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## Investigation of the properties of oxide coatings on titanium alloys obtained by plasma electrolytic oxidation

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### ABSTRACT

The use of structures made of titanium and its alloys, which have improved corrosion, physical and mechanical properties, are in demand in many industries. In this regard, the processes of modifying the surface of metals are of interest. One of the modern and promising methods of metal surface treatment is plasma electrolytic oxidation. Currently, there is a problem of widespread use of this process when DC modes are used in the implementation of the process. This is due to the large consumption of electricity. The purpose of this work is to study the morphological and corrosion properties of oxide coatings obtained in the pulsed anode-cathode mode on titanium alloys VT1-0 and VT5 in various electrolyte solutions. Modification of the surface of titanium alloys was carried out at the duration of the anode current pulse of  $250 \pm 25$  microseconds, the duration of the cathode current pulse of  $5 \pm 0.5$  ms, the repetition frequency of the anode and cathode pulses of  $50 \pm 0.5$  Hz. Alkaline solutions served as electrolytes. Oxide coatings obtained in various electrolytes are characterized by different porosity and coating thickness. Coatings with a finely porous structure, with an average pore diameter from 0.09 microns to 0.4 microns, and larger pore sizes of 0.6 – 0.7 microns were obtained. The porosity of the coatings ranges from 6.12% to 12.2%. According to the data of energy dispersion analysis, it was found that the structure of oxide coatings includes both components of the processed metal and components of the electrolyte solution. The main components, in this case, are oxygen and processed metal, as well as other elements such as boron, phosphorus, aluminium, fluorine, sodium, silicon and others. Corrosion tests according to GOST 9.308-85 under the influence of neutral salt mist at a temperature of  $(35 \pm 2)$  °C with 1500 hours in the Ascott CC 450 chamber, it was shown that there was no corrosion damage to the coatings.

**Keywords:** oxide coating, electrolyte, plasma-electrolytic oxidation, corrosion tests, coating morphology, porosity.

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## Introduction

Titanium and its alloys are widely used in many sectors of the manufacturing economy. However, to create promising and modernize existing products, it is necessary to give them a set of properties, such as a combination of corrosion resistance and good physical and mechanical properties. By introducing alloying additives, the properties of titanium alloys are improved [[1], [2]]. Aluminum, vanadium,

molybdenum, silicon, chromium, zirconium and others can be used as alloying additives. The introduction of alloying additives during titanium smelting positively affects its corrosion resistance and other properties [2]. However, this leads to an increase in the cost of the product and the weight of the structure.

Currently, classical materials science is giving way to methods of modifying the properties of materials, such as surface treatment, since the

surface characteristics determine the level of product properties [[3], [4]]. In addition, surface modification is more attractive compared to other methods of changing the characteristics of materials and products.

Currently, plasma electrolytic oxidation (PEO) is one of the promising methods for modifying the surface of titanium [[5], [6], [7]]. This method allows you to change the surface condition of valve metals significantly, which makes it possible to replace traditional materials with inexpensive and non-scarce materials. At the same time, there is a significant improvement in the operational properties of products [[8], [9], [10]].

A distinctive feature of the PEO method is the participation in the modification process of surface micro-discharges, which have a significant and specific effect on the phase and structural properties of the coating [11]. The obtained oxide coatings by the PEO method in electrolyte solutions have high adhesion to the substrate, wear resistance, and heat resistance [[12], [13]].

The implementation of the PEO process can be carried out in various modes using stationary and slowly changing energy influences. In these modes, the application of the method is not widely used due to the large consumption of electricity. Recently, PEO modes based on fast-flowing (pulsed) energy effects have been used [14]. With a small value of the duration of the anode current pulse of the order of 250 microseconds, coatings characterized by low roughness are formed [15]. The use of these modes does not require a large consumption of electricity, and can contribute to a wider application of the PEO method in production.

