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Methods and instruments for measuring surface morphology and mechanical parameters of oxide coatings

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Abstract. Morphological studies of coatings were carried out on aluminum samples obtained by microarc oxidation. The sinusoidal current density in the anodic and anode-cathode modes was 15 A/dm^2, and the processing time varied from 120 s to 960 s. The formation of oxide coatings occurred in a silicate-alkaline electrolyte. Studies of the surface topology and mechanical parameters of oxide coatings were carried out using an SEM, a laser profilometer and a universal electrical strength meter, which, in turn, made it possible to establish a relationship between the properties of the coatings and the sample processing time. Thus, an increase in the processing time during the micro-arc oxidation of products made of valve group alloys leads to a complication of the surface morphology, as well as an increase in the size and number of pores. In addition, the coating roughness increases in the anode and anode-cathode modes. Electrical strength tests showed that all samples with the resulting coatings withstood a voltage of 600 V. Multifunctional coatings obtained using the developed technological modes are multilayer structures. They consist of a base layer with excellent adhesion, an intermediate layer with porous structure and a top layer with high porosity and actively functioning surface. The changes revealed during morphological studies are characteristic of the plasma growth model of coatings. The results of the conducted morphological studies of coatings can be implemented in the development of a digital twin of the microarc oxidation process.

Keywords: micro-arc oxidation, digital twin, surface morphology, roughness, electrical strength, porosity

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Методы и средства измерений морфологии поверхности и механических параметров оксидных покрытий

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Аннотация. Проведены морфологические исследования покрытий на образцах алюминия, полученных микродуговым оксидированием. Плотность синусоидального тока в анодном и анодно-катодном режимах составляла 15 А/дм^2, а время обработки варьировалось от 120 с до 960 с. Формирование оксидных покрытий происходило в силикатно-щелочном электролите. Исследования топологии поверхности и механических параметров оксидных покрытий проводились посредством применения СЭМ, лазерного профилометра и универсального измерителя электрической прочности, что, в свою очередь, позволило установить взаимосвязь между свойствами покрытий и временем обработки образца. Так, увеличение длительности обработки в процессе микродугового оксидирования изделий из сплавов вентильной группы приводит к усложнению морфологии поверхности, а также увеличению размера и количества пор. Кроме того, в анодном и анодно-катодном режимах увеличивается шероховатость покрытия. Испытания на электрическую прочность показали, что все образцы с полученными покрытиями выдержали напряжение 600 В. Многофункциональные покрытия, полученные с использованием разработанных технологических режимов, представляют собой многослойные структуры. Они состоят из базового слоя с отличной адгезией, промежуточного слоя с пористой структурой и верхнего слоя с высокой пористостью и активно функционирующей поверхностью. Изменения, выявленные в ходе морфологических исследований, характерны для модели плазменного роста покрытий. Результаты проведенных морфологических исследований покрытий могут быть внедрены при разработке цифрового двойника процесса микродугового оксидирования.

Ключевые слова: микродуговое оксидирование, цифровой двойник, морфология поверхности, шероховатость, электрическая прочность, пористость, плазма

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Introduction

Nowadays, most studies are aimed at studying the influence of heterogeneous factors of the microarc oxidation (MAO) process on the properties of the formed coatings, including mechanical ones. For example, the study in [1] demonstrates that increasing the number of plasma discharges at low current densities increases the porosity and roughness of the coating. According to the data from [2], electron microscopy revealed that the porosity of coatings decreases during time treatment, but the roughness increases. Adding TiO_2 nanoparticles to the electrolyte is also achieved by the increase porosity, thickness and surface roughness coating [3]. In [4] it is shown that that adding glycerol to the electrolyte leads to an increase in the number of pores and reducing their size, which reduces surface roughness. These studies are not unified, since they were carried out on different technological equipment under different processing modes and do not allow compare the results of experiments with each other, which limits their use as training data for the digital twin of the micro-arc process oxidation [5].

