4. This might be due to intensified deposition of Ca2+ ions from the electrolyte. Additionally, as the coating thickness increases, the titanium content decreases while the calcium content increases in elemental analysis with changes in the MAO pulse current duty cycle.

The surface roughness of an implant is one of the critical characteristics during osteointegration. According to the literature [[25], [26]], the roughness parameter Ra in the range of 4 < Ra < 7 µm is considered optimal, as it promotes enhanced osteogenic differentiation on such rough titanium surfaces. The obtained CaP coatings had roughness within this range, indicating that depending on the application mode, the roughness parameter Ra was recorded within the range of 5.7 to 6.11 µm. The other roughness parameters are shown in Table 2.

*Tribological study of CaP coatings*

During tribological testing, the surface of the CaP coating in contact with the SHX15 steel counter-body underwent degradation, leading to the formation of wear tracks. Figure 3 shows typical morphologies of worn coating surfaces after tribological tests at a load of 20 N against a steel ball for different MAO application modes. At a 5 N load, wear tracks were barely noticeable, so to study the wear characteristics in greater detail, tracks formed at a 20 N load were selected. As a result, extensive wear tracks were observed on the worn surfaces.

The wear tracks for the sample processed in mode A exhibited two distinct zones: the first was a wear track with surface layer removal, and the second had pits corresponding to locations where the coating delaminated due to intense wear. The width of the wear track in the first zone was 488 ± 65 µm, while in the second zone, it was 234 ± 66 µm. Among the three coatings studied, the sample processed in mode A showed the lowest wear resistance, as indicated by the greater depth of the wear track. One reason for this low resistance could be the higher surface roughness [[27], [28]]. In contrast, the tribological behavior of the other coatings showed a different wear pattern. As shown in Figure 3, the coatings obtained in modes B and C exhibited surface wear without noticeable exposed substrate. The width of the wear tracks for these coatings was 538 ± 138 µm for mode B and 745 ± 191 µm for mode C.

Roughness measurements using SEM within the wear tracks were 3.63 µm for the mode A coating, 585 nm for the mode B coating, and 947 nm for the mode C coating. This data helps characterize the extent of wear-related roughness. A visual comparison of surface degradation, wear pattern, and wear volume after tribological testing suggests that the coating produced in mode B has the highest wear resistance among those studied, supported by the absence of delamination and a narrower wear track.

**Table 1** - Elemental composition of calcium phosphate coatings on titanium and the Ca/P ratio

| Coating | Elemental composition (ат. %) | | | | Ca/P |
|---|---|---|---|---|---|
| | Ca | P | O | Ti | |
| A-300 V | 3.26 | 10.52 | 77.33 | 8.89 | 0.3 |
| B-300 V | 3.50 | 10.38 | 77.43 | 8.69 | 0.34 |
| C-300 V | 4.60 | 11.37 | 78.13 | 5.90 | 0.4 |

**Table 2** - Parameters for measuring the surface roughness of CaP coatings deposited with different duty cycles in MAO

| Coating | Surface roughness measurement parameters | | | | | |
|---|---|---|---|---|---|---|
| | Mean arithmetic deviation of profile, Ra (µm) | Profile roughness height across ten points, Rz (µm) | Maximum profile height, Rmax (µm) | R3z, (µm) | Rt, (µm) | Rq, (µm) |
| A | 6.11 | 35.5 | 43.5 | 25.6 | 43.5 | 7.75 |
| B | 5.70 | 30.7 | 40.6 | 26.5 | 40.6 | 7.11 |
| C | 5.76 | 20.0 | 20.0 | 20.0 | 20.0 | 6.62 |

