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Abrasion resistance of titanium-based biofunctionalised oxide ceramic coatings

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Abstract. The requirements of modern production for details are very high. Particular attention is paid to the friction resistance parts that work in the friction mechanism. Titanium alloys are characterised by low wear resistance and have the ability to stick to the surface of the body of greater hardness. Therefore, applying wear-resistant coatings to the surface of titanium alloys is relevant. The purpose of this work was to increase the operational properties of titanium alloys by creating protective coatings on their surface. The work investigated the wear resistance of plasma electrolyte oxidised coatings on titanium alloy. Experimental studies revealed that the coatings have different oxides (stoichiometric and non-stoichiometric) in their structure. Non-stoichiometric oxides are characterised by low physical and mechanical properties and settle in the pores after primary wear. In the process of wear, a non-stoichiometric oxide serves as a lubricant. This has a positive effect on their tribological properties. It has been established that applying a coating to the surface of a titanium alloy increases the abrasion resistance by 10 times. The interface between the metal base and the coating is uneven, which is achieved due to the melting of the surface of the titanium alloy during PEO. This feature demonstrates the high adhesion properties of coatings formed by the PEO method to the titanium base. The paper established the dependence of the size and shape of the pores on the working modes. It was found that the size of the pores on the surface of the coating is 10…70 μm, and high internal porosity of the coating material was established. It was found that porous coatings melt internally, so the porosity is open, not through. The studied property of the coatings can help in implantology, since the connection of muscle tissue with the implant occurs without the formation of a capsule

Keywords: phosphates; friction; plasma electrolytic oxidation; durability; wear; porosity

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Introduction

Changing the surface composition is one of the most promising methods of increasing the resistance to surface activation and mechanical properties of light alloys. A. Kurup *et al.* (2021) explained in their work the acceleration of the osseointegration process of titanium alloys that have a coating with complex morphology. The authors investigated the coating by the plasma electrolytic oxidation method. The uniqueness of this method lies in the ability to change the properties of the surface by changing the working modes of the synthesis. Researchers K. Babaei *et al.* (2020) investigated the effect of carbon allotropes in the form of carbon nanotubes, graphite, diamond, and graphene in an electrolyte solution. Increased corrosion and wear resistance of coatings synthesised on the basis of aluminium has been established. G. Mortazavi *et al.* (2019) showed, how the value of the current affects the porosity of the coating. The authors indicated that a higher current makes it possible to obtain a coating with a higher amount of porosity and a higher roughness. This is achieved due to a large number of spark discharges that occur on the surface. This is achieved due to a large number of spark discharges that occur on the surface. High temperature leads to the formation of both stable rutile and metastable anatase. Increasing the current density makes it possible to obtain a coating with stable amorphous dense anatase. It was established that the thickness of the oxide ceramic coatings on the titanium alloy increases due to the increase in the current density.

The authors M. Riabchykov *et al.* (2023b) aimed to create a coating on a titanium alloy for biomedical purposes. Therefore, coating materials should be very porous. Researchers H. Fakhr Nabavi & M. Aliofkhazraei (2019) showed in their research that coatings with high porosity and roughness applied to titanium alloy by the PEO method make it biocompatible with the living organism. The authors achieve an increase in the biocompatibility of coatings by adjusting the phases of rutile and anatase in the coating material. M. Riabchykov *et al.* (2023a) in their work indicated the higher porosity of materials, when using nanocomponents that would be added to them. Therefore, adding nanoparticles of biocomponents to the alkaline electrolyte is completely justified.

The scientists P. Bocchetta *et al.* (2021) described the problems of using titanium alloys Ti-6Al-4V in implantology due to the presence of toxic components such as Al and V. It is known that after oxidation, layers with high porosity are formed on titanium alloys. With the PEO method can control the amount of titanium oxide phases and their modification. Yu. Yuferov & K. Borodyanskyi (2024) explained the importance of Ca/P content in PEO coatings. The introduction of Ca and P particles in the form of ammonium dihydrogen phosphate into the electrolyte was investigated. The morphology, chemical and phase composition of the coatings are characterised by high biocompatibility. Therefore, adding biocomponents to the coating can increase their biocompatibility. N. Imbirovych *et al.* (2023) found that the introduction of diatomite and hydroxyapatite into a ceramic coating on a titanium alloy makes it rougher. These properties have a positive effect on the excellent implantation and growth of the implant with muscle tissue.