In addition, there is a need for a deeper study and understanding of the processes and phenomena occurring during micro-arc oxidation. In turn, it will allow us to investigate the modification of the coatings structure during their formation. In accordance with the studies of coatings obtained by the MAO method and described in [6], a plasma growth model was created for a specific coating, the structure of which includes a barrier layer, an inner layer with numerous closed pores and an outer layer with a rough surface.

Materials and methods

To study surface morphology and mechanical parameters of MAO coatings, 8 aluminum samples, presented in the form plates measuring 20×15×2 mm (surface area 0.05971 dm^2) and whose surface was previously brought to a roughness R_a in the range from 0.1 to 0.2 μm were selected. At the first stage, the samples were milled on a universal machine; at the second stage were polished on a manual grinding – polishing machine MP-100S MTDI (Korea) by sequentially sorting sandpapers with grades from P240 to P2000 (ISO6344 marking) and final polishing with a felt cloth using paste GOI. The treated samples were placed in a galvanic bath with silivate-alkaline electrolyte. Coatings were obtained using a MAO installation [7, 8] at a sinusoidal current (current density 15 A/dm^2), anode (A) and anode-cathode (AC) current modes) for 120 s, 240 s, 480 s, 960 s, respectively. Table 1 shows the technological parameters and oxidation time for each test sample.

Table 1

Technological parameters and process time of the studied samples

Sample No.	Mode	J, A/dm^2	t, s
1	A	15	120
2	A	15	240
3	A	15	480
4	A	15	960
5	AC	15	120
6	AC	15	240
7	AC	15	480
8	AC	15	960

Notations: A – anode mode; AC – anode-cathode mode; J – current density, A/dm^2; t – oxidation time, s.

Then, the topography of the surface of the selected samples was studied using a VEGA 3 scanning electron microscope, coating roughness measurements were carried out using a Mitaka FS150 3D laser contourograph, and the characteristics of the electrical parameters of MAO coatings were studied using a universal electrical strength meter. To do this, aluminum contacts were sprayed onto the samples at the UVN-71 P3 vacuum spraying unit, as shown in Fig. 1. Two electrodes were connected to the middle and one of the extreme contacts, between which a gradually increasing electrical voltage was created. The electrical strength was determined by the magnitude of the voltage at which the dielectric breakdown of the coating occurred.

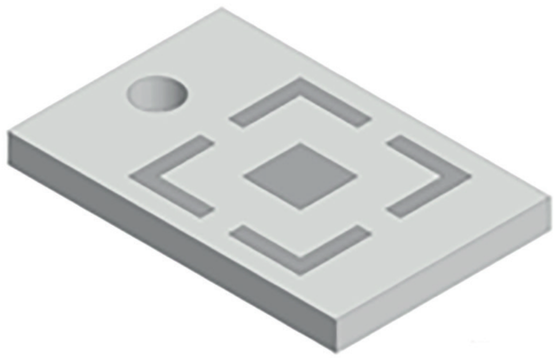

Fig. 1. The sample on which the MAO coating and metal contacts were sprayed

Results and discussion

Fig. 2 and 3 show topological images of the formed MAO coatings obtained using a VEGA 3 scanning electron microscope. Coatings on the samples were formed at a current density of 15 A/dm^2 in the anode and anode-cathode modes, depending on the oxidation time.