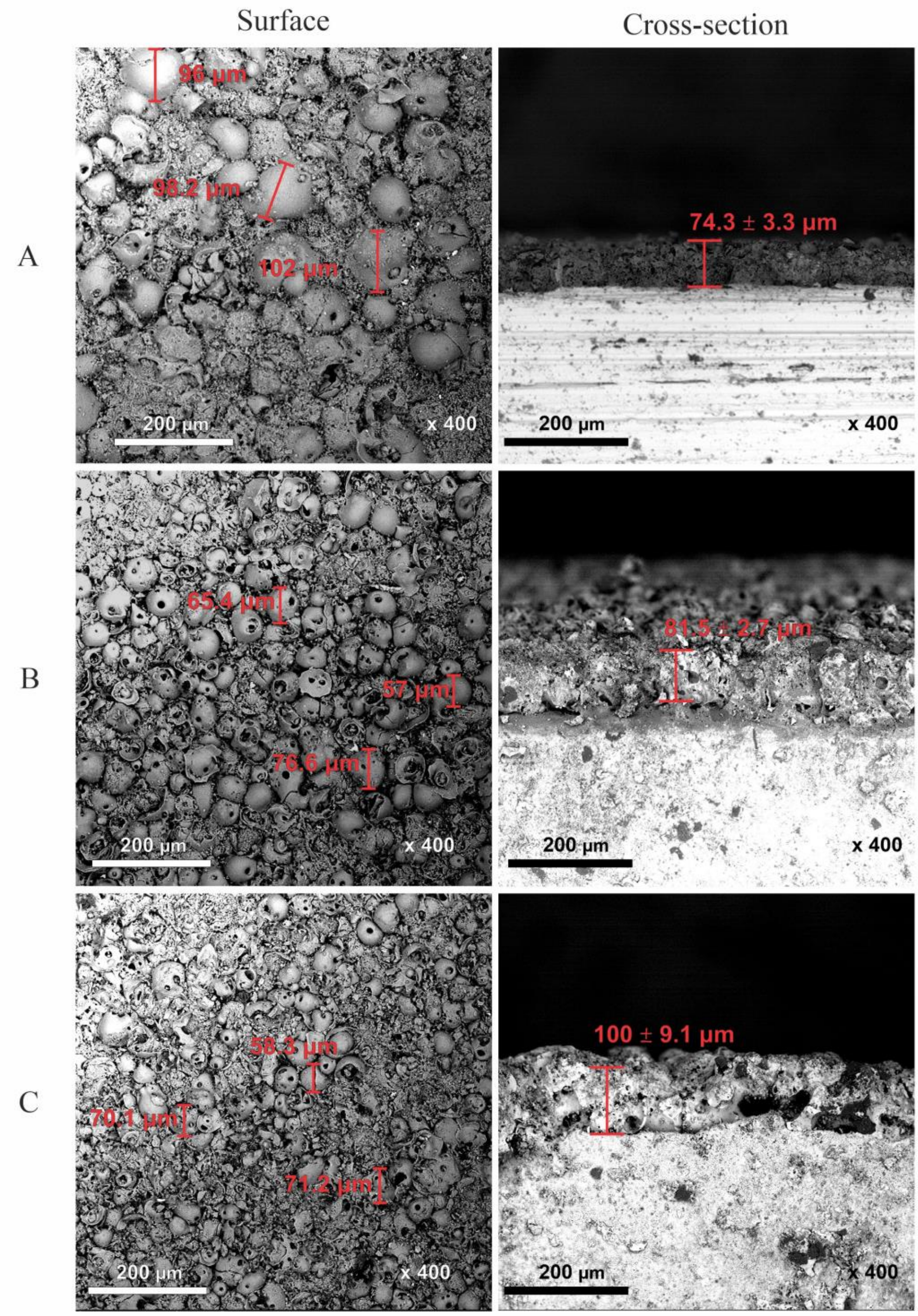


**Figure 2** - Surface morphology and thickness of CaP coatings obtained in duty cycle modes: A, B, and C

According to the friction test results (Figure 4), the coefficient of friction (CoF) in all studied coatings showed stable behavior, except for minor spikes or changes. During tests with a 5 N load (depicted by the black line in Figure 5), there were initial minor spikes in the CoF graphs, indicating a break-in period for the surface. The break-in period could be due to the smoothing of surface roughness peaks on the coatings [29]. Increasing the normal load from 5 to 20 N led to a higher CoF. These results indicate a significant influence of load on the wear response of the materials studied. As for the CoF graphs at a 20 N load (depicted by the red line in Figure 5), after 50 cycles of friction, if a decrease in CoF is observed for coatings deposited in modes B and C, there is an increase in CoF for mode A, which continues up to 200 cycles. A possible reason for this might be the delamination of ceramic material particles during tribological testing, which increases the surface roughness and consequently leads to higher CoF values [17].