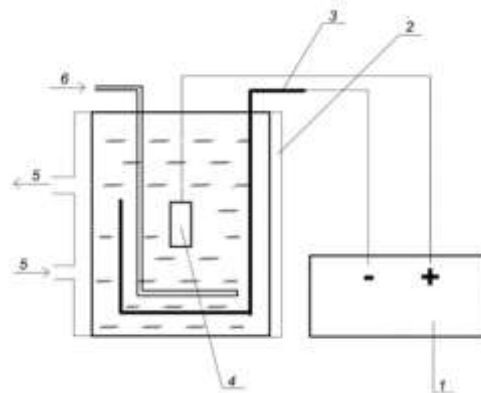
It is known that the properties of oxide coatings are influenced by the composition of electrolyte solutions [16]. In view of this, it is relevant to study the corrosion resistance and morphology of coatings obtained in various electrolytes. This makes it possible to purposefully form functional coatings with certain properties.

This work aims to study the properties of complex oxide coatings, consisting of two or more elements obtained in the pulsed anode-cathode mode of PEO in various electrolyte solutions.

### Experimental part

Samples from titanium alloys VT1-0 and VT5 were subjected to surface treatment by plasma electrolytic oxidation. The samples had a rectangular shape with a thickness of 3 mm and a size of 20 x 40 mm and 15 x 40 mm. The samples were ground

before the PEO process to remove irregularities and scratches. Further, the surfaces of the samples were degreased using ethyl alcohol. Drying was carried out at room temperature. The scheme of the installation for obtaining oxide coatings by the PEO method is shown in Figure 1.



**Figure 1** - Installation diagram: 1 - power supply, 2 - electrochemical bath with water cooling jacket, 3 - auxiliary electrode (cathode), 4 - coated sample (anode), 5 - water, 6 - air

The research facility consisted of a bath with a cooling system. Stainless steel was used as the cathode. The processed samples served as the anode. A switching power supply "Corundum M0", not mass-produced, was used to obtain oxide coatings. The power supply generates positive and negative voltage pulses of trapezoidal shape. The PEO processing of titanium samples took place under the following conditions: the duration of the anode current pulse is  $250 \pm 25$  microseconds; the duration of the cathode current pulse is  $5 \pm 0.5$  ms; the interval between the anode and cathode current pulses is  $250 \pm 25$  microseconds; the frequency of the anode and cathode pulses is  $50 \pm 0.5$  Hz, the voltage is within 360-365 V. The current density was in the range of 109 - 115 A/dm<sup>2</sup>, and this parameter depends on the composition of the electrolyte and the metal alloy. Processing time 600 sec. The electrolyte temperature was maintained within 20 °C – 25 °C. To obtain coatings, electrolytes were used, the compositions of which are presented in Table 1.

Electrolyte solutions were prepared from chemical substances of the chemically pure grade, analytical grade. Electrolytes were prepared in distilled water. The thickness of the oxide coatings was measured using a NOVOTEST TP-1 thickness gauge with an NF-2 sensor with a digital indicator indication. When measuring the thickness of oxide coatings, 7 measurements were carried out on both sides of the sample. Next, the arithmetic mean of the coating thickness was calculated.

**Table 1** - Compositions of electrolytes

No. electrolyte	Name of the component	Concentration, g/l
1	Sodium silicate (metasilicate) 9 aqueous	100
	NaOH	8
	Aluminum oxide (1.1-1.5 microns) powder	20
2	Sodium phosphoric acid 3- substituted, 12 aqueous	70
	Aluminium hydroxide (0.6 μm)	20
3	Sodium phosphoric acid, two-substituted, 12 aqueous	40
	Sodium tetraborate 10 aqueous	30
	Boric acid	22
	Ammonium fluoride (NH <sub>4</sub> F)	10
	Aluminium oxide	20

Micrographs and energy dispersion analysis of oxide coatings on titanium alloys were obtained using a Hitachi TM3030 scanning electron microscope in SEI mode at an accelerating voltage of 15 kV with an Oxford Instruments energy dispersion analysis prefix.