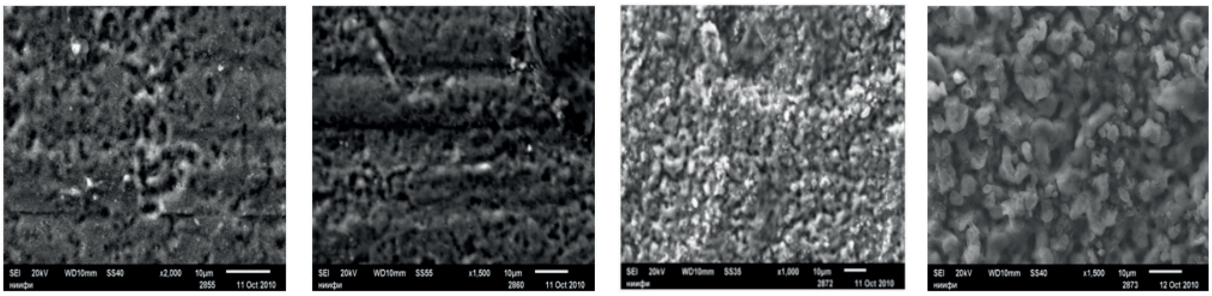

Fig. 2. Morphology of the surface of MAO coatings obtained at current density 15 A/dm^2 in anode mode for 120 s (*a*), 240 s (*b*), 480 s (*c*), 960 s (*d*)

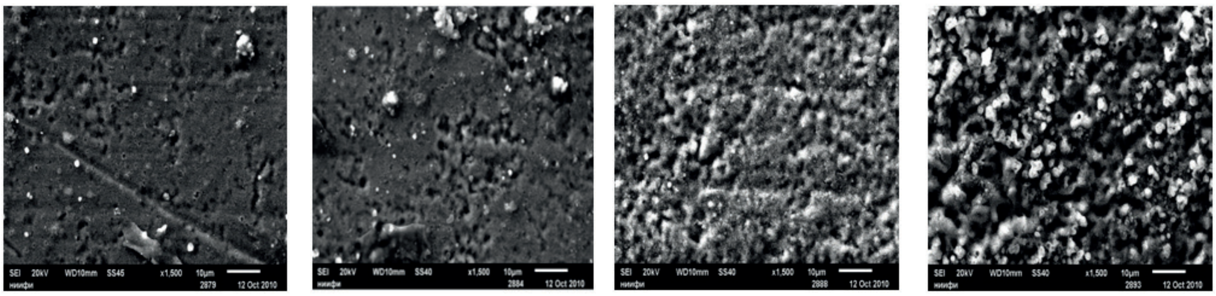

Fig. 3. Morphology of the MAO coatings surface obtained at current density 15 A/dm^2 in anode-cathode mode for 120 s (*a*), 240 s (*b*), 480 s (*c*), 960 s (*d*)

Analysis of morphology of the surface of samples with MAO coating (Fig. 2), as well as those obtained using the anode mode with current density 15 A/dm^2, confirmed that with increasing processing time of aluminum plates, the surface development, the size and number of pores increase. Thus, we can talk about the multilayering of the resulting ceramic coating. From the analysis It also follows that the first sample (Fig. 2, *a*), processed for the minimum time, is not porous and repeats the substrate structure. Such a coating can be attributed to the basic adhesive sub layer. Further exposure time leads to the appearance of small pores, and the coating

Table 2

The roughness of the MAO coatings samples obtained in the anode mode

Sample No.	Parameter	Side 1	Side 2
1	R_z, μm	2.62	2.33
	R_{max}, μm	3.72	3.00
	R_a, μm	0.17	0.32
2	R_z, μm	2.24	1.19
	R_{max}, μm	3.20	1.66
	R_a, μm	0.32	0.18
3	R_z, μm	2.56	12.90
	R_{max}, μm	3.55	55.00
	R_a, μm	0.35	0.68
4	R_z, μm	4.92	4.44
	R_{max}, μm	7.64	6.88
	R_a, μm	0.55	0.43

Notations: R_z – the height of the irregularities at ten points, μm; R_{max} – maximum height of irregularities, μm; R_a – the arithmetic mean deviation, μm.

structure gradually develops. Such layers have a well-developed structure. Thus, the fourth sample (Fig. 2, *d*) has a highly porous and developed structure.

