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### **Metrological aspect of oxide coatings growth on products with an aluminum substrate**

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**Abstract.** The article presents the study results of the influence of process mode parameters on the thickness of the oxide coating of aluminum-based products. Ceramic-like coatings were formed by the microarc oxidation method on AD31 alloy using a capacitor process installation with a sinusoidal current at a constant current density of 15 A/dm<sup>2</sup> in the anodic-cathode mode. The study of oxide film growth was performed using a B7-517 thickness gauge. It is shown that for approximating the dependence of the oxide film thickness on the processing time of products, it is advisable to use piecewise approximation by mathematical functions of different types: exponential and linear. The use of such an approach is due to the influence of diffusion and chemical processes that dominate at the stages of spark and microarc discharges of the microarc oxidation process. However, this approach is not applicable for the purpose of analyzing the dependence of the film thickness on the concentration of sodium hydroxide and the ratio of the anodic and cathodic current components. In this case, the approximation is made by a quadratic function, due to the influence of nonlinear factors-impacts of the technological process. The results of the metrological analysis confirmed that the error in the adequacy of these models does not exceed 5%. It is advisable to use the obtained results when producing coatings with the required functional characteristics by the microarc oxidation method.

**Keywords:** microarc oxidation, digital twin, thickness, processing time, concentration, ratio of current components

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## Метрологический аспект роста оксидных покрытий на изделиях с алюминиевой подложкой

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**Аннотация.** Исследованы корреляционные зависимости, позволяющие оценить характер влияния параметров технологических режимов (время оксидирования, соотношение анодного и катодного составляющих тока, концентрация гидроксида натрия) на динамику формирования толщины микродугового оксидного покрытия на изделиях с алюминиевой основой. Керамикоподобные покрытия формировались на сплаве марки АД31 с использованием конденсаторной технологической установки процесса микродугового оксидирования на синусоидальном токе при постоянном значении плотности тока 15 А/дм<sup>2</sup> в анодно-катодном режиме. Непосредственное исследование роста оксидной пленки проводилось толщиномером В517-7. Аналитические выражения зависимости кинетики роста микродугового оксидного покрытия на изделиях с алюминиевой основой получены с использованием программной среды MatLAB. Показано, что для аппроксимации зависимости роста толщины оксидной пленки от времени обработки изделий, необходимо применять комбинированный подход, заключающийся в частичной аппроксимации математическими функциями разного вида: экспоненциальными и линейными. Использование такого подхода обусловлено влиянием диффузионных и химических процессов, доминирующих на стадиях искровых и микродуговых разрядов процесса микродугового оксидирования. Однако, такой подход не применим с целью анализа зависимости толщины пленки от концентрации гидроксида натрия и соотношения анодного и катодного составляющих тока. В этом случае аппроксимация производится квадратичной функцией, в следствии проявления нелинейных факторов-воздействий на технологический процесс. Метрологический анализ выражений показал, что погрешность адекватности этих моделей составляет не более 5%. Полученные результаты применимы как для определения погрешностей измерений параметров-воздействий процесса микродугового оксидирования, так и для получения покрытий с требуемыми функциональными характеристиками.

**Ключевые слова:** микродуговое оксидирование, цифровой двойник, толщина, время обработки, концентрация, соотношение составляющих тока

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### Introduction

The growth of oxide coatings formed by the microarc oxidation (MAO) method is characterized by an increase in thickness, the formation of a porous structure [1]. It should be noted that the coating itself grows not only over the metal base, but also penetrates into the substrate structure, modifying it. Based on studies of the dependence of the MAO coating thickness on the factors-impacts, it is possible to identify new analytical mathematical models. For example, in [2], it is shown that the coating thickness increases with processing time and has an exponential dependence. In [3], at the spark discharge stage, the dependence of the MAO coating thickness on the processing time is described by an exponential function, and at the micro-arc discharge stage, by a linear function. With an increase in the NaOH concentration in the silicate-alkaline electrolyte, the oxide film thickness decreases [4]. In connection with the introduction of digital twins of various technological processes into production cycles, the task of metrological analysis and control of the coatings, properties formed in real time and predicted during the digital twin operation is relevant [5]. The solution to this problem will improve the MAO coatings quality, the accuracy of reproducible performance characteristics, and technical and economic indicators when developing new technological modes.