When calculating porosity, the number of pores per surface unit, and evaluating their shapes, the planimetry method was used by processing micrographs of the surface of oxide coatings [17]. In this case, porosity was determined as the ratio of the sum of the pore areas  $F_p$  to the total area of the

observation site  $F_{total}$ : 
$$\Pi = \frac{\sum F_p}{F_{total}} 100\% .$$

The corrosion resistance of samples with oxide coatings was carried out under the influence of neutral salt mist at a temperature of  $(35 \pm 2)^\circ\text{C}$  for 1000 hours in the Ascott CC 450 chamber according to GOST 9.308-85. In the Ascott CC 450 chamber, the distance between the test samples was 20 mm; the distance from the chamber walls was at least 100 mm, and from the day of the chamber – at least 200 mm. The tests were carried out at a  $(35 \pm 2)^\circ\text{C}$ . According to GOST 9.308-85, a sodium chloride solution was continuously sprayed in the chamber (concentration  $50 \pm 5 \text{ g/dm}^3$ , pH of the solution 6.5 – 7.2). Spraying of salt mist was carried out so that the condensate volume during the chamber's operation for 24 hours was maintained within 1.2 - 1.5 cm<sup>3</sup>/hour. The state of the oxide coatings was monitored visually through 2, 24, 96, 294, 460, 720, 1000 and 1500 hours of testing. During the control of coatings, samples were removed from the chamber, washed with tap water, dried.

The appearance was assessed for the presence of corrosion damage by the following signs: discolouration, pitting corrosion, peeling of the coating, etc.

## Results and discussion

As a result of the PEO process, dense oxide layers are formed on the surface of titanium alloys VT1-0 and VT5, the thickness data given in Table 2. The thickness of the oxide coatings is uniform over the entire surface of the samples.

**Table 2** - Data on the thickness of oxide coatings on titanium alloys

No. electrolyte	Coating thickness on VT1-0 alloy, microns	Coating thickness on VT5 alloy, microns
1	5.0	7.5
2	15.0	9.5
3	21.0	19.5

Energy dispersion analysis of the coatings obtained on titanium alloys showed that the main elements included in their composition are both components of the processed material (base) and the electrolyte solution. The composition of the coating mainly consists of oxygen and processed metal. Other elements are boron, phosphorus, aluminium, fluorine, sodium, silicon, etc. (Tables 3, 4).

In the work of researchers [18], the production of complex PEO coatings is confirmed. The authors have shown that the composition of the oxide layers formed on titanium from the calcium phosphate electrolyte system contains the elements Ti, P, Ca and O [18].

It is known that the pores in the PEO are formed at the place of micro-arc combustion [19]. During the initial period of the plasma electrolytic oxidation process, when a positive voltage is applied to the sample, a porous, thin barrier layer forms on the surface of the processed material according to the mechanism of electrochemical oxidation. Micro-

**Table 3** - Composition of coatings on VT1-0 alloys

Electrolytes	Content of elements, at. %							
	O	Ti	B	P	F	Na	Al	Si
1	65.42	15.7	-	-	-	1.62	0.53	13.2
2	64.74	31.48	-	2.66	-	0.95	0.17	-
3	63.84	27.47	4.75	2.42	0.5	0.42	0.6	-

**Table 4** - Composition of coatings on VT5 alloys

Electrolytes	Content of elements, at. %							
	O	Ti	B	P	F	Na	Al	Si
1	60.45	20.26	-	-	-	1.55	1.02	16.76
2	64.39	31.42	-	2.21	-	0.76	1.22	-
3	65.84	20.79	5.61	3.22	1.21	1.63	1.70	-

discharges do not occur when a barrier layer is formed (Figure 2a). Provided that the electrical strength of the barrier layer is less than the voltage at the electrode-solution interface, a breakdown occurs, a spark discharge occurs (Figure 2b), which, as the coating thickness increases, turns into a micro-arc (Figure 2c). High temperatures develop on the arc axis [[20], [21]].

