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RAMAZANOVA J. M., ZAMALITDINOVA M. G.

National Center of Space Exploration and Technology, Almaty, Kazakhstan, e-mail: zhanat2005@yandex.kz

PHYSICAL AND MECHANICAL PROPERTIES INVESTIGATION OF OXIDE COATINGS ON TITANIUM

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Abstract. This paper studies the impact of rapidly flowing impulse effect of electrolytic plasma oxidation on physical and mechanical specifications of oxide coatings through the surface modification of VT1-0 titanium alloy. The present mode allows obtaining dense coatings with high mechanical properties. The electrolytic plasma oxidation process implementation leads to micro arc-discharge emergency in a short period through the small duration values of 250 µs anodic impulse. The achieved oxide coatings have high wearing features. Frictional testing resulted in wearing features increase in 4-15 times comparing to the sample off coating at to15 µm oxide layer thickness. Friction coefficient curves of oxide coating samples have shown no destruction of the coating to the bottom. A run-in area is recognized on the curves; sliding surfaces adapt to each other and pass to the stable friction regime. The later leads to the friction coefficient reduce and wear intensity reduction.

Keywords: electrolytic plasma oxidation, oxide coating, frictional tests

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РАМАЗАНОВА Ж. М., ЗАМАЛИТДИНОВА М. Г.
Ғарыштық зерттеулер мен технологиялар ұлттық орталығы, Алматы, Қазақстан e-mail: zhanat2005@yandex.kz

ТИТАНДАҒЫ ТОТЫҚ ЖАБЫНДЫЛАРДЫҢ ФИЗИКА-МЕХАНИКАЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Түйіндеме. Бұл жұмыста ВТ1-0 маркалы титан қорытпасының бетін модификациялау кезіндегі алынған тотықты жабындардың физика-механикалық сипаттамаларына плазмалық электролиттік тотығу үрдісіндегі жылдам өтетін импульстік әсерлері зерттелген. Бұндай ұсынылып отырған режим, жоғары механикалық қасиеттері болатын тығыз жабындарды алуға мүмкіндік береді. Осы үрдістегі анодтың импульс ұзақтығы 250 мкс кішігірім болған кезде, аз уақытта микродоғалы разрядттар туындайды. Осылайша алынған тотықты жабындылар жоғары тозуға төзімділікпен сипатталады. Трибологиялық сынамалар бойынша қапталмаған үлгімен салыстырғанда осы жабындылардың қалыңдығы 15 мкм-ге дейін болған кезде олардың тозуға төзімділігі 4-15 есе ұлғаяды. Алынған үйкеліс коэфициент қисықтарын сараптау кезінде жабындылардың толық бұзылуы өтпейтіні көрсетілген. Осы қисықтардың көрінісі бойнша үйкелістегі беттер бір-біріне бейімделеді және тұрақты үйкеліс режиміне ауысатыны көрінеді. Яғни үйкелу коэффициентінің төмендеуіне және тозу қарқынын төмендетуге алып келеді.

Түйін сөздер: плазмалық-элеткролитикалық тотығу, тотық жабындылары, трибологиялық сынамалар

РАМАЗАНОВА Ж. М., ЗАМАЛИТДИНОВА М.Г.
Национальный центр космических исследований и технологий, Алматы, Казахстан, e-mail: zhanat2005@yandex.kz

ИССЛЕДОВАНИЕ ФИЗИКО-МЕХАНИЧЕСКИХ СВОЙСТВ ОКСИДНЫХ ПОКРЫТИЙ НА ТИТАНЕ

Резюме. В данной работе при модифицировании поверхности сплава титана ВТ1-0 изучено влияние быстротекущих импульсных воздействий плазменного электролитического оксидирования на физико-механические характеристики оксидных покрытий. Данный режим позволяет получать плотные покрытия с высокими механическими свойствами. Реализация процесса плазменного электролитического оксидирования при малых значениях длительности анодного импульса 250 мкс приводит к возникновению микродуговых разрядов в течение короткого периода времени. Полученные оксидные покрытия характеризуются высокой износостойкостью. Трибологические испытания показали увеличение износостойкости в 4-15 раз по сравнению с образцом без покрытия при толщине оксидного слоя до 15 мкм. Полученные кривые коэффициента трения для образцов с оксидным покрытием показали, что разрушения покрытия до основания не происходит. На кривых отмечается зона приработки, трущиеся поверхности приспосабливаются друг к другу и переходят в режим стабильного трения. Последнее приводит к уменьшению коэффициента трения и уменьшению интенсивности изнашивания.