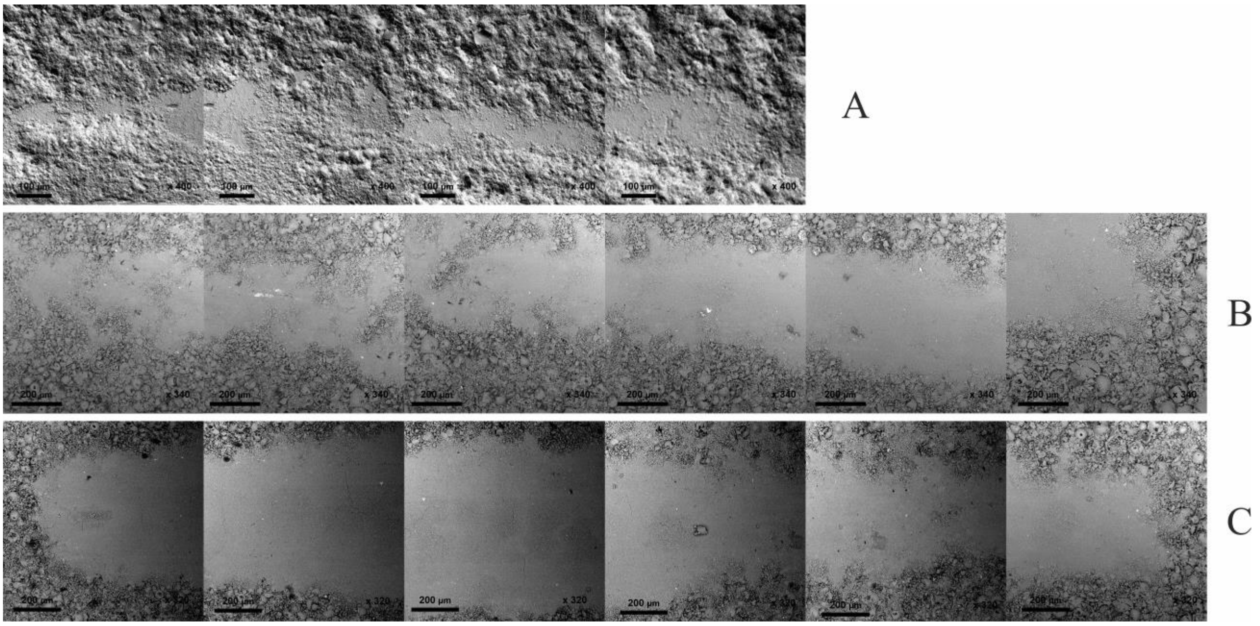


**Figure 3** - SEM Images of wear tracks after tribological testing of CaP coatings deposited in duty cycle modes: A, B, and C