In 2024, the field of implantology has developed greatly, but there are still some problems. In practice, it is very important, which implant system the doctor uses, as well as its design. N. Usmaniya *et al.* (2024) described in their work that with poor-quality processing of titanium alloy and an undeveloped design, its atoms migrate into the surrounding bone tissue, causing its fibrous degeneration (damage to bone tissue). Later, it became clear that in such cases, the implants are rejected quite quickly. This direction of research refers to the problem of increasing the biocompatibility of surfaces, therefore, innovative metal processing was applied, such as plasma electrolytic oxidation (PEO).

The purpose of this work was to develop biocompatible coatings by the method of plasma-electrolytic oxidation in alkaline silicate-phosphate electrolytes with the addition of hydroxyapatite and diatomite. As a result of adjusting the ratio of current density and concentration of electrolyte components, it will be possible to obtain coatings with high physical and mechanical properties and with a highly developed surface.

Materials and Methods

The process of plasma-electrolytic oxidation was carried out in the anode-cathode mode with different modes of operation. When the sample is oxidised, the voltage at the anode gradually changes. The time, when the first spark starts depends on the composition of the electrolyte and the current to be set.

As a result, a discharge occurs between the sample and the electrolyte, and hydrogen begins to be released at the anode. The breakdown of the primary film on the sample under study leads to the formation of discharge channels in which high-temperature plasma-electrolyte reactions occur. Titanium interacts with oxygen, forming gaseous and solid/liquid phases Ti, TiO, TiO_2. During plasma-chemical reactions in an alkaline medium (electrolyte), narrow channels with increased ionic conductivity are formed on certain areas of the sample surface. Such areas become foci of local damage to the alloy, namely the barrier oxide film that forms on the surface of the metal under natural conditions. A spark on the surface of a titanium alloy is shown in Figure 1. This result indicates a discontinuity of the semiconductor field.

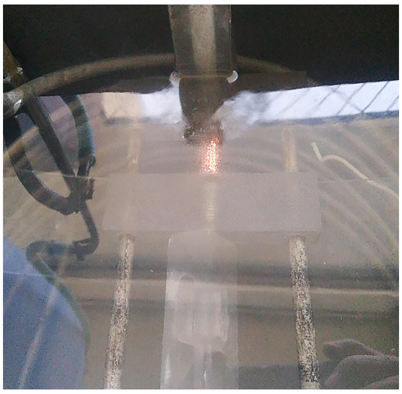


Figure 1. *Synthesis of an oxide ceramic coating in a spark discharge plasma on a titanium alloy sample*
Source: *developed by the authors*

Working electrolytes were solutions based on distilled water containing potassium hydroxide, calcium, liquid glass, sodium pyrophosphate and polyphosphate, diatomite and hydroxyapatite with a concentration of components from 3 to 20 g/l. The ratio of cathodic to anodic current density was for the first sample Ia/Ic = 1A/dm^2. The oxidation time was 30 min. The investigated effects of the composition of the electrolyte were recently changed according to the conditions of the experiment. After the synthesis, the samples were washed and dried.

Experiments on friction with a reversibly forward movement on titanium alloy samples were carried out at the installation. The principle of operation of the installation involved reversible friction of the sample by its reciprocating movement in relation to the counterbody. A flat sample is fixed on the slider of the crank mechanism. The sample was loaded of force from 0.2 to 1.5 kg through a vertical rod. The contact surface area of the sample was 0.8 cm^2. The counterbody material is alloy 2024 with an Al_2O_3 coating 150 thick and a microhardness of 1500 MPa, formed in an electrolytic plasma. The kind of friction was dry.

The surface after synthesis of plasma electrolytic oxidation coatings was observed using a Tagarno Prestige FHD Controller XPlus microscope (Denmark) connected to a computer. The magnification was more than 600 times, with high frame rate and high image resolution, with manual and autofocus magnification and with an automated zoom scale.

Results and Discussion

The coating formation process occurs within an electric field that supplies energy for plasma-chemical reactions on the working electrode. When the anode potential exceeds the Faraday zone, the dielectric or semiconductor film on the anode undergoes breakdown. This breakdown results from electron injection from the valence band into the conduction band. Within the breakdown channel, a significant temperature increase occurs, leading to electrolyte ionisation and plasma formation, where plasma-chemical reactions drive oxide synthesis.