Ключевые слова: плазменно-электролитическое оксидирование, оксидное покрытие, трибологические испытания

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Introduction. Aerospace and other industries are widely use titanium and its alloys owing to their advanced specific characteristics. Although these alloys use is restricted by some industries due to low hardness and wearing capacity. In this connection, a surface modification of titanium alloys is a challenging issue.

Therefore, a relatively new method of valve metals surface refinement – the method of electrolytic plasma refinement (EPR) is of interest. The EPR is peculiar to the surface micro discharge involvement into the process of surfacing, having a substantial specific effect on the surfaces under development [1-7].

However, the EPR implementation using permanent and slowly varying energy deposition is accompanied by a wide use application difficulty of this method. This is due to large energy consumption. A short-impulse mode will contribute to oxide coatings quality improvement. That is why, the development and providing researches to obtain multifunctional coatings by means of EPR in an energy saving mode is a challenging issue. Unfortunately, a great number of researches do not pay attention to the wear-resistance. As the authors consider [8-10], when rutile TiO_2 is detected in the coating composition without frictional studies, the resulting coatings are assumed wear-resistant. At the same time, the micro hardness of coatings is not always well balanced to their wear resistance.

This paper is aimed to study physical and mechanical properties of oxide coatings upon titanium obtained under rapidly flow impulse effect of electrolytic plasma oxidation.

Experimental part. An installation consisting of a two-electrode electrochemical cell and a power source was used to study the modification of the titanium surface and its alloy, (Figure 1). The cell consists of a bath tank, a stainless steel auxiliary electrode of grade and a generating electrode - a metal sample piece. The auxiliary electrode surface is 50 times exceeded the surface of the generating electrode. The source of power was "Corundum M0" impulse power source of thyristor type.

The "Corundum M0" power source of non-mass production allows producing alternating positive and negative voltage pulses of a trapezoidal shape. These shape impulses provide the most complete use of the supplied energy, and the duration of the pause between the impulses is sufficient so that a strong overheating of the near-electrode layer does not occur. The conditions under EPO are as follows: 250 ± 25 μs is the duration of the anode current impulse; 5 ± 0.5 ms is the duration of the cathode current pulse; 200 ± 25 μs is a pause between anode and cathode impulses, 50 ± 0.5 Hz is the anode and cathode impulse repetition rate, voltage is within 360–365V, current density is within 110–144 A/dm^2, process time 600 seconds for all samples.

The samples made of VT1-0 titanium alloy of rectangular shape with a 3 mm thickness and 20x40 mm and 15x40 mm size were subjected to surface modification. Pre-samples were subjected to grinding to remove the oxide film and scratches. Then the surface was cleaned from organic impurities, the samples were washed with distilled water, a C_2H_5OH solution of ethyl alcohol and distilled water again. Samples were dried at room temperature. Alkaline solutions were used as electrolytes, their compositions are provided in Table 1.

Chemical reagents of P, CP, AR were used to manufacture electrolyte solutions.

Vickersat KB 30S Pruftechnik GmbH hardness testing instrument was used to study superficial micro hardness of oxide coatings upon titanium and its alloy. Micro hardness measurement of coated samples was carried out under a 200 g load.

Figure 1 – An installation for modifying the titanium surface and its alloy

Table 1 - The electrolyte compositions

N	Electrolyte compositions	Concentration, g/l
1	Disodium phosphate dodecahydrate	40
	Pyroborate sodium decahydrate	30
	Boric acid	22
	Ammonium fluoride (NH_4F)	10
2	Trisodium phosphate dodecahydrate	70
	Aluminum oxide powder (1,1-1,5µm of dispersiveness)	20
3	Trisodium phosphate dodecahydrate	70
	Aluminum hydroxide (0,65µm of dispersiveness)	20
4	Disodium phosphate dodecahydrate	40
	Pyroborate sodium decahydrate	30
	Boric acid	22
	Ammonium fluoride (NH_4F)	10
	Aluminum oxide	20

Frictional studies of the wear resistance of oxide coatings upon titanium samples were carried out using a high-temperature TNT-S-AX0000 frictional machine. Studying the friction properties of coatings, a ball indenter with a 3 mm diameter, made of a VK alloy was used. The conditions for frictional testing are 1N load, 4 cm/s is linear speed; 25 °C is temperature, 50 % is air humidity, 3 mm of track radius, 1000 of turning numbers.

