

*Наведено результати дослідження фазового складу і властивостей МДО-покримтів на алюмінієвих сплавах. Покриття були одержані в лужно-селикатному електроліті на змінному синусоїdalному струмі і в імпульсному режимі струму. Показано, що підвищена щільність мікророзрядів при імпульсній технології збільшує сумарну енергію, що виділяється в них. Це обумовлює підвищення швидкості зростання оксидного покриття і ймовірність утворення  $\alpha\text{-Al}_2\text{O}_3$  фази. Одержані при мікроплазмовому оксидуванні в імпульсному струмовому режимі покриття мають високу твердість і електричну міцність*

*Ключові слова: структурна інженерія, анодно-катодний режим, товщина покримтів, фазовий склад, корунд, властивості*

*Приведены результаты исследования фазового состава и свойств МДО-покрытий на алюминиевых сплавах, полученных в щелочно-селикатном электролите на переменном синусоидальном токе и в импульсном токовом режиме. Показано, что увеличенная плотность микроразрядов в случае импульсной технологии повышает суммарную энергию, выделяемую в них. Это обуславливает повышение скорости роста оксидного покрытия и вероятность образования  $\alpha\text{-Al}_2\text{O}_3$  фазы. Полученные при микроплазменном оксидировании в импульсном токовом режиме покрытия имеют высокую твердость и электрическую прочность*

*Ключевые слова: структурная инженерия, анодно-катодный режим, толщина покрытия, фазовый состав, корунд, свойства*

# EFFECT OF ELECTROLYSIS REGIMES ON THE STRUCTURE AND PROPERTIES OF COATINGS ON ALUMINUM ALLOYS FORMED BY ANODE-CATHODE MICRO ARC OXIDATION

V. Belozerov

PhD, Professor\*

O. Sobol'

Doctor of Physics and Mathematics Sciences, Professor\*

E-mail: sool@kpi.kharkov.ua

A. Mahatilova

PhD, Senior Researcher\*

V. Subbotina

PhD, Associate Professor\*

Taha A. Tabaza

PhD, Associate Professor\*\*

Ubeidulla F. Al-Qawabeha

PhD, Associate Professor

Department of Mechanical Engineering

Tafila Technical University

P O Box, 179, Tafila, Jordan, 66110

Safwan M. Al-Qawabah

PhD, Associate Professor\*\*

\*Department of Materials Science

National Technical University "Kharkiv Polytechnic Institute"

Kypruchova str., 2, Kharkiv, Ukraine, 61002

\*\*Department of Mechanical Engineering

Al-Zaytoonah University

Queen Alia Airport str., 594, Amman, Jordan, 11733

## 1. Introduction

At present, structural engineering is the basic method of creating materials with high functional properties [1, 2]. This is determined by a significant expansion of the possibilities of managing the structural state in nonequilibrium conditions, which is characteristic of modern methods of obtaining materials [3, 4]. Based on structural engineering, the highest mechanical properties have been achieved through the design of the material in nonequilibrium conditions at the nanoscale. High properties are obtained by ordering supersaturated metal solutions [5], forming supersaturated systems of interstitial phases [6], creating nanolayers of "metal – interstitial phases" systems [7] or by using several

interstitial phases [8] and formation of multielement solid solutions [9].

The possibilities of structural surface engineering in recent years have been significantly expanded [10]. This became possible due to the use of plasma technologies with a very high degree of disequilibrium in the process of material formation (ultrafast thermalization) [11, 12]. As a result of using such technologies, it is possible to obtain new phase states [13], form highly supersaturated solid solutions [14], create the necessary structural-stress state [15], and create special configurations of atoms [16, 17]. Moreover, it is possible to achieve high performance properties by creating multiperiod structures [18, 19] or specially mutually oriented multiphase materials [20].

To obtain highly effective protective coatings on valve materials, an anode spark electrolysis method has recently been introduced into the industry [21, 22] (often called micro arc oxidation (MAO) [23]). This method is based on using an anode spark discharge phenomenon.

Micro arc oxidation is an electrochemical process that occurs at high electric field intensity [24] and is accompanied by the formation of microplasma [25]. In this case, micro regions with high pressure are formed due to the gases formed [26]. This leads to the occurrence of high-temperature chemical transformations [27] and the transport of matter in the arc [28].

High efficiency of the method creates good prospects for wide industrial use of the micro arc oxidation technology for modifying the surface of valve metals (aluminum, titanium, magnesium, etc.). Therefore, nowadays, an important task is to develop the scientific basis for managing the properties of MOA-processed materials on the basis of the results of structural engineering.