Notably, the introduction of diatomite helps stabilise the synthesis process, as the voltage remains nearly constant due to the higher silicate content in the solution, which enhances electrolyte conductivity. Increased electrical conductivity in the electrolyte reduces the required synthesis voltage. Nevertheless, the voltage generated at the anode and the resulting current density are adequate to form craters surrounding the pores. These properties increase the biocompatibility of the coating (Imbirovych *et al.*, 2023).

Due to its non-toxic properties, excellent biocompatibility and strength properties, titanium alloys are suitable for use in implantology. L. Chen *et al.* (2018) investigated the wear of titanium alloys and explained their limitations in friction units, as they have the ability to stick to the body, so "scratches" appear during the friction process, which limits their use in friction units. In Figure 2, it can see "streams" of metals remaining after contact with the counterbody in the friction process.

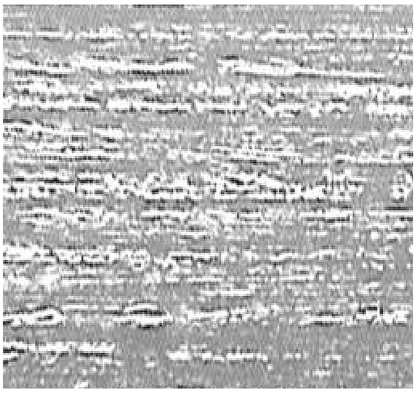


Figure 2. *Titanium alloy after wear resistance tests*
Source: *developed by the authors*

This effect is negative of mechanical nodes, therefore the formation of a strong surface of the structural state, which is characterised by high hardness, is relevant. L.M. Elias *et al.* (2006) developed titanium alloys with Nb and investigated their mechanical properties. It was found that the mechanical properties of these alloys are suitable for the use in implantology. M. Geetha *et al.* (2009) discussed the demand for titanium alloys in implantology. Their good combination of strength and density promotes biocompatibility. Thus, modification of the state of the surface with bioelements to increase roughness has a positive effect and leads to rapid growth of human tissue with an implant. X. Lu *et al.* (2015) proposed the idea of introducing reactive particles into the coating by adding them to the electrolyte. This made it possible to seal the porosity of coatings and expand the possibilities of creating compositions by the PEO method. J. Qin *et al*. (2022) investigated the effect of graphene nanoparticles on the mechanical properties and porosity of PEO coatings on magnesium alloys. They found an increase in hardness, wear resistance, and porosity by adding nanoparticles to the magnesium-based coating.

Based on the results of the research conducted by scientists, a coating with hydroxyapatite and diatomite particles was developed (Fig. 3). The obtaining coating is characterised by a large number of pores on the surface. It should be noted that the pores form craters above themselves, which form the roughness of the surface (Fig. 3, a). These pores are of different sizes and shapes (Fig. 3, b). The pores can be examined in more detail at higher magnification (Fig. 3, c and d). Pores in the coating are formed due to a spark discharge. In the future, the coating grows and the pores overlap each other. This leads to closed porosity. This effect is very positive for the rapid introduction of muscle tissue into the implant. Therefore, implants with a similar surface are not rejected by the living organism. This leads to high biocompatibility of the implant material.

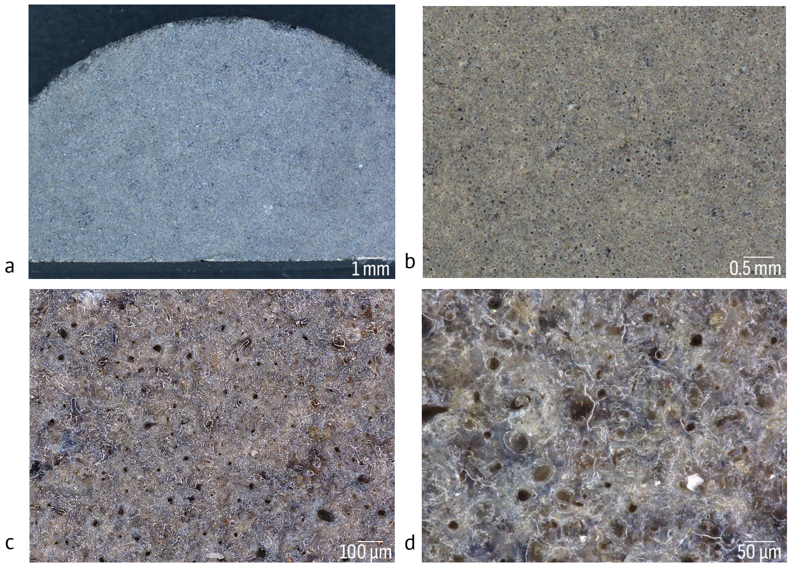


Figure 3. *Oxide ceramic coatings synthesised by PEO on a titanium base at different magnifications*