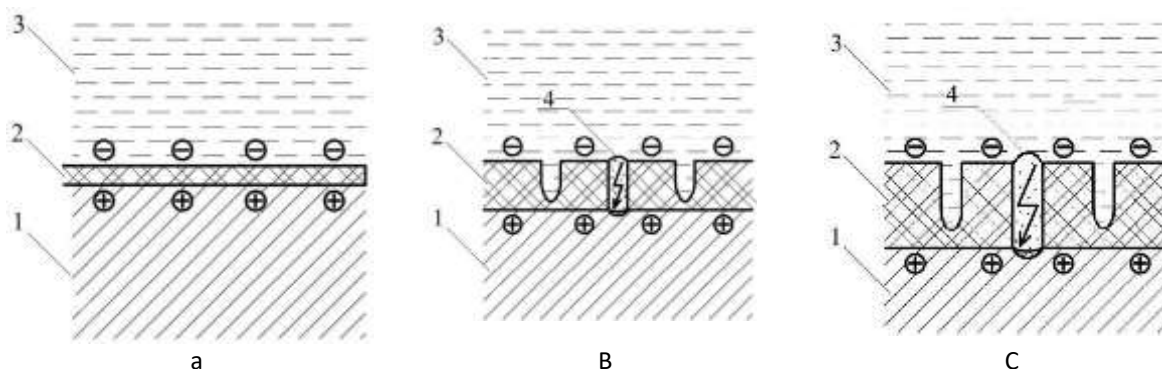
When using the pulse mode, the burning time of the micro-arc depends on the duration of the anode pulse of the anode current pulse. As the coating thickness increases, the size of the micro-discharges may increase, and their number may decrease. This leads to the formation of pores of large diameters.

Morphological studies of the surface of the oxide coating have shown that as a result of PEO under pulsed anode-cathode mode in electrolyte No.1, both on the VT1-0 alloy and the VT5 alloy, many small pores with an average diameter of 0.09 microns on the VT1-0 alloy, and 0.2 microns on the VT5 alloy are formed (Figure 3 a- b; Table 5). The porosity of the coatings is 6.12% and 6.25%. The pores mostly have a rounded shape. Almost under the same conditions of the PEO process for all

electrolyte solutions, a thinner coating is formed in this electrolyte. This is due to the formation of a finely porous coating structure. To obtain coatings with a more significant value of its thickness, it requires a further increase in voltage from an external power source.

A finely porous structure also characterizes the oxide coatings obtained in electrolyte No.2. The average pore diameter for the VT1-0 alloy is 0.4 microns, and the porosity is 7.7% (Figure 3c; Table 6). On the VT5 alloy, the average pore diameter is 0.2 microns, and the porosity is 7.7%. (Figure 3d; Table 6). The pores are evenly distributed over the entire surface of the sample. It should be noted that the pore sizes in this electrolyte are larger than on the coating obtained in the first electrolyte, and, accordingly, the coating thickness is more significant. Small pores contribute to the reduction of mechanical stresses in the coating [22].