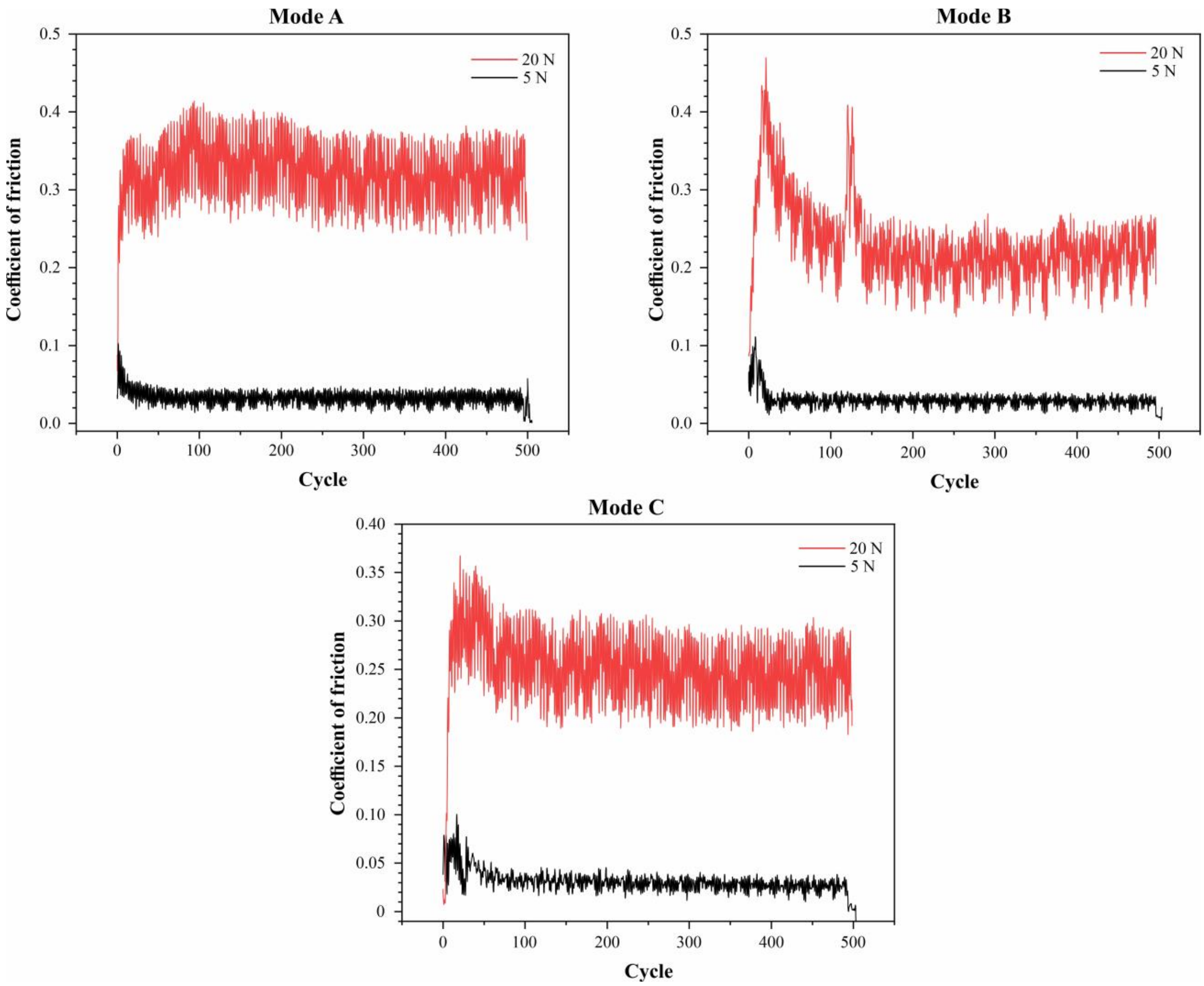


**Figure 4** - CoF graph for CaP coatings deposited in duty cycle modes: A, B, and C

Figure 5 shows the averaged values of the CoF at 5 N and 20 N loads and the wear track width for the CaP coatings. The lowest average CoF is observed for the CaP coating obtained in Mode B of the MAO process, with a CoF of 0.029 at 5 N and 0.909 at 20 N. The highest CoF, 0.034, is noted for the coating in Mode A. The median CoF values for all coatings were as follows: A 5N – 0.034, A 20N – 1.396, B 5N – 0.026, B 20N – 0.89, C 5N – 0.024, C 20N – 1.058. When comparing wear track width, the coating in Mode B demonstrates the highest wear resistance due to its narrower width at 5 N. For 20 N, the narrowest wear track is recorded for the coating in Mode A at 488 µm. However, it's important to note that this coating also exhibits deeper wear grooves. Consequently, when considering wear volume, the coating in Mode B displays lower wear than other coatings. In summary, the tribological data suggest that Mode B of the MAO process allows for the deposition coating with high wear resistance on titanium surfaces.