Note: *a, b, c, d – pores on the surface of the coating at different magnifications*

Source: *developed by the authors*

After the synthesised coating, a brittle oxide may appear on their surface, which easily falls off. At minimal loads, the oxide crumbles easily and closes the pores. In addition, this non-stoichiometric oxide behaves as a lubricant under friction conditions in the future. Therefore, an oxide with low mechanical properties has a positive effect on wear resistance.

In the future, studies of the wear resistance of the obtained coatings were carried out. Figure 4 shows the surfaces of the coatings, which were formed as a result of the synthesis of the titanium alloy, before and after friction. From the conducted research and visual inspection of the surface, it becomes clear that the roughness of the surface, which is determined by the craters of the pores, has a positive effect on the wear resistance of the surface of the coating material. By appearance, it can observe a change in the surface before and after friction. Smoothing the surface after wear leads to a reduction in its thickness and removal of the roughness that was formed as a result of the PEO process. From the conducted research, it follows that starting from the first stages of the friction process, smoothing occurs, during which the upper, non-stoichiometric oxide layer (anatase), which clogs the pores on the surface, is quickly erased. At the next stage, the friction coating becomes smooth, and wear practically stops. Lower is a study of the wear resistance of the coating obtained on a titanium alloy at different distances between the cathode and anode into the electrolyte without diatomite (modes 1 and 2 in Table 1) and with it (mode 3 in Table 1).

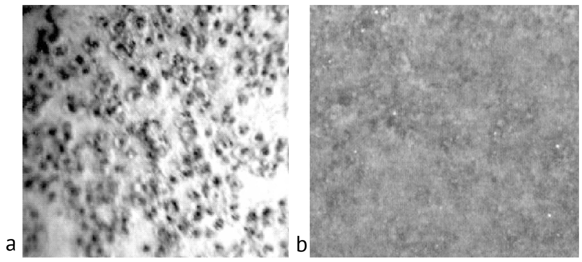


Figure 4. *External pores on the coating after oxidation of the titanium alloy by the PEO method (a); coating after friction tests (b) (x150)*

Source: *developed by the authors*

Figure 5 shows the samples that were synthesised in order to establish their wear resistance. As a result of tests of samples from the studied synthesised alloy of Grade 5, traces of wear were obtained. As a result of testing the samples, the wear intensity of the coating surface under the given conditions was established.

Figure 5. *Traces of coating wear after friction tests*
Source: *developed by the authors*

The obtained results show the intensity of wear of coatings during the first 500 m for the synthesised surface with phosphates and calcium of about 0.12...0.1 μm/m. The addition of diatomite to the electrolyte (mode 3) characterises the lowest wear of the coating in this area and further. For systems with phosphates and calcium, from a distance of 500 m to 1000 m, the wear of coatings increases almost twice. This result demonstrates a greater lapping distance compared to the coating that was synthesised in the electrolyte using diatomite. As a result of the experiment, it was found that the wear of the coating synthesised in an electrolyte with sodium pyrophosphate (electrolyte No. 1) is somewhat higher compared to the coating synthesised in an electrolyte without phosphate components (electrolyte No. 2) (Table 1).