For further analysis of the MAO process, the roughness of the samples surface was studied on a 3D laser conturograph profilometer. Table 2 contains the results of the measurements taken.

As a result of studying the values of the arithmetic mean deviation R_a, it was found that samples 1 (Fig. 2, *a*), 2 (Fig. 2, *b*) have them in the range from 0.1 to 0.3 μm. The information provided demonstrates that the coatings in the initial stages of growth have the same structure as the substrate due to the same surface roughness. Over time, the roughness of the samples gradually increases due to an increase in porosity and relief. Roughness values range from 0.1 to 0.7 μm.

At the second stage of the research, the following 4 aluminum plates were subjected to the MAO process in the mode of anode-cathode current (AC) with current density 15 A/dm^2.

Having analyzed the images of the surfaces of MAO coatings obtained using the anodic-cathode mode (Fig. 3), one can reveal a similar picture as in the anodic current mode. An increase in the processing time of aluminum plates leads to a complication of the surface structure of the formed coatings; accordingly, an increase in the size and number of pores is observed. Therefore, coatings obtained according to these modes can be considered multilayer, with a base adhesive coating, an intermediate porous and highly porous layer with a developed surface. In order to identify differences in the modes of obtaining coatings of the MAO process, an analysis of surface roughness was performed on a 3D laser conturograph profilometer. The obtained results of roughness measurements are presented in Table 3.

Table 3

Roughness of MAO coatings samples obtained in the anode-cathode mode

Sample No.	Parameter	Side 1	Side 2
5	R_z, μm	8.05	2.10
	R_{max}, μm	15.10	2.38
	R_a, μm	0.41	0.32
6	R_z, μm	3.10	2.32
	R_{max}, μm	5.78	2.63
	R_a, μm	0.32	0.34
7	R_z, μm	2.87	2.50
	R_{max}, μm	3.56	3.36
	R_a, μm	0.40	0.25
8	R_z, μm	2.58	2.40
	R_{max}, μm	3.23	2.83
	R_a, μm	0.59	0.43

Notations: R_z – the height of the irregularities at ten points, μm; R_{max} – maximum height of irregularities, μm; R_a – the arithmetic mean deviation, μm.

Analyzing the data given in Table 3, we can conclude that with increasing time of MAO treatment in the anode-cathode mode, the surface roughness increases, according to the arithmetic mean deviation Ra. The values of the first two samples (Fig. 3, *a*, *b*) are within 0.3 μm. The second two samples (Fig. 3, *c*, *d*) have values from 0.2 to 0.6 μm. The results obtained differ somewhat from the anode current mode. This is most likely due to plate defects. But we should not forget that the technological process itself contributes to the formation of oxide coatings.

Thus, the changes identified at the first and second stages of surface morphology studies are characteristic of the plasma coating growth model.

MAO coatings obtained using technological modes can be considered multilayer. They consist of a base layer with good adhesive properties, an intermediate porous layer and a top layer with high porosity and an actively functioning surface.

According to the results of data analysis, it can be concluded that with increasing the time of MAO process in anodic and anode-cathode modes, there is an increase in surface roughness determined by the values of arithmetic mean deviation R_a.

All samples with the obtained coatings, during the electrical strength tests, withstood a voltage of 600 V.

The process is characterized by high reproducibility and is also traceable dependence of coating properties (roughness, porosity, electrical strength) on processing time.

Conclusion

The topology of the MAO coatings surface on aluminum alloy samples, as well as their mechanical parameters, were studied. It has been revealed that an increase in the time of MAO treatment in the anode and anode-cathode modes leads to an increase in porosity and roughness of coatings. Tests for electrical strength showed that all the test samples withstood a breakdown voltage of 600 V. The research results allow the use of the plasma growth model of oxide coatings as the basis for the development of a digital process twin of microarc oxidation. The obtained empirical results are appropriate use as training data for intelligent algorithms as a part of digital twin.

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