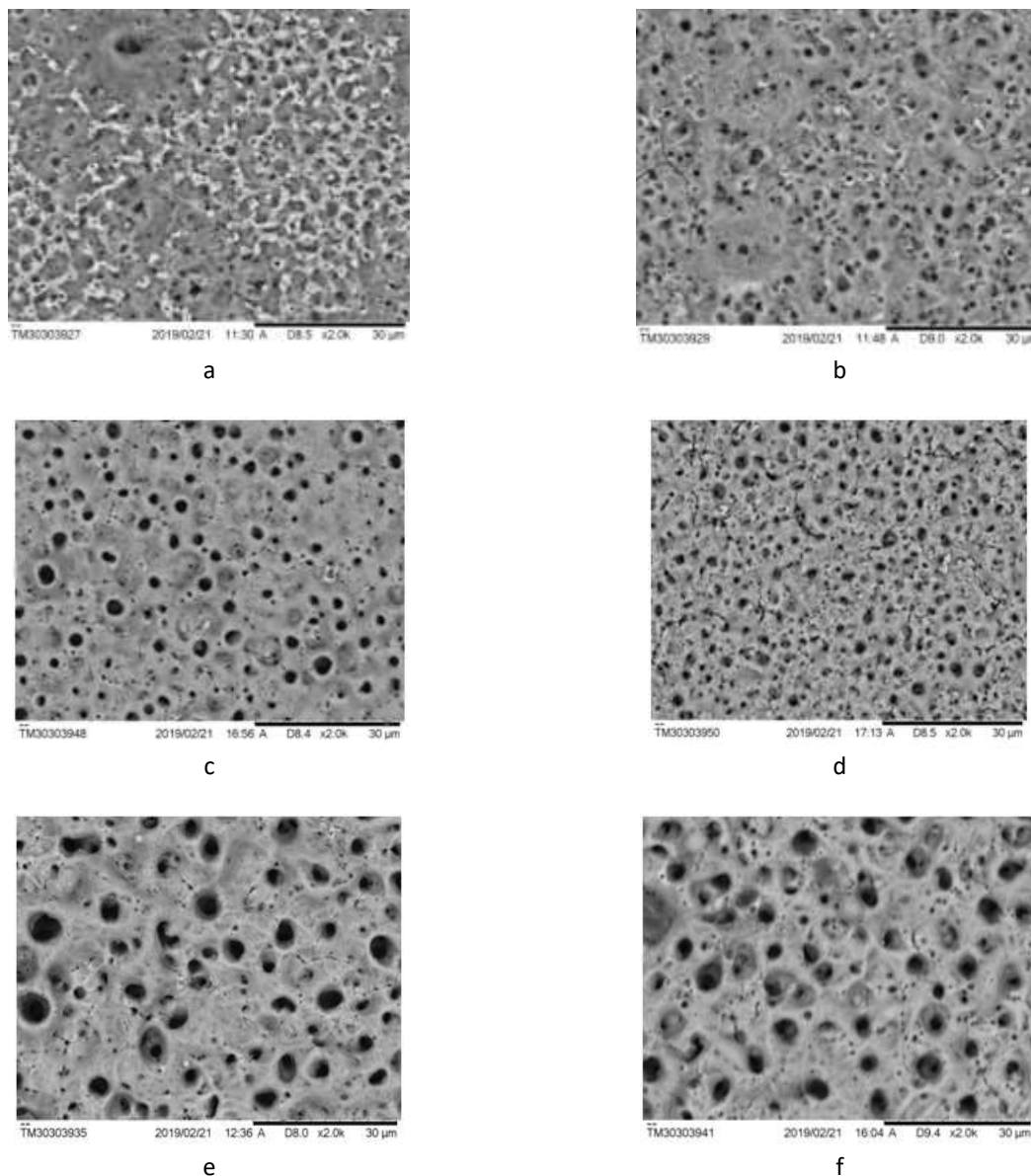
Larger pores are formed in electrolyte No.3 (Figure 3 e - f). The average pore diameter is 0.7 microns on the VT1-0 alloy, 0.6 microns on the VT5 alloy. The porosity of the coatings is 11.3% on the VT1-0 alloy, and 12.2% on the VT5 alloy (Table 7).



a – barrier layer formation; b - spark discharge stage; c - micro-arc discharge stage; 1 - metal, 2 – an oxide layer, 3 - electrolyte solution, 4 - plasma formation



**Figure 2** - Scheme of the development of plasma formations



**Figure 3** - Micrographs of oxide coatings obtained: a – in electrolyte 1 on VT1-0 alloy; b - in electrolyte 1 on VT5 alloy; c - in electrolyte 2 on VT1-0 alloy; d - in electrolyte 2 on VT5 alloy; e - in electrolyte 3 on VT1-0 alloy; f - in electrolyte 3 on VT5 alloy

Calculated data on the porosity of oxide coatings obtained in various electrolytes on titanium alloys VT1-0 and VT5 are given in Tables 5-7.

During corrosion tests, the change in the condition of the coatings was determined by visual inspection through 2, 24, 96, 294, 460, 720, 1000 and 1500 hours (Table 8).