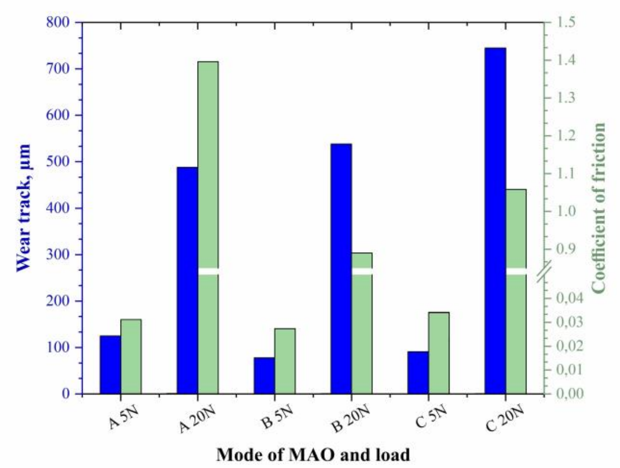


**Figure 5** - The wear track and the averaged CoF values for loads of 5 N and 20 N

Coatings applied in mode B exhibit less wear and better integrity retention under load compared to other modes, which can be explained by several reasons outlined below. Mode B results in the formation of smaller spherical aggregates (66 ± 5.5 µm) compared to mode A and, unlike mode C, prevents the formation of fragmented structures. The consistency and uniformity of these smaller spherical aggregates may contribute to the creation of a more compact and homogeneous coating, thereby enhancing its wear resistance. Transitioning from mode A to B increases the calcium content, which contributes to structural integrity through a denser phosphate matrix. Consequently, mode B likely induces beneficial structural changes, such as a more uniform and compact microstructure, optimal roughness, and improved chemical composition, enhancing its wear resistance.

## Conclusions

The micro-arc oxidation method successfully applied calcium-phosphate coatings to the VT1-0 titanium surface with varying pulse duty cycles in an electrolyte containing a 30% aqueous solution of orthophosphoric acid, hydroxyapatite, and calcium carbonate. The coatings underwent tribological testing against SHX15 steel under normal loads of 5 N and 20 N. From the study, the following conclusions can be drawn:

1. Investigation into the morphology of coatings resulting from MAO with various pulse duty cycles on the titanium surface showed a common structure featuring volcano-like formations with pores. The average size of these structures ranged from 66 to 98 micrometers. Decreasing the pulse duty cycle (from 35% to 8,3%) caused the coating thickness to increase from 74.3 micrometers to 100 micrometers due to the higher number of pulses.
2. The surface roughness of the coatings fell within the optimal range of 5.7 to 6.11 micrometers, conducive to enhanced osteogenic differentiation. Following tribological testing, the roughness within the wear track decreased by 6 to 10 times compared to the initial condition.
3. The Ca/P ratio, a critical bioactivity indicator, varies with changes in the MAO pulse current duty cycle. A decrease in this ratio is observed as the duty cycle increases, suggesting an enhancement in the bioactive potential of the coatings. The presence of bioresorbable phases in the coatings indicates their suitability for biomedical implant applications, particularly where nutrient circulation within the implant material is crucial.
4. Wear resistance studies showed that the coating produced in duty cycle 17.3% had the highest wear resistance among the coatings tested. The absence of delamination, the narrow wear track width, and a favorable coefficient of friction indicated the high wear resistance of the coatings obtained in this mode. This is attributed to the smaller, more uniform spherical aggregates formed, which result in a more compact and homogenous layer, offering better protection against wear. The wear patterns and friction coefficient data corroborate the superior performance of MAO mode (voltage 300 V, duty cycle 17.3%), where the coatings exhibit minimal delamination and maintain structural integrity even under higher loads.