Table 1. *Wear intensity of titanium-based coatings in different working environments*

No. composition	Electrolyte composition	Intensity of PEO-coating wear *I*, μm /m			
		L, m 500	L, m 1000	L, m 1500	L, m 2500
1	0.5 g/l KOH+0.5 g/l l.g.+0.5 g/l $Ca(OH)_2$+0.5 g/l $Na_4P_2O_7$	0.12	0.25	0.2	0.19
2	3 g/l KOH+2 g/l l.g.+3 g/l $Ca(OH)_2$	0.1	0.18	0.175	0.17
3	20 g/l KOH+20 g/l l.g.+20 g/l $Ca(OH)_2$+20 g/l $Na_4P_2O_7$+20 g/l $Na_6P_6O_{18}$+20 g/l diatomite	0.092	0.09	0.052	0.03

Source: *developed by the authors*

The lowest value of the friction intensity during the grinding process is characteristic of the coating synthesised by electrolysis with diatomite, and is equal to 0.03 μm/m at the largest distance traveled, which is 2500 m. In the course of the experiment, it was established that as a result of surface treatment of Grade 5 titanium alloy, coatings are synthesised that are characterised by high wear resistance, which was evaluated by dry friction (Table 1). Thus, at the first stage of abrasion (Fig. 6) v_f is the largest for oxide ceramics synthesised according to mode 1. Therefore, secondary structures on the counterbody develop faster here. According to the obtained results of wear of bio-coatings, it was established that after 1000 m of the covered path, the coating wears off and the rate of wear of the coating decreases sharply. Thus, coatings synthesised in an electrolyte No. 1 wear out at an average rate 0.076 μm/m, the coatings synthesised in an electrolyte with a composition of No. 2 are worn by 0.17 μm after a 2500 m path, and therefore the speed wear of such coatings is 0.068 μm/m. The lowest rate of wear is characteristic of coatings synthesised in the electrolyte according to modes No. 3 since the rate of wear of coatings is 0.012 μm/m (Fig. 6). At the next stage of friction, the intensity of wear increases for the two systems. The coating, oxidised in an electrolyte with a higher content of components and additionally with diatomite wears out with less intensity.

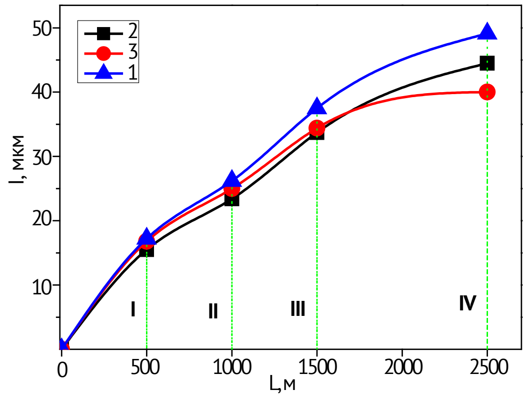


Figure 6. *The wear intensity I of the coating on the titanium alloy from the travelled distance L*
Source: *developed by the authors*

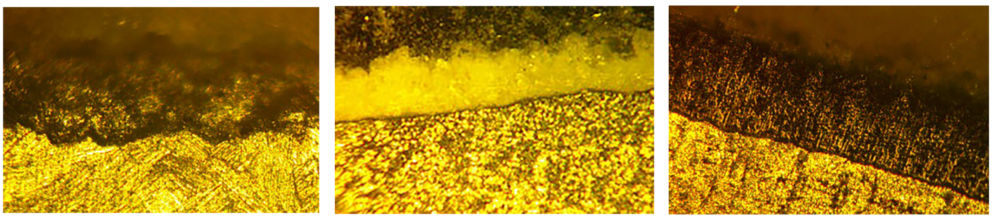
In the third and fourth stages, the intensity of wear decreases for these systems. However, the lowest value of wear intensity is for the coating, which was synthesised according to mode No. 2, which does not contain sulfates and bio-additives. After rubbing the surface to the counterbody, the angle of inclination of all the curves shown in Figure 5 decreases. For the sample, which was oxidised in an electrolyte containing KOH, liquid glass, and diatomite, after grinding, a smear is formed that contains a horizontal section, which indicates the high wear resistance of such a coating. The intensity in this area is 0.001. The rate of wear of biocoatings after each stage of the distance is presented in Table 2. Experimental results show that after 500 m of travel, the coating containing datomite in its layer has the lowest wear rate. From the obtained results, it can be seen that the lapping for the coating, which was synthesised in an environment without sodium diatomite and pyrophosphate, ended already at the first stage, after which the rate of wear begins. After 2.5 km of the covered path, the rate of wear of the coating is the highest for the sample that was processed under mode No. 3. As can be seen from the conducted studies, v_f decreases by an order of magnitude after 1 km of travel, which is associated with the completion of the process of rubbing the coatings with the counterbody. The greatest rubbing is characteristic of coatings that were synthesised in an environment with diatomite. This is explained by the highest roughness of such coatings.

