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Influence of process parameters on the properties of microarc oxide coatings

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Abstract. In this work, oxide coatings on aluminum samples were obtained by the method of micro-arc oxidation at a sinusoidal current in an anode-cathode alloy with an anode and cathode current ratio of 1, with a current approximation of 10.88; 13.99; 17.10; 20.21; 23.32 A/dm^2 in four electrolytes containing 0.5 g/l NaOH and 80, 90, 100 and 110 g/l Na_2SiO_3. An analytical description of the thickness and porosity dependence of micro-arc oxide coatings on the decrease in current, treatment time, and electrolyte components detection in the form of empirical regression formulas is obtained. Based on the obtained equations a technology for the formation of micro-arc oxide coatings with desired properties was proposed. As a result of experimental verification, the reproducibility of the technology for obtaining micro-arc oxide coatings with a thickness of 25 μm and minimal porosity (P = 19.5%) was confirmed. The relative error of the appearance reproducibility does not exceed ± 0.5%. The results of the study were used in the development of intelligent algorithms that underlie the digital twin of the micro-arc oxidation process.

Keywords: micro-arc oxidation, digital twin, the relationship of technological parameters and properties of coatings, empirical regression formulas, technique for obtaining coatings with desired properties

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Влияние параметров технологического процесса на свойства микродуговых оксидных покрытий

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Аннотация. В данной работе получены оксидные покрытия на алюминиевых образцах методом микродугового оксидирования на синусоидальном токе в анодно-катодном режиме с соотношением анодного и катодного токов, равном 1, при плотности тока,

равной 10,88; 13,99; 17,10; 20,21; 23,32 А/дм^2 в четырех электролитах, содержащих 0,5 г/л NaOH и 80, 90, 100 и 110 г/л Na_2SiO_3. Получено аналитическое описание зависимостей толщины и пористости микродуговых оксидных покрытий от плотности тока, времени обработки и концентрации компонентов электролита в виде эмпирических регрессионных формул. На основе полученных уравнений предложена методика формирования микродуговых оксидных покрытий с заданными свойствами. В результате экспериментальной проверки подтверждена работоспособность данной методики путем формирования микродуговых оксидных покрытий с толщиной 25 мкм и минимальной пористостью (P = 19.5%). Относительная погрешность воспроизводимости толщины покрытий не превышает ± 0.5%. Результаты исследования могут быть использованы при разработке интеллектуальных алгоритмов, лежащих в основе цифрового двойника процесса микродугового оксидирования.

Ключевые слова: микродуговое оксидирование, цифровой двойник, взаимосвязь технологических параметров и свойств покрытий, эмпирические регрессионные формулы, методика формирования покрытий с заданными свойствами

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Introduction

Micro-arc oxidation (MAO) is a promising technological process of plasma-chemical modification of the light metals and alloys surface. Oxide coatings formed by this method have special properties and are used in many industries: automotive, oil and gas, rocket and space, aviation, electronics, medicine, etc. [1–5].

Currently, there are problems associated with the technological features of the MAO process, which hinder its industrial implementation. First of all, this is the insufficient study of the mechanism of the oxide layers formation and the combined influence of many factors on the synthesized coatings properties [6]. This causes certain difficulties in the selection of technological parameters, which leads to an increase in the energy consumption of the coating process.

A promising method for solving such problems is the development of digital twins of technological processes [7] using intelligent algorithms, which the correct operation requires a large amount of experimental data. For example, when training neural networks, in order to build training and control samples that are consistent with the results of a real MAO process, it is convenient to use regression formulas for the dependences of coating properties on process parameters and influencing factors. A review of foreign literature [8, 9] showed that not all of these dependences have a mathematical description. In addition, the analytical description of the same dependences in the works of different authors differs significantly, which is due to the peculiarities of specific technological regimes for various applications of MAO coatings [10, 11]. In this regard, obtaining empirical regression formulas for the dependences of coating properties on the technological process parameters is an urgent scientific task.

Materials and Methods

MAO coatings were obtained on samples of commercial aluminum grade AD31T1 with 23×15×1.5 mm dimensions. As a current lead, pieces of aluminum wire with a diameter of 1.8 mm were used, which were insulated with a polyolefin heat shrink tube. MAO processing was

carried out on a thyristor-capacitor automated MAO installation in the anode-cathode mode at a ratio of anode and cathode currents equal to 1, in four electrolytes containing 0.5 g/l NaOH and Na_2SiO_3 at a concentration of 80, 90, 100 and 110 g/l. The current density was 10.88; 13.99; 17.10; 20.21; 23.32 A/dm^2. The processing time was chosen according to Table 1 so that for each current density there were samples corresponding to the stages of anodization, spark and micro-arc discharges.