Wear resistance was evaluated by the track area, which was measured using a non-contact ZD-profilometer MICROMEASURE 3Dstation. A three-dimensional image of the surface and automatic calculation of the track area were obtained using the profilometer. Before testing the samples using profilometer, a thin aluminum layer of 50–60 nm thickness was sprayed onto the samples after testing with a frictional machine to increase the reflective properties of the surface.

A QuaNix-1500 thickness indicator with digital indexation was used to determine the thickness of the oxide coating.

Discussion of the results. The process of oxide coating by the method of electrolytic plasma oxidation is represented in several consistent stages as follows the dissociation of salts into ions; delivery of ions to the electrode surface; electrochemical reaction and accompanying micro plasma process; an oxide or ceramic coating formation; subsequent chemical reaction, removal of gaseous reaction products [11]. As a result of local high-powered effect, the layers are formed on the surfaces of products, including both matrix elements (oxidate metal) and electrolyte elements [12]. The high-quality coatings occur if the limiting stage of the process is the stage of ions delivery to the electrode surface. This is due to the fact that the rates of the electrochemical reaction and the micro plasma process are large.

Judged by the phosphate ions contribute to an increase in electrochemical polarization, since form barrier films on the metal surface of the base the electrolytes were chosen. Having borates in a solution at micro plasma discharges, solid and heat-resistant boroxide compounds are formed on the sample surface [12-15]. Fluoride compounds increase the hardness of the synthesized coating. Alumina powders were also added to the electrolyte solutions. Under the influence of the electric field intensity, the powder particles are transported to the electrode surface. The powder particles are deposited on the surface of the oxide layer or embedded in it.

Dense uniform oxide coatings are formed in all electrolyte solutions under the 250 µs anodic current duration. The 10–21 µm thickness was of the oxide coatings.

Table 2 provides the studies of the superficial micro hardness of oxide coatings obtained in various electrolyte solutions. The highest micro hardness is observed in the oxide coating obtained in №. 4 solution (Table 2).

Table 2 – Micro hardness data of oxide coatings

Measuring No.	Coatings micro hardness on VT1-0 alloy in electrolytes, HV				
	Off coating	1	2	3	4
1	200	648	629	527	1016
2	234	683	274	746	665
3	253	559	593	740	539
4	240	556	520	684	557
5	241	716	422	667	989
6	257	575	411	498	991
Average	237,5	622,8	474,8	643,7	792,8

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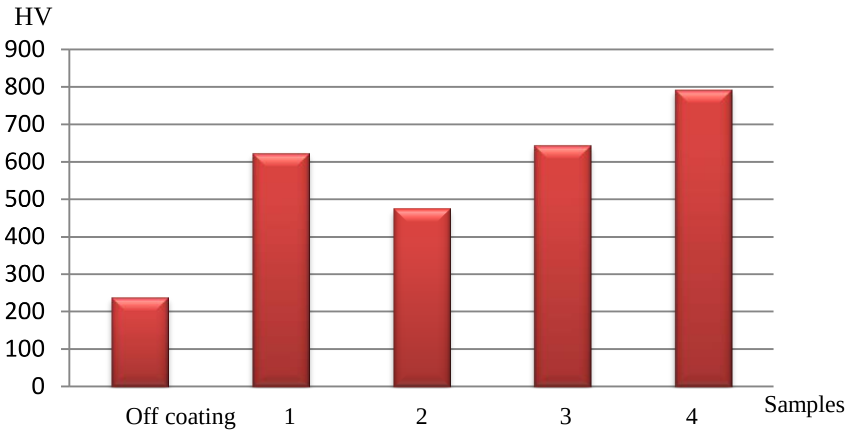

1, 2, 3, 4 - coated samples obtained in 1, 2, 3, 4 electrolytes (Table 1)

Figure 2 – Micro hardness of oxide coatings on VT1-0 alloy data compared to the sample off coating

The micro hardness increases 3.3 times in comparison with the off coat sample. The coating process in an electrolyte containing powders has a different mechanism than a conventional electrolyte. Due to the presence of suspended particles in the solution, the process of shaping is intensified. As the electrophoresis effect occurs, solid alumina particles can be incorporated into the coating composition. Thereby produces a composite coating consisting of titanium oxide and alumina. Solid particles of aluminum oxide embedded in the coating increase the micro hardness of the resulting composite layer. An increase in the surface micro hardness by two or more times is observed on coated samples formed in 1, 2, 3 solutions (Figure 2).

Titanium samples without an oxide coating and with an oxide coating obtained in the EPO impulsed mode in various electrolytes were also subjected to wear resistance tests.

Frictional studies provided three-dimensional images of the surfaces of samples with a track, curves of friction coefficient changes, and areas of wear tracks data were obtained. Off coated and coated samples were subjected to testing.