Table 2. *Wear rate of titanium-based coatings in different working environments*

No. composition	Electrolyte composition	The rate of wear of biocoating $v \cdot 10^{-3}$, g/m			
		L, m 500	L, m 1000	L, m 1500	L, m 2500
1	0.5 g/l KOH+0.5 g/l l.g.+0,5 g/l $Ca(OH)_2$+0.5 g/l $Na_4P_2O_7$	0.24	0.26	0.1	0.02
2	3 g/l KOH+2 g/l l.g.+3 g/l $Ca(OH)_2$	0.2	0.16	0.01	0.001
3	20 g/l KOH+20 g/l l.g.+20 g/l $Ca(OH)_2$+20 g/l $Na_4P_2O_7$+20 g/l $Na_6P_6O_{18}$+20 g/l diatomite	0.18	0.9	0.076	0.044

Source: *developed by the authors*

S. Aliasghari *et al.* (2014) investigated that the obtained coatings have a thickness of 40...50 μm, and their structure consists of titanium oxides, which are characterised by stoichiometric and non-stoichiometric composition. Rutile is the most stable form of titanium oxide. V. Korzhyk *et al.* (2024) in their work determined the dependence of the mechanical properties of the coating on the structural state of the metal base. The appearance of cracks in the coating was established and the location of the elements in the coating was explained. Research coatings synthesised on titanium alloy in various modes are presented in Figure 7.



a b c

Figure 7. *The microstructure of the coating synthesised in an alkaline electrolyte for 60 min.*

Note: *a – in an electrolyte without diatomite and with hydroxyapatite at Ia/Ic = 1.5 (x250); b – in an electrolyte without diatomite and hydroxyapatite at Ia/Ic = 1 (after etching) (x250); c – in an electrolyte with diatomite and hydroxyapatite at Ia/Ic = 1 (x200)*
Source: *developed by the authors*

It can be seen from the given photos that the border between the coating and the metal is uneven, which is explained by the mechanism of its production. Figure 7, b shows a PEO coating on a titanium alloy after etching. The obtained microstructures make it possible to determine that the obtained coating is ceramic and does not have a crystalline structure. It can be noted that the coatings synthesised in different electrolytes have different thicknesses. In Figure 7, a, metallic particles of the substrate were found in the coating. This may cause it to stick to another surface during friction. The introduction of diatomite and hydroxyapatite into the coating by adding these components to the electrolyte makes it possible to obtain a coating of greater thickness and a laminar structure (Fig. 7, c).

In practice, it is very important which implant system the doctor uses, as well as its design. Poor titanium alloy processing and inadequate design can lead to atom migration into the surrounding bone tissue, resulting in its fibrous degeneration (damage to bone tissue) (Kaur & Singh, 2019). To eliminate the possibility of aluminum and vanadium elements entering the surface, H. Fakhr Nabavi *et al.* (2017) developed a two-layer coating on a titanium alloy. The authors studied the influence of the morphology of the first layer on the morphology of the second layer. Scientists conducted research on the synthesis of titanium alloys in an alkaline electrolyte with phosphates. It was established that the application of the second layer reduces the corrosion resistance of the surface. The dependence of the porosity of the second layer on the first was revealed. Coatings obtained on titanium alloy by the PEO method are not through-through, so they exclude the possibility of the corrosive environment getting on the metal base.

Therefore, surface morphology is of great importance in the direction of biocompatibility of the surface material. For a more complete analysis of the internal construction coating, a fractographic analysis was conducted (Fig. 8). Photo shows that there are craters at the site of pore formation, which are clearly visible in Figure 8, a. Study of the fractograms, which are presented in Figure 8, b, show the presence of pores inside the coating. These pores are melted down and have different shapes and size. Also, they often merge with each other. As a result, the pores form longer shapes. In the field of implantology, the shape of such pores is acceptable.

a

b

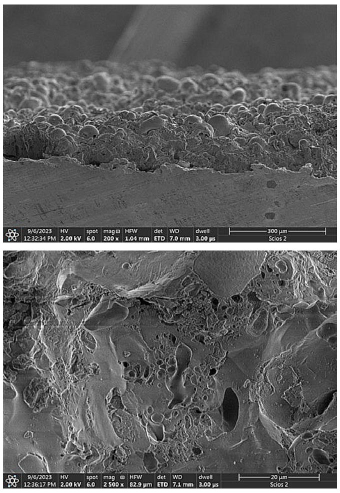


Figure 8. *Fractogram of a sample of titanium alloy with PEO coating*

Note: *a, b – coatings are synthesised in an environment with hydroxyapatite and diatomite*