Table 1

Oxidation time

Current density, A/dm^2	t_1, s	t_2, s	t_3, s	t_4, s
10.88	60	240	600	900
13.99	60	240	420	600
17.10	60	120	240	420
20.21	60	120	240	420
23.32	60	120	180	240

Notations: $t_1 - t_4$ are the oxidatiom times.

The geometric dimensions and mass of the sample were measured before and after MAO treatment. The length and width of the sample were measured with a caliper with a digital reading device with a resolution step of 0.01 mm; the thickness was measured with a Syntek micrometer with a digital reading device with a discrete step of 0.001 mm at five points, after which the average value was calculated. The mass of the sample was measured using digital jewelry scales of the 8028 series (the main error in measuring the mass in the range from 0 to 100 g is $\pm$ 0.001 g). The thickness h and bulk porosity P of the samples were calculated using the formulas:

$$h = \frac{d_{2sr} - d_{1sr}}{2}, \tag{1}$$

$$P = \frac{m_2 - m_1}{\rho_{Al_2O_3}\left(a_{2sr}b_{2sr}d_{2sr} - a_{1sr}b_{1sr}d_{1sr}\right)} \cdot 100\%, \tag{2}$$

where m_1, m_2 is the mass of the sample without coating and with coating, $\rho_{Al_2O_3}$ is the density of aluminum oxide, a_{1sr}, b_{1sr}, d_{1sr}, a_{2sr}, b_{2sr}, d_{2sr} are the average geometric dimensions of the sample before and after coating. The deviation of the thickness and porosity of the synthesized coatings from the required values was determined as a relative error.

Results and Discussion

In the course of the study, a regression analysis of the experimental curves was performed, as a result of which regression equations were obtained for the thickness and porosity dependences of the coating on the current density, treatment time, and concentration of Na_2SiO_3 in the electrolyte in the form of exponential functions. As an example, Fig. 1–3 show graphs of approximating functions constructed using these equations.

The resulting regression equations are intended to form a database of a digital twin of the micro-arc oxidation process and can be used to automatically select the parameters of the technological regime for deposition of MAO coatings with desired properties, for which an appropriate technique has been developed. One of the options for the selection of technological parameters is described below.

It is necessary to obtain a coating with a thickness of 25 μm with a minimum porosity; deviation of the coating thickness from the required value should not exceed $\pm$ 0.5%. To do this, perform the following steps:

1. We choose an electrolyte in which, at the minimum current density, coatings are formed that satisfy the problem condition. To do this, from Fig. 1, *a* it is necessary to find the processing time t corresponding to a thickness of 25 μm for each electrolyte by graphical analysis or by solving the appropriate regression equations with respect to t, and then from Fig. 1, *b* determine the porosity corresponding to time t. The obtained values are shown in Table 2. It can be seen that the coatings obtained in electrolyte No. 1 have the smallest porosity.

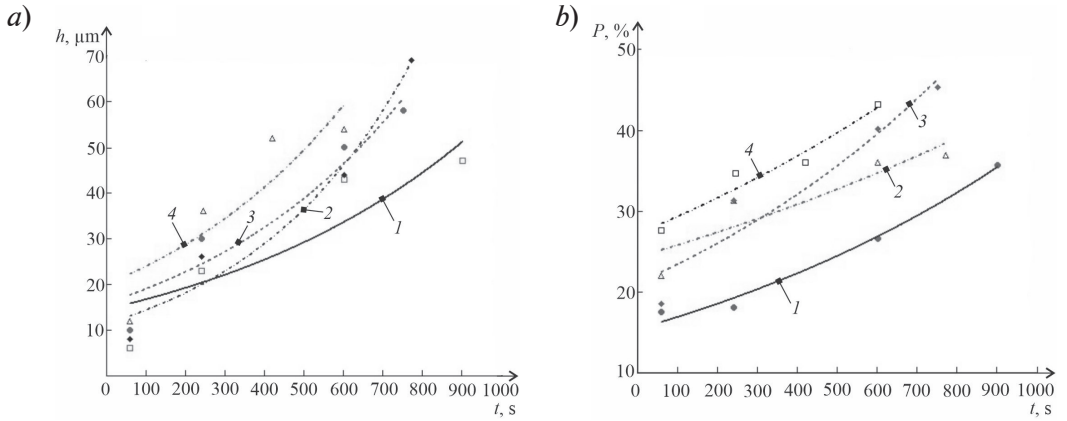

Fig. 1. Dependences of the coating thickness h (*a*) and porosity P (*b*) on time t for the current density j = 10.88 A/dm^2 and Na_2SiO_3 concentration equal to: *1* – 80 g/l; *2* – 90 g/l; *3* – 100 g/l; *4* – 110 g/l

Table 2

Choice of electrolyte composition

Electrolyte No.	C_K, g/l	C_N, g/l	Oxidation time, t, s	Porosity of coating, P, %
1	0.5	80	420	23
2	0.5	90	360	30
3	0.5	100	270	27
4	0.5	110	140	31

Notations: C_K and C_N are the concentrations of NaOH and Na_2SiO_3 in the electrolyte.