Thus, at all stages of the test under the influence of neutral salt mist at a temperature of 35°C for 1500 hours at a solution concentration of 50 g/l of sodium chloride, the pH of the solution is 6.5 – 7.2, there is no corrosion damage on samples with oxide coatings, no traces of corrosion damage are observed. Oxide coatings have proven to be corrosion resistant.

**Table 5** - Data on porosity of oxide coatings obtained in electrolyte 1

Alloy	Porosity $\Delta S$ , %	Number of pores per 1 cm <sup>2</sup> of coating	Average pore diameter, microns
VT1-0	6.12	$2.2 \cdot 10^7$	0.09
VT5	6.25	$1.3 \cdot 10^7$	0.2




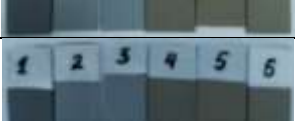
**Table 6** - Data on porosity of oxide coatings obtained in electrolyte 2

Alloy	Porosity $\Delta S$ , %	Number of pores per 1 cm <sup>2</sup> of coating	Average pore diameter, microns
VT1-0	7.7	$1.3 \cdot 10^7$	0.4
VT5	7.7	$1.6 \cdot 10^7$	0.2

**Table 7** - Data on porosity of oxide coatings obtained in electrolyte 3

Alloy	Porosity $\Delta S$ , %	Number of pores per 1 cm <sup>2</sup> of coating	Average pore diameter, microns
VT1-0	11.3	$7.0 \cdot 10^6$	0.7
VT5	12.2	$7.8 \cdot 10^6$	0.6

**Table 8** - Results of corrosion tests

Test stage, hours	The appearance of the samples treated with PEO	Appearance of coatings
0		Initial samples
2		Without changes
24		Without changes
96		Without changes
294		Without changes
460		Without changes
720		Without changes
1000		Without changes
1500		Without changes

## Conclusions

The process of obtaining oxide coatings on titanium alloys VT1-0 and VT5 by plasma electrolytic oxidation has been studied. Uniform porous oxide coatings were obtained in the pulsed mode of the PEO process.

It is shown that the porosity of the coatings depends on the composition and nature of the electrolyte. In electrolytes containing sodium silicic acid, sodium hydroxide, aluminium oxide (electrolyte No.1), as well as sodium phosphoric acid and aluminium hydroxide (electrolyte No.2), the coatings are finely porous. The average pore

diameter of oxide coatings on titanium alloys in electrolyte No.1 is 0.09 microns and 0.2 microns, respectively, at VT1-0 and VT5. In electrolyte No.2, the average pore diameter of oxide coatings is 0.4 microns (alloy VT1-0) and 0.2 microns (alloy VT5). In electrolyte No.3, pores of a larger size of 0.6 – 0.7 microns are formed.

The coating composition includes both components of the processed material and

components of the electrolyte solution. Oxide coatings are corrosion resistant when tested in accordance with GOST 9.308-85 under the influence of neutral salt fog at a temperature of  $(35 \pm 2)^\circ\text{C}$  for 1500 hours in an Ascott CC 450 chamber.

**Conflict of interests.** On behalf of all authors, the correspondent author declares that there is no conflict of interests.

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## Плазмалық электролиттік оксидтеу арқылы алынған титан қорытпаларындағы оксидті жабындардың қасиеттерін зерттеу