**Conflicts of interest.** On behalf of all authors, the corresponding author states that there is no conflict of interest.

***CRediT author statement***
**A. Mamaeva**: Conceptualization, Validation, Investigation, Project administration. **A. Kenzhegulov**: Methodology, Software, Validation, Investigation, Data curation, Writing - Original Draft, Visualization. **A. Panichkin**: Conceptualization, Validation, Visualization, Investigation. **M. Panigrahi**: Writing - Review & Editing. **D. Fischer**: Software, Resources, Funding acquisition.

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# Титандағы микродоғалы кальций-фосфат жабындарының трибологиялық сипаттамаларын зерттеу

[1] Мамаева А.Ә., [1] Кенжегулов А.Қ., [1] Паничкин А.В., [2]Panigrahi M., [1] Фишер Д.Е.

*[1] Металлургия және кен байыту институты, Сәтбаев университеті, Алматы, Қазақстан*
*[2]Веллор Технологиялық Институты, Ченнаи, Тамил Наду, Үндістан*

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**ТҮЙІНДЕМЕ**
Импланттардың трибологиялық сипаттамалары, мысалы, тозуға төзімділігі және үйкеліс коэффициенті, олардың қоршаған тіндермен өзара әрекеттесу кезінде төзімділігі мен функционалдылығын қамтамасыз етуде маңызды рөл атқарады. Бұл параметрлер имплантаттың механикалық жүктемелерге төтеп беру қабілетіне және оның қызмет ету мерзімін ұлғайтуға, тозуды минималдауға ықпал етеді. Имплантат пен биологиялық тіндер арасындағы үйкелісті минималдау механикалық зақымданудың алдын алуға ғана емес, сонымен бірге қабыну реакцияларының қаупін төмендетуге көмектеседі, бұл жақсы биологиялық үйлесімділікті қамтамасыз етеді. Бұл жұмыста олардың трибологиялық сипаттамаларын зерттеу үшін импульстік токтың әртүрлі жұмыс циклдерінде микродоғалық тотығу арқылы кальций фосфатты жабындары алынды. Титанға отырғызылған жабындар 66-дан 98 микрометрге дейінгі вулкан тәрізді тесіктері бар құрылымдармен болды. Жабындардың қалыңдығы микродоғалық тотығу кезінде алынған жағдайға байланысты 74,3-тен 100 микрометрге дейін өзгерді. Трибологиялық сынақтар шар-жазықтық схемасы бойынша қайтымды ілгерілеме қозғалыс арқылы жүргізілді. Жабындар 5 және 20 Н қалыпты жүктемелерде ShKh15 маркалы болатқа қарсы трибологиялық сынақтардан өтті. Түсірілген күшке байланысты жабындардың үйкеліс коэффициенттері 5 Н кезінде 0,029-дан 0,034-ке дейін және 20 Н кезінде 0,9-дан 1,26-ға дейін болды. Тозу көрсеткіштерін талдау және тозған беттердің микрокескіндері микродоғалық тотығу кезінде импульстік токтың импульсті жұмыс циклі 17,3% болатын режимінің титан беттерінде тозуға төзімділігі жоғары жабындарды алуға мүмкіндік беретінін көрсетті.