2. Similarly to item 1 in Fig. 2, *a*, we determine the processing time to form a coating with a thickness of 25 μm for different current densities in electrolyte No. 1, as well as the porosity corresponding to this processing time (from Fig. 2, *b*). According to the obtained results (Table 3), in this case, it is advisable to choose the current density j = 20.21 A/dm^2 due to the low porosity of the coating.

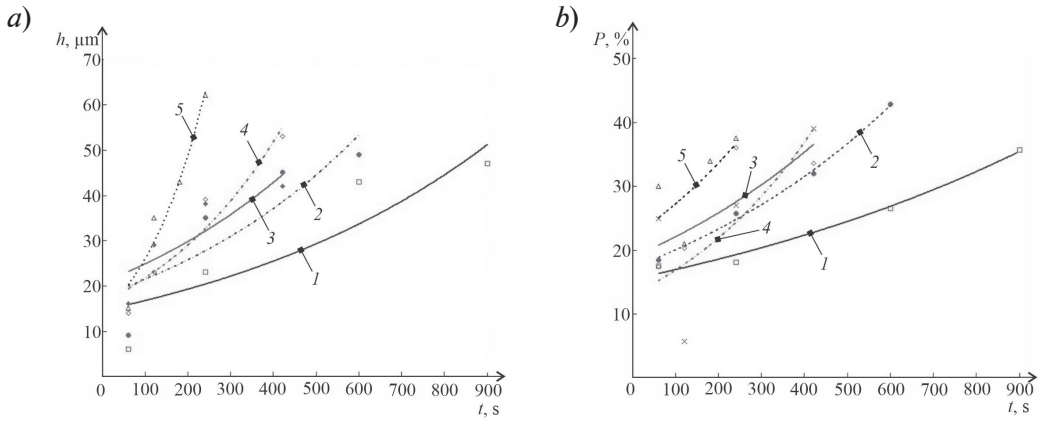

Fig. 2. Dependences of the coating thickness h (*a*) and porosity P (*b*) on time t for the electrolyte containing 80 g/l of Na_2SiO_3 and current density equal to: *1* – 10.88 A/dm^2; *2* – 13.99 A/dm^2; *3* – 17.10 A/dm^2; *4* – 20.21 A/dm^2; *5* – 23.32 A/dm^2

3. In the first approximation, we have the following technological parameters of MAO treatment: electrolyte No. 1 (0.5 g/L NaOH and 80 g/L Na_2SiO_3), current density j = 20.21 A/dm^2, treatment time t = 150 s. To check the correctness of the selected mode according to Fig. 3, we determine the thickness and porosity corresponding to the processing time of 150 s. We have the following values: coating thickness h = 25.9 μm, porosity P = 19.4%, which corresponds to the results obtained earlier.

Table 3

Choice of current density

Current density, j, A/dm^2	Oxidation time, t, s	Porosity of coating, P, %
10.88	400	22
13.99	190	23
17.10	110	22
20.21	150	19
23.32	100	27

MAO coating, obtained on an automated MAO installation with selected technological parameters, has a thickness and porosity, respectively, equal to 26 μm and 19.5%; the relative error in reproduction of the coating thickness is 0.39%, which satisfies the stated requirements.

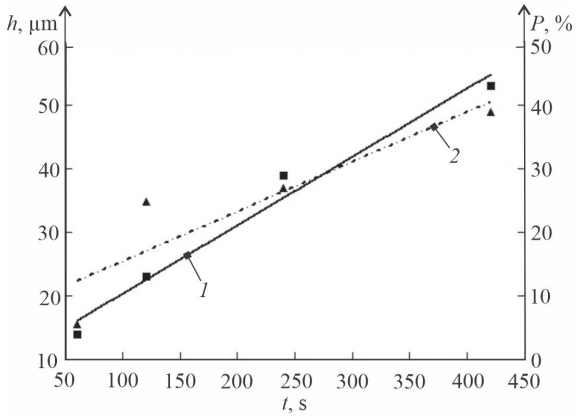

Fig. 3. Dependence of the coating thickness h (curve 1) and porosity P (curve 2) on time t for the current density j = 20,21 A/dm^2 and Na_2SiO_3 concentration equal to 80 g/l

Conclusion

Thus, the proposed method for the oxide layers formation makes it possible, on the basis of the obtained regression equations of experimental dependences of the thickness and porosity of coatings on the technological parameters of the MAO process, to select the optimal technological regime for the synthesis of MAO coatings with desired properties. The software implementation of the proposed technique using neural networks or optimization algorithms will significantly improve the properties reproducibility of micro-arc oxide coatings in industrial production.

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