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### ТҮЙІНДЕМЕ

Жақсартылған коррозиялық, физика-механикалық қасиеттері бар титан мен оның қорытпаларынан жасалған құрылмаларды пайдалану көптеген салаларда сұранысқа ие. Осыған байланысты металдардың бетін түрлендіру процестері қызығушылық тудырады. Металл бетін өңдеудің заманауи және перспективті әдістерінің бірі-плазмалық электролиттік тотықтыру. Қазіргі уақытта процесті жүзеге асырғанда тұрақты ток режимдері қолданылады. Бұдан процесті кеңінен қолдану проблемасы туындайды, себебі электр энергиясы көп тұтынылады. Бұл жұмыстың мақсаты-электролиттердің әртүрлі ерітінділеріндегі VT1-0 және VT5 титан қорытпаларында импульсті анод-катод режимінде алынған оксид жабындарының морфологиялық және коррозиялық қасиеттерін зерттеу. Титан қорытпаларының бетін модификациялау  $250 \pm 25$  мкс токтың анодты импульсінің ұзақтығы,  $5 \pm 0.5$  мс токтың катодты импульсінің ұзақтығы,  $50 \pm 0,5$  Гц анодты және катодты импульстардың өту жиілігі кезінде жүзеге асырылды. Сілтілік ерітінділер электролит қызметін атқарды. Әртүрлі электролиттерде алынған оксидті жабындар әртүрлі кеуектілікпен және жабын қалыңдығымен сипатталады. Қаптамалар орташа кеуек диаметрі 0,09 мкм-ден 0,4 мкм-ге дейінгі жіңішке кеуекті құрылымымен де, 0,6 – 0,7 мкм үлкен кеуек өлшемдерімен де алынған. Қаптамалардың кеуектілігі 6,12%-дан 12,2%-ға дейін. Энергетикалық дисперсиялық талдау деректері бойынша оксидті жабындардың құрамына өңделетін металдың құрамдас бөліктері де, электролит ерітіндісінің компоненттері де кіретіні анықталды. Негізгі құрамдастарға оттегі және өңделетін металл, сонымен қатар бор, фосфор, алюминий, фтор, натрий, кремний және т.б. жатады. Ascott CC 450 камерасында 1500 сағат бойы  $(35 \pm 2)^\circ\text{C}$  температурада бейтарап тұзды тұманның әсерінен ГОСТ 9,308-85 сәйкес жүргізілген коррозиялық сынаулар жабындарда коррозиялық зақымданулардың жоқтығын көрсетті.

**Түйін сөздер:** оксидті жабын, электролит, плазмалық-электролиттік оксидтеу, коррозиялық сынау, жабындардың морфологиясы, кеуектілік.

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## Исследование свойств оксидных покрытий на сплавах титана, полученных плазменным электролитическим оксидированием

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### АННОТАЦИЯ

Использование конструкций из титана и его сплавов, которые имеют улучшенные коррозионные, физико-механические свойства, востребованы во многих отраслях производства. В связи с этим представляет интерес процессы модифицирования поверхности металлов. Одним из современных и перспективных методов обработки поверхности металлов является плазменное электролитическое оксидирование. В настоящее время существует проблема широкого применения данного процесса, когда при реализации процесса используются режимы на постоянном токе. Это связано с большим потреблением электроэнергии. Целью данной работы является исследование морфологических и коррозионных свойств оксидных покрытий, полученных в импульсном анодно-катодном режиме на сплавах титана BT1-0 и BT5 в различных растворах электролитов. Модифицирование поверхности сплавов титана осуществлялось при продолжительности анодного импульса тока  $250 \pm 25$  мкс, продолжительности катодного импульса тока  $5 \pm 0.5$  мс, частоте следования анодных и катодных импульсов  $50 \pm 0.5$  Гц. Электролитами служили щелочные растворы. Оксидные покрытия, полученные в различных электролитах, характеризуются различной пористостью и толщиной покрытия. Покрытия характеризуются мелкопористой структурой, со средним диаметром пор от 0,09 мкм до 0,4 мкм, так и с более крупными размерами пор 0,6 – 0,7 мкм. Пористость покрытий составляет от 6,12% до 12,2%. По данным энергодисперсионного анализа было установлено, что в состав оксидных покрытий включаются как компоненты обрабатываемого металла, так и компоненты раствора электролита. Основными компонентами при этом являются кислород и обрабатываемый металл, а также другие элементы как бор, фосфор, алюминий, фтор, натрий, кремний и другие. Коррозионные испытания согласно ГОСТ 9,308-85 под воздействием нейтрального соляного тумана при температуре  $(35 \pm 2)$  °С в течение 1500 часов в камере Ascott CC 450 показали, что коррозионных разрушений покрытий нет.

**Ключевые слова:** оксидное покрытие, электролит, плазменно-электролитическое оксидирование, коррозионные испытания, морфология покрытий, пористость.

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