## 2. Literature review and problem statement

Aluminum and its alloys on the scale of production and use in the industry are in the top ratings. Corrosion resistance of aluminum and its alloys depends both on the amount of impurities or special additives to the alloy and on the quality of the protective film [29]. The natural aluminum oxide film is not considered sufficient to protect the base metal (alloy) from corrosion; this film is artificially thickened and compacted, depending on the requirements imposed on the work characteristics [30]. The most promising way to harden the surface and apply protective coatings to parts made of aluminum alloys is micro arc oxidation [31]. Oxidation is usually carried out by electrolysis with a voltage of up to 1000 V [32] and a current density of up to 100 A/dm<sup>2</sup> [33]. This makes it possible to create sparking of micro arc and arc discharges in aluminum-based alloys [34]. Micro arc discharges produce a flow of plasmochanical [35] and thermochemical [36] reactions as a result of which high-temperature modifications of aluminum oxides are formed [37].

The oxidation technology in conditions of micro arc discharges is very promising because it allows using parts made of aluminum alloys for obtaining ceramic coatings that are firmly associated with the substrate. Such coatings have high hardness, thermal and corrosion resistance, wear resistance and have antifriction properties. Due to these properties, aluminum-based coatings are increasingly used in machine building, aerospace engineering, instrumentation, and repairing.

By varying the composition of the electrolyte and changing the electrical processing modes, it is possible to obtain ceramic-like coatings with unique functional properties. However, due to the lack of targeted comparative studies on the kinetics of coating growth on the surface of various aluminum alloys, there arises the problem of a long empirical selection of optimal micro arc oxidation regimes. This problem can be largely solved by establishing general laws governing the formation of the structural-phase state (for structural engineering). At the same time, based on the results of structural engineering, it is possible to conduct an express comparison for widely differing technologies. Such technologies include the production of coatings by micro

arc oxidation in an alternating sinusoidal current and in a pulsed mode.

## 3. The aim and objectives of the study

The aim of this work is to determine the regularities of phase formation in coatings on an aluminum alloy, depending on the type and duration of the current regime for micro arc oxidation.

To achieve this aim, the following tasks are set and accomplished:

- to study the features of the formation of the oxide phase on an aluminum alloy, depending on the type of the current regime;
- to establish the dependence of the coating thickness on the duration and type of the current regime for micro arc oxidation;
- to determine the features of the phase formation during MOA treatment for small and large thicknesses of the oxide layer being formed;
- to describe the mechanism of formation of a high-temperature  $\alpha\text{-Al}_2\text{O}_3$  phase in micro arc oxidation.

## 4. Conditions for obtaining MOA coatings and methods for researching them

The tests involved processing samples made of the aluminum alloy D16T (Al base; the main alloying elements are Cu (up to 4.9 %) and Mg (up to 1.8 %)) in a size of 50×50×3 mm in a 100-liter bath. During the MAO process, the electrolyte was cooled and bubbled. The electrolyte was based on an aqueous solution of potassium hydroxide with the addition of liquid glass.

The power source applied in the work provided the following modes of operation:

- an alternating current at a frequency of 50 Hz, with the ratio of the anode and cathode current components  $I_a/I_k=1$  (the source of the capacitor type);
- a pulsed current as the power source producing bipolar voltage impulses of a rectangular shape and providing adjustment of the duration of each of them. The frequency and the amplitude of the voltage pulses were also regulated.

The studied features were the kinetics of formation of coatings, phase composition, microhardness, and electrical strength.

The phase composition of MOA coatings was determined based on the results of X-ray phase analysis. The tests were carried out on DRON-3 (Burevesnik, Russia) in monochromatized  $K_{\alpha}$ -Cu radiation. The results were registered in a pointwise mode with a step of  $2\theta=0.1^\circ$ . The maximum error in determining the content of the structural crystalline constituents (with detectability of 10 vol %) did not exceed  $\pm 0.7\%$ . The minimum detectability of the structural constituents was about 1 vol %.

For the quantitative phase analysis, the reference mixture method was used [38].

The thickness of the coating was determined using the VT-10 NC instrument (Control Instrument, Russia). The error in measuring the thickness of the coating was not more than 5 % with the smallest coating thickness (about 10  $\mu\text{m}$ ). If the thickness of the coating was thicker, the accuracy of

determining the thickness increased (for example, at a thickness of 50  $\mu\text{m}$ , the measurement error did not exceed 2 %).

The microhardness was determined on the PMT-3 instrument (LOMO, Russia). The electrical strength was determined by the breakdown voltage.