### Materials and methods

For the purpose of metrological analysis of the growth process of microarc oxide coatings on products with an aluminum substrate, 20 samples with a microarc coating were obtained. The test samples have a rectangular shape with dimensions of 20×15×2 mm and a treated surface area of  $S = 0.05971 \text{ dm}^2$ . The main material of the sample substrate is AD31 alloy - aluminum alloyed with manganese, silicon, titanium. Before MAO treatment, the test samples were milled and then polished. After, the samples that had passed the preparation stage were placed in a galvanic bath

Table 1

**Parameters of the process modes for studying the influence of processing time, ratio of current components and NaOH concentration on the formation of coating thickness**

Sample No.	$t, \text{ s}$	$j, \text{ A/dm}^2$	$C_N, \text{ g/l}$	$I_A/I_C$
1	100	15	0.5	1.00
2	200	15	0.5	1.00
3	300	15	0.5	1.00
4	600	15	0.5	1.00
5	900	15	0.5	1.00
6	1200	15	0.5	1.00
7	1500	15	0.5	1.00
8	1800	15	0.5	1.00
9	1800	15	0.5	0.25
10	1800	15	0.5	0.50
11	1800	15	0.5	1.00
12	1800	15	0.5	1.25
13	1800	15	0.5	1.50
14	1800	15	0.5	1.75
15	1800	15	0.5	1.00
16	1800	15	1.0	1.00
17	1800	15	1.5	1.00
18	1800	15	2.0	1.00
19	1800	15	2.5	1.00
20	1800	15	3.0	1.00

Notations:  $t$  is the oxidation time, s;  $j$  is the current density, A/dm<sup>2</sup>;  $C_N$  is the concentration of sodium hydroxide, g/l;  $I_A/I_C$  is the ratio of the anodic and cathodic current components.

of a microarc oxidation installation with electrolyte containing  $C_{NaSi}$  80 g/l  $Na_2SiO_3$  and  $C_N$  0.5, 1, 1.5, 2, 2.5 and 3 g/l NaOH. The current density  $j$  in the anodic-cathode mode was constant and amounted to 15 A/dm<sup>2</sup>. The ratio of the anodic and cathodic current components  $I_A/I_C$  varied in the range from 0.25 to 1.5 (in increments of 0.25). The duration of sample treatment  $t$  was 100, 200, 300, 600, 900, 1200, 1500 and 1800 s. The thickness of the obtained oxide coatings was measured using a B7-517 thickness gauge. Using the MatLAB software environment, expressions for the dependences of the coating thickness on the factors affecting the structure of the coating itself were obtained, and graphs of the obtained dependences were constructed. Errors in indirect measurements of the thickness of the formed oxide coating were calculated.

Table 1 shows the parameters of the process modes under which the influence of the duration of product processing (samples 1–8), the ratio of current components (samples 9–14) and the concentration of NaOH (samples 15–20) on the coating thickness was studied.

### Results and discussion

Table 2 presents the results of measuring the thickness of the coatings obtained under different process modes, respectively, the graphs of the influence of process parameters on the coating thickness are shown in Fig. 1.

Table 2

Results of measuring the thickness of the formed MAO coatings

Sample No.	$t$ , s	$h(t)$ , $\mu\text{m}$	Sample No.	$I_C/I_A$	$h(I_A/I_C)$ , $\mu\text{m}$	Sample No.	$C_N$ , g/l	$h(C_N)$ , $\mu\text{m}$
1	100	0.68	9	0.25	29.53	15	0.5	28.64
2	200	2.07	10	0.50	19.11	16	1.0	29.32
3	300	6.68	11	0.75	12.37	17	1.5	28.27
4	600	8.98	12	1.00	7.99	18	2.0	27.76
5	900	12.17	13	1.25	5.18	19	2.5	27.39
6	1200	16.15	14	1.50	3.99	20	3.0	26.75
7	1500	22.28						
8	1800	29.98						

Notations:  $h(t)$  is the coating thickness from the oxidation time,  $\mu\text{m}$ ;  $h(I_A/I_C)$  is the coating thickness from the ratio of the current components,  $\mu\text{m}$ ;  $h(C_N)$  is the coating thickness from the concentration of NaOH,  $\mu\text{m}$ .

As a result of processing the measurements results of the obtained MAO coatings thickness by the least squares method, analytical mathematical models of the studied dependencies of the oxide coating thickness on the oxidation time (1), the ratio of the anodic and cathodic current components (2) and the concentration of sodium hydroxide (3), respectively, were identified. The obtained mathematical expressions are given below:

$$h(t) = \begin{cases} e^{0.0058 \cdot t} - 1; & 0 < t \leq 300, \\ 0.0022 \cdot t + 3.4747; & 300 \leq t \leq 1800, \end{cases} \quad (1)$$

$$h(I_A/I_C) = 21.243 \cdot (I_A/I_C)^2 - 59.0496 \cdot I_A/I_C + 43.9807, \quad (2)$$

$$h(C_N) = -0.2643 \cdot C_N^2 + 0.0107 \cdot C_N + 28.95. \quad (3)$$

The graphs of the corresponding functions are shown in Fig. 1, *a–c*.

The study of the graphs (Fig. 1, *a–c*) showed that the dependence of the formed oxide film thickness on the oxidation time is advisable to approximate piecewise by mathematical functions of different types: exponential and linear according to expression (1). The use of this approach is due to the influence of diffusion and chemical processes dominating at the stages of spark and microarc discharges of the microarc oxidation process, which is confirmed by other studies [3].