Three-dimensional images of sample surfaces with a track for VT1-0 alloy off coated and coated, obtained in №. 2, 4 electrolyte solutions (Table 1), as well as the corresponding curves of friction coefficients are provided in Figure 3.

By the three-dimensional images of the surfaces of samples with a track, the width and depth of the sample track off coating significantly exceeds the width and depth of samples tracks with coating is observed. Following this, the wear resistance of samples with an oxide coating is higher than that of the original sample. Coating resistance to abrasion can be estimated by the curves of the coefficient of friction. The curves of friction coefficients of the coating have the tribosystem run-in areas, where the friction surfaces adapt to each other. In addition, the stages of stable friction conditions are visible and just about constant and relatively low wear rate. There is no abrupt change in the friction coefficient characteristic of the coating destruction. Oxide coatings are not destroyed and do not wear out to the bottom. These tracks areas data are provided in Table 3.

Comparative data regarding coatings wear resistance represented by a diagram in Figure 4.

As the Figure 4 provides, a high wear resistance as compared with the uncoated sample characterizes the oxide coatings obtained on the VT1-0 alloy. The wear resistance of the oxide coating obtained in No. 2 electrolyte (Table 1) is increased by 15 times compared with the initial material. This coating is made in an electrolyte containing an alkali metal phosphate with the addition of alumina powder. Aluminum oxide embedded in the coating has an effect on the wear resistance of the coating. The wear resistance for an oxide coating obtained in No.4 electrolyte (Table 1) is increased 12 times with respect to the original off coated sample. During frictional tests, the process of pair of article / counter body run-in occurs, the friction surfaces adapt to each other. The frictional system becomes steady state. The wear resistance is also influenced by the initial surface roughness and micro hardness.

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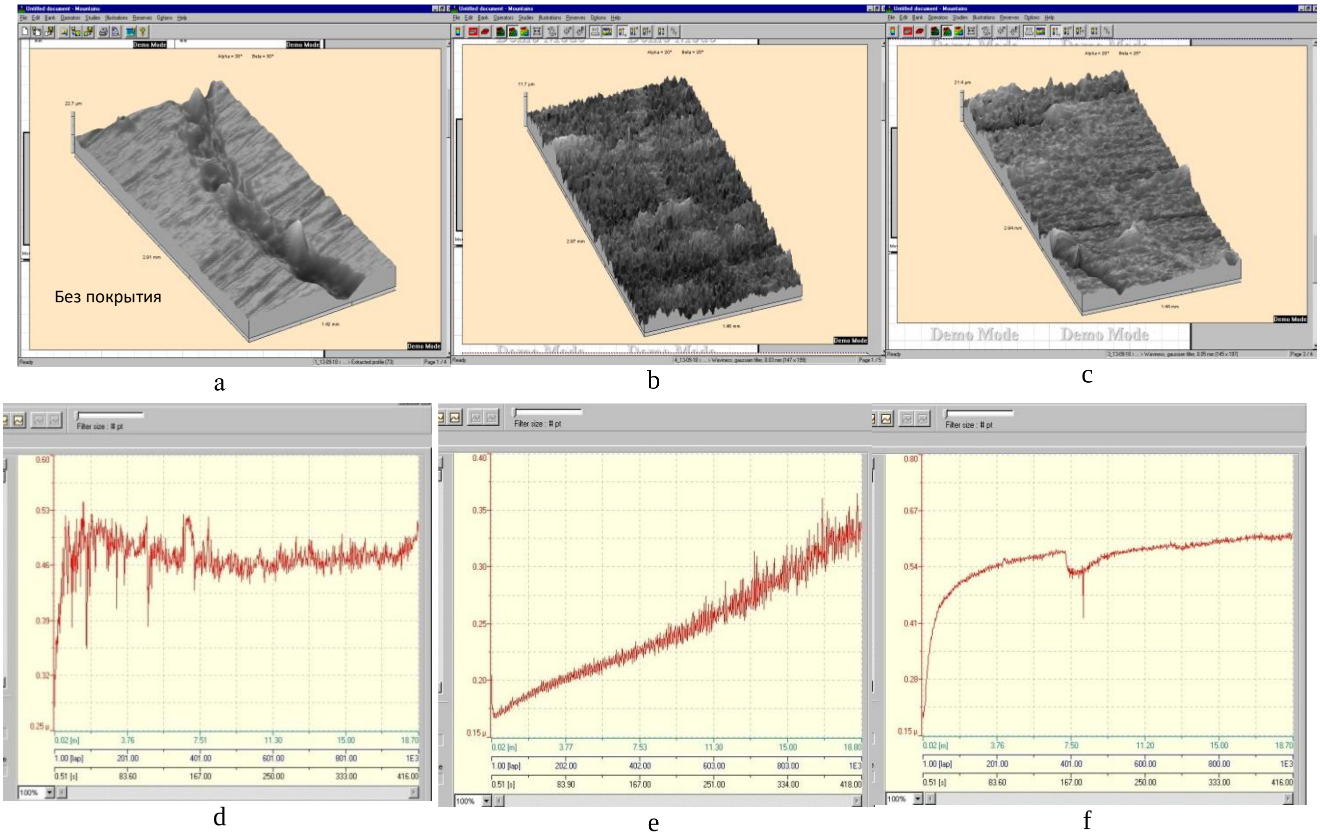
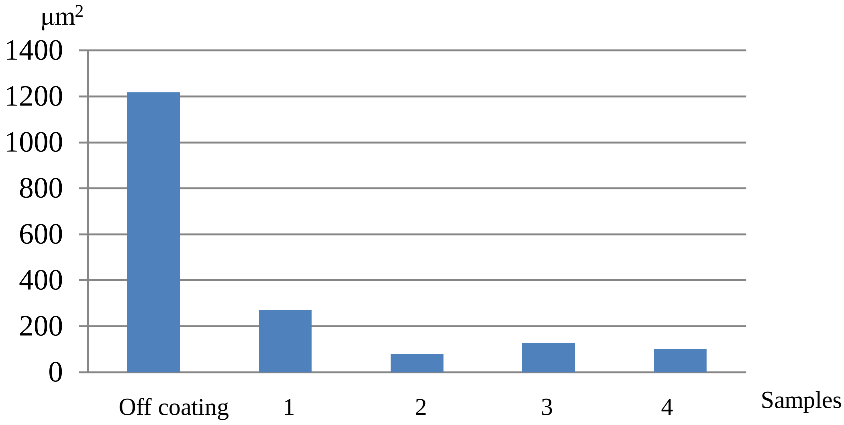