### 5. The results of studying the influence of the MAO treatment modes on the thickness of the layers and the phase composition

The dependence of the coating thickness on the oxidation time for different oxidation regimes is shown in Fig. 1. The treatment was carried out in an electrolyte of the composition  $\text{KOH} - 1 \text{ g/l} + \text{Na}_2\text{SiO}_3 - 3 \text{ g/l}$  at a current density of  $j = 20 \text{ A/dm}^2$ .

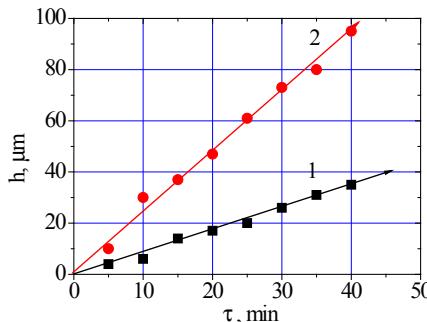


Fig. 1. The kinetics of the MOA coating formation:  
1 – a sinusoidal current, 2 – a pulsed current

The linearity of the obtained dependence indicates that the thickness of the coating is proportional to the deposition time, and, accordingly, to the amount of electricity that has passed. However, the pulse technology provides a high speed of the coating formation. Thus, with a sinusoidal current, the coating formation rate is about 1  $\mu\text{m}/\text{min}$ , and with a current in the pulsed mode, the growth rate of the coating is about 2  $\mu\text{m}/\text{min}$ . This result shows that the dependence of the growth rate of the coating on the current is affected by the form of the polarization pulses.

In the process of the coating growth, heat transfer and mass transfer through the oxide layer play an important role. In connection with the limitation of the heat dissipation, the fusion processes increase (Fig. 2). Such a fusion in a pore region results in a decrease in the through porosity of the oxide coating.

The energy released in the channels of microdischarges should ensure the formation of a high-temperature modification of aluminum oxide. Therefore, the shape of the current (continuous (sinusoidal) or pulsed), passing between the electrodes, must influence the phase composition of the coating.

The process of increasing the coverage results in visually clearly registered different density, mobility and brightness of discharges for various types of processing. A high density of discharges (in the pulsed processing mode) and their high mobility influence the phase formation.

With a small thickness of the oxide layer, the high rate of heat transfer to both the metal and the electrolyte facilitates the development of aluminum oxide in the form of the  $\gamma\text{-Al}_2\text{O}_3$  phase (spectrum 1 in Fig. 3). Over time, the discharges begin to fade not on the surface of the metal but on the surface of the formed oxide layer. In this case, the en-

ergy concentration in this oxide layer occurs, which causes the formation of a high-temperature modification of  $\alpha\text{-Al}_2\text{O}_3$  (spectrum 2 in Fig. 3). With increasing the coating thickness, the content of  $\alpha\text{-Al}_2\text{O}_3$  for different process regimes has a different limiting value (Fig. 4).

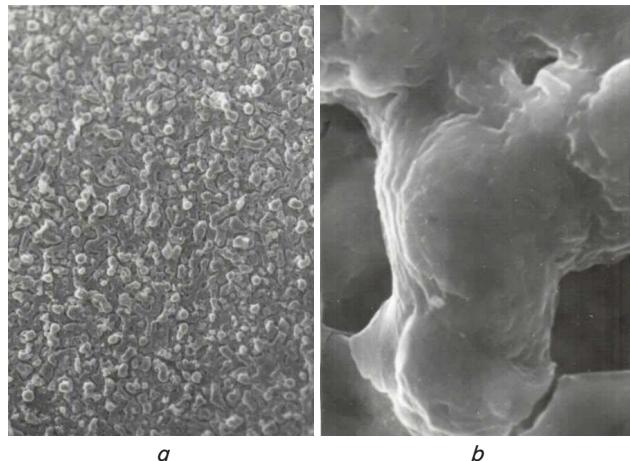


Fig. 2. The morphology of the surface of the MOA coating at different magnifications: a –  $\times 300$ , b –  $\times 7000$

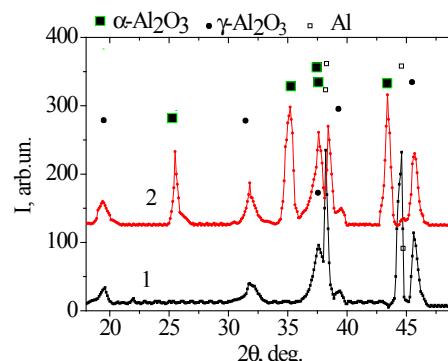


Fig. 3. A fragment of the diffraction patterns of the MOA coating on the D16T alloy ( $K_\alpha$ -Cu radiation): 1 – the coating thickness of 20  $\mu\text{m}$ , 2 – the coating thickness of 50  $\mu\text{m}$