***Түйін сөздер:*** кальций-фосфат жабыны, микродоғалық тотығу, ток қуыстылығы, үйкеліс коэффициенті, тозу.

***Авторлар туралы ақпарат:***

| | |
|---|---|
| ***Мамаева Ақсәуле Әліпқызы*** | *Қауымдастырылған профессор, Физика-математика ғылымдарының кандидаты, жетекші ғылыми қызметкер, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан. Email: ak78@mail.ru* |
| ***Кенжеғұлов Айдар Қарауылұлы*** | *PhD, Металтану зертханасының меңгерушісі, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан. Email: a.kenzhegulov@satbayev.university* |
| ***Паничкин Александр Владмимрович*** | *Техника ғылымдарының кандидаты, Ұжымдық пайдаланылатын Ұлттық ғылыми зертханасының меңгерушісі, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан. Email: a.panichkin@satbayev.university* |
| ***Panigrahi Mrutyunjay*** | *Қауымдастырылған профессор, Механикалық инженерия мектебі, Веллор Технологиялық Институты, Ченнаи, Тамил Наду, Үндістан. Email: mrutyunjayjapan@gmail.com* |
| ***Фишер Даметкен Едиловна*** | *Химия ғылымдарының кандидаты, Сирек шашыраңқы элементтер зертханасы меңгерушісінің міндетін атқарушы, «Металлургия және кен байыту институты» АҚ, Сәтбаев университеті, Алматы, Қазақстан. Email: damishka3004@gmail.com* |

# Исследование трибологических характеристик микродуговых кальций-фосфатных покрытий на титана

[1] Мамаева А.А., [1] Кенжегулов А.К., [1] Паничкин А.В., [2]Panigrahi M., [1] Фишер Д.Е.

*[1] Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан*
*[2]Веллорский технологический институт, Ченнаи, Тамил Наду, Индия*

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**АННОТАЦИЯ**

Трибологические характеристики имплантатов, такие как сопротивление износу и коэффициент трения, играют критическую роль в обеспечении их долговечности и функциональности при взаимодействии с окружающими тканями. Эти параметры влияют на способность имплантата выдерживать механические нагрузки и минимизировать износ в течение всего срока его службы. Минимизация трения между имплантатом и биологическими тканями не только помогает предотвратить механические повреждения, но и снижает риск воспалительных реакций, обеспечивая лучшую биологическую совместимость. В данной работе были получены кальций-фосфатные покрытия методом микродугового оксидирования при разной скважности импульсного тока для исследования их трибологических характеристик. Осажденные покрытия на титане имели структуру с вулканоподобными образованиями с порами от 66 до 98 микрометров. Толщина покрытий варьировалась от 74,3 до 100 микрометров в зависимости от условия получения при микродуговом оксидировании. Трибологические испытания проводились по схеме шар-плоскость с помощью возвратно-поступательного движения. Покрытия были подвергнуты трибологическим испытаниям против стали ШХ15 при нормальных нагрузках 5 и 20 Н. В зависимости от приложенной нагрузки коэффициенты трения покрытий составляли от 0,029 до 0,034 при 5 Н и от 0,9 до 1,26 при 20 Н. Анализ показателей износа и микрофотографии изношенных поверхностей свидетельствуют о том, что режим со скважностью импульсного тока 17,3 % при микродуговом оксидировании позволяет получать на поверхностях титана покрытия с высокой износостойкостью.

***Ключевые слова:*** кальций-фосфатное покрытие, микродуговое оксидирование, скважность тока, коэффициент трения, износ.

| | ***Информация об авторах:*** |
|---|---|
| ***Мамаева Аксауле Алиповна*** | *Ассоциированный профессор, кандидат физико-математических наук, ведущий научный сотрудник, Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан. Email: ak78@mail.ru* |
| ***Кенжегулов Айдар Караулович*** | *PhD, Заведующий лабораторией металловедение, Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан. Email: a.kenzhegulov@satbayev.university* |
| ***Паничкин Александр Владмимрович*** | *Кандидат технических наук, заведующий Национальной научной лабораторией коллективного пользования, Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан. Email: a.panichkin@satbayev.university* |
| ***Panigrahi Mrutyunjay*** | *Ассоциированный профессор, Школа механической инженерии, Веллорский технологический институт, Ченнаи, Тамил Наду, Индия. Email: mrutyunjayjapan@gmail.com* |
| ***Фишер Даметкен Едиловна*** | *Кандидат химических наук, Исполняющий обязанности заведующего лабораторией редких рассеянных элементов, Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан. Email: damishka3004@gmail.com* |

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