Source: *developed by the authors*

The results obtained confirm the studies conducted by J. Jakubowicz (2020) indicated that surface treatment accelerates osseointegration. According to this study, it can be established that the pores inside the coating are fused and are not through. The internal chaotic porosity of the coating material leads to rapid growth of the implant with the living organism and its strong contact. Such a result of the implant introduction allows to assert the good quality of growth, when the post-operative capsule is removed. Also, this result reduces the percentage of rejection of the implant by the body.

Conclusions

In this work, it was established that the structure of the coating is characterised by a lamellar and a highly porous structure. The highly porous internal structure of the coating makes the material lighter and promotes better contact of the surface of the synthesised titanium alloy with the muscle tissue.

The study of the structural state of the coating material makes it possible to confirm the high adhesive properties of the coating with the metal. After all, the uneven border between the base and the PEO coating confirms the melting process of the metal surface in the process of plasma electrolytic oxidation. Energy parameters directly affect the value of the crater height, and therefore the surface roughness. Thus, with higher ratios of current densities, surface roughness increases. Adding diatomite particles to the electrolyte increases their roughness.

The increase in wear resistance due to the presence of a PEO coating on a titanium alloy can be explained by the presence of an unstable brittle non-stoichiometric oxide on its surface, which in the process of friction clogs surface pores, acting as a lubricant, as a result of which surface wear during reverse forward movement practically stops and increases 10 times. The coefficient of friction for coatings obtained in an electrolyte with phosphates is not much higher. Importantly, these coatings did not contain a flat area after the friction experiment. It was established that all coatings synthesised on titanium alloy increase their tribological properties.

The influence of the studied amount of hydroxyapatite and diatomite on the porosity of the PEO coating and the established ratio of Ca/P in the coating is promising. This result affects the biocompatibility of the material being synthesised. Therefore, studies of the influence of work modes on the elemental composition and porosity of PEO coatings are relevant. These studies will be investigated in subsequent works. Studying this correlation will give the opportunity to develop a synthesis regime with high biocompatibility of the coating material.

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Conflict of Interest

None.

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Стійкість до стирання біофункціональних оксидних керамічних покриттів на основі титану

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Анотація. Вимоги сучасного виробництва до деталей є надзвичайно високими. Особливу увагу приділяють частинам, що працюють у механізмах тертя. Титанові сплави характеризуються низькою зносостійкістю та здатністю прилипати до поверхонь із більшою твердістю. Тому нанесення зносостійких покриттів на поверхню титанових сплавів є актуальним завданням. Метою даної роботи було підвищення експлуатаційних властивостей титанових сплавів шляхом створення захисних покриттів на їхній поверхні. У роботі досліджувалася зносостійкість покриттів, отриманих методом плазмового електролітного оксидування (ПЕО), на титанових сплавах. Експериментальні дослідження показали, що покриття мають у своїй структурі різні оксиди (стехіометричні та нестехіометричні). Нестехіометричні оксиди характеризуються низькими фізико-механічними властивостями та накопичуються в порах після первинного зношування. У процесі зношування нестехіометричний оксид виконує функцію мастила, що позитивно впливає на їхні трибологічні властивості. Встановлено, що нанесення покриття на поверхню титанових сплавів збільшує зносостійкість у 10 разів. Межа між металевою основою та покриттям є нерівномірною, що забезпечується плавленням поверхні титанових сплавів під час ПЕО. Ця особливість демонструє високі адгезійні властивості покриттів, сформованих методом ПЕО, до титанової основи. У роботі встановлено залежність розмірів і форми пор від режимів обробки. Виявлено, що розмір пор на поверхні покриття становить 10…70 мкм, а матеріал покриття характеризується високою внутрішньою пористістю. Встановлено, що пористі покриття плавляться всередині, тому пористість є відкритою, але не наскрізною. Досліджена властивість покриттів має значення для імплантології, оскільки забезпечує з’єднання м’язової тканини з імплантом без утворення капсули

Ключові слова: фосфати; тертя; плазмове електролітне оксидування; довговічність; зношування; пористість