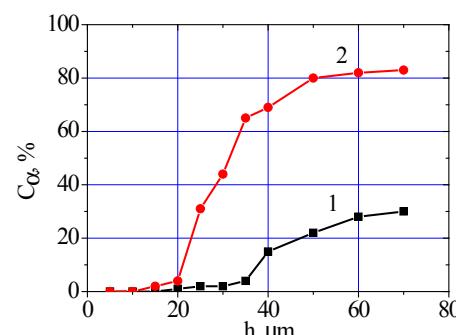


Fig. 4. The content of the  $\alpha\text{-Al}_2\text{O}_3$  phase for different coating thicknesses and different treatment modes:  
1 – the sinusoidal current, 2 – the pulsed current

The high density of microdischarges in the case of the pulse technology increases the total energy released in them, which increases the probability of formation of the  $\alpha\text{-Al}_2\text{O}_3$  phase. In this case, the maximum achievable percentage of the content of the  $\alpha\text{-Al}_2\text{O}_3$  phase in the coating is greater than 80 vol. % (Fig. 4, dependence 2).

## 6. Discussion of the results of studying the effect of current regimes under micro arc oxidation on the structure and kinetics of coating growth

It can be seen from the obtained results that the formation of various modifications of the aluminum oxide is due to the uneven heating of its various sections. When the local volumes of the coating are heated by micro arc discharges (the temperature is more than 2500 °C), the  $\gamma\text{-Al}_2\text{O}_3$  phase passes into the  $\alpha\text{-Al}_2\text{O}_3$  phase, i. e., there occurs the polymorphous transformation of  $\gamma\rightarrow\alpha$ . However, apparently, we cannot ignore the influence of the energy of formation of the crystallization centers for  $\gamma\text{-Al}_2\text{O}_3$  and  $\alpha\text{-Al}_2\text{O}_3$ . Since the formation energies of these phases are different, the phase formation is complex, on the basis of both  $\gamma\text{-Al}_2\text{O}_3$  and  $\alpha\text{-Al}_2\text{O}_3$  phases.

The difference in the phase composition of coatings formed under different regimes determines the difference in their properties. Thus, when using the pulse technology, the maximum hardness is 23 GPa, and with the variable sinusoidal current, it is 14 GPa. The electrical strength is 20 V/ $\mu\text{m}$  for the pulsed coating technology and only about 10 V/ $\mu\text{m}$  with using the variable sine wave.

Thus, the coatings obtained by microplasma (micro arc) oxidation in the pulsed current mode have a unique combination of properties – high hardness and electrical strength. Therefore, the use of this technology is promising in aerospace engineering and automotive engineering. Meanwhile, it is still essential to solve the problem of reproducibility of the results for parts of different sizes and geometry of the surface.

Further research is planned in the direction of optimizing the technological regimes in order to stabilize the

high-temperature  $\alpha\text{-Al}_2\text{O}_3$  phase for parts of different surface geometries and to increase the growth rate of the oxide coating.

## 7. Conclusion

1. Micro arc treatment of aluminum alloys in an alkali-silicate electrolyte using a pulse technology and an anode-cathode regime helps form coatings consisting mainly of oxides of the  $\alpha\text{-Al}_2\text{O}_3$  type (corundum). The high density of microdischarges in the case of the pulse technology increases the total energy released in them, which increases the probability of formation of the  $\alpha\text{-Al}_2\text{O}_3$  phase.

2. A linear dependence of the thickness of the coating on the duration of the process time and, accordingly, on the amount of transmitted electricity has been established. The greater tangent of the angle of inclination indicates that the pulse technology provides a higher growth rate of the coating.

3. It has been found that, with a small thickness of the oxide layer, the high rate of heat transfer both to the metal and to the electrolyte promotes the formation of aluminum oxide in the form of the  $\gamma\text{-Al}_2\text{O}_3$  phase. The energy concentration in a thick oxide layer causes the formation of a high-temperature modification of  $\alpha\text{-Al}_2\text{O}_3$ .

4. It has been proven that the mechanism of the formation of  $\alpha\text{-Al}_2\text{O}_3$  is determined by the action of two facts: the difference in the energies of the formation of the  $\gamma\text{-Al}_2\text{O}_3$  and  $\alpha\text{-Al}_2\text{O}_3$  phases as well as the polymorphic high-temperature transformation of  $\gamma\text{-Al}_2\text{O}_3\rightarrow\alpha\text{-Al}_2\text{O}_3$  in the high-temperature region of a micro arc discharge.

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