a - VT1-0 alloy off coating; b - coating obtained in №.2 electrolyte; c - the coating obtained in the №.4 electrolyte;
d - curve of friction coefficient on VT1-0 alloy off coating; e - coating curve friction coefficient obtained in the №.2 electrolyte;
f - coating curve friction coefficient obtained the №.4 electrolyte.

Figure 3 - Three-dimensional image of the surfaces of samples with a track and the corresponding curves of friction coefficients for VT1-0 alloy off coated and coated

Table 3 - Track Area Data

n	Track area, μm^2				
	Off coating	Electrolyte 1	Electrolyte 2	Electrolyte 3	Electrolyte 4
1	1761	240	48,9	208	185
2	978	286	25,7	102	96,8
3	748	569	149	77,4	198
4	479	358	28,5	172	77,8
5	1059	76,3	124	135	58,4
6	1228	347	83,2	128	49,4
7	1761	177	160	57,8	71,9
8	978	225	65,6	94,9	131
9	748	157	35,0	166	47,3
10	1178	-	-	-	-
11	2192	-	-	-	-
12	1500	-	-	-	-
Σ/n	1217,5	270,6	79,9	126,8	101,7



1, 2, 3, 4 - VT1-0 samples with a coating obtained in 1, 2, 3, 4 electrolytes (Table 1)

Figure 4 – Wear resistance comparative data

Conclusions. The process of plasma electrolytic oxidation of the surface of a VT1-0 titanium alloy in an impulse mode was studied. Impulse mode allows obtaining dense, uniform oxide coatings that do not require additional grinding of the surface. Such coatings thickness is 10-21 µm.

The layer oxidation leads to an increase in the micro hardness of the surface of the VT1-0 titanium alloy to 792.8 HV with a 21 µm coating thickness, which is up to 3.3 times higher than the initial value.

The wear resistance of oxide coatings studies has shown a significant increase compared with the off coated sample. The oxide coating obtained in the electrolyte containing sodium phosphate and aluminum oxide increases wear resistance by 15 times compared with the sample off coating.

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Information about authors:
Ramazanova Zhanat - PhD in Chemistry, Joint-Stock Company "National Center of Space Research and Technology", Almaty, Kazakhstan e-mail: zhanat2005@yandex.kz
Zamalitdinova Marina - Magister of Information Systems, Joint-Stock Company "National Center of Space Research and Technology", Almaty, Kazakhstan, https://orcid.org/0000-0002-8746-1664

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