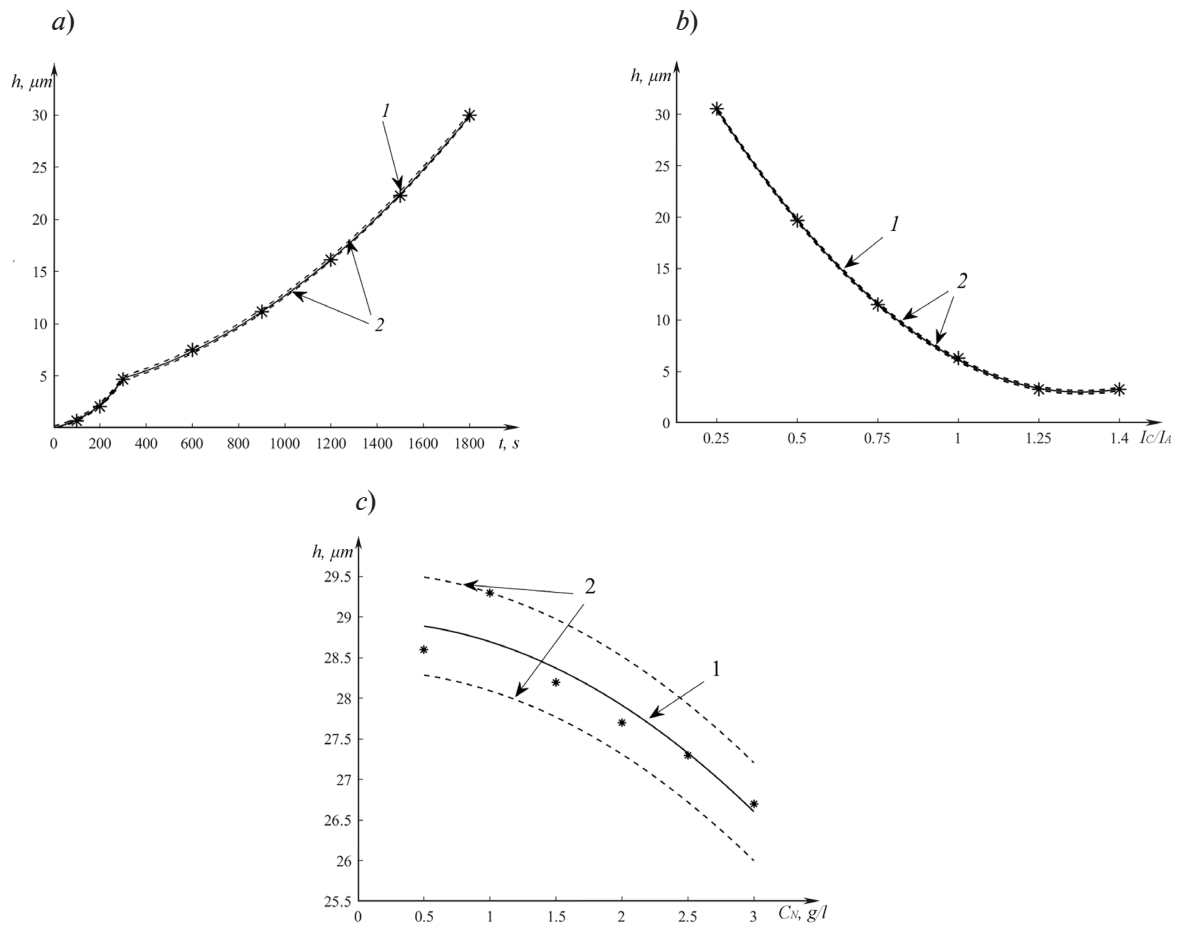


Fig. 1. Dependences of the coating thickness on the processing time  $t$  (a), on the ratio of the anodic and cathodic current components  $I_c/I_a$  (b) and on the NaOH concentration  $C_N$  (c): 1 is the approximating curve; 2 is the upper and lower error limits that form the uncertainty region; experimental data works in dots

However, such an approach is not applicable for the purpose of analyzing the dependence of the film thickness on the concentration of sodium hydroxide and the ratio of the anodic and cathodic components of the current. In this case, the approximation is made by a quadratic function due to the manifestation of nonlinear factors—impacts on the technological process.

A metrological analysis of the dynamics of a microarc oxide coating formation on products with an aluminum substrate was carried out. The error in the adequacy of the obtained mathematical expressions does not exceed  $\pm 5\%$ , which is confirmed by the lower and upper boundaries of the uncertainty regions shown in Fig. 1. It is advisable to use the obtained results when obtaining coatings with the required functional characteristics by the microarc oxidation method.

### Conclusion

The results of the metrological analysis of the MAO coatings growth and the identified mathematical models should be used in developing a digital twin of the MAO process. The obtained analytical expressions are described by different mathematical functions due to the presence of both diffusion and chemical processes dominating at the stages of arc and microarc discharges, and the nonlinearity of the factors influencing the technological process. The dependence of the thickness growth of the ceramic-like coating on the duration of processing of a product made of valve metal or alloy is described by exponential and linear functions, and the dependence of the thickness on the ratio of the current components and the concentration of sodium hydroxide is described by quadratic functions. The error in the adequacy of the obtained models does not exceed 5%.

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