

Наведено дослідження щодо впливу режимів мікроплазмового оксидування в активованих додатками електролітах на фазово-структурний стан покриттів, що формуються на основі алюмінію. Виявлено багата-стадійність фазоутворення в процесі формування покриттів на алюмінієвих сплавах в лужно-силікатному електроліті і анодно-катодному режимі мікроплазмового оксидування. Показано вплив кристаллохімічних характеристик катіонів оброблюваного сплаву і катіонів, що входять до складу електроліту, на процес перетворення  $\gamma\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$

Ключові слова: структурна інженерія, мікроплазмова обробка, анодно-катодний режим, фазовий склад

Приведены исследования по влиянию режимов микроплазменного оксидирования в электролитах с активизирующими добавками на фазово-структурное состояние формируемых на основе алюминия покрытий. Обнаружена многостадийность фазообразования в процессе формирования покрытий на алюминиевых сплавах в щелочно-силикатном электролите и анодно-катодном режиме микроплазменного оксидирования. Показано влияние кристаллохимических характеристик катионов обрабатываемого сплава и катионов, входящих в состав электролита, на процесс превращения  $\gamma\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$

Ключевые слова: структурная инженерия, микроплазменная обработка, анодно-катодный режим, фазовый состав

## 1. Introduction

Modification of surface layers makes it possible to use the properties of base materials and modified layers most rationally as well as to spare expensive and rare metals and alloys.

Methods of modifying the surface are divided into two groups – modifications with and without an increase of thickness [1]. This helps achieve the most effective use of a combination of properties of the base material and of the modified surface layer. Therefore, surface modification procedures can be easily integrated into the technological process of producing or repairing products [2].

Currently, a wide range of technological processes is used to modify the surfaces of materials and parts made of

them in order to improve their performance characteristics. Among them, there are various options for thermal processing [3], chemical-thermal treatment [4], electrochemical technology [1], coatings with electron beam [5], magnetron [6], vacuum arc [7] and other methods. These types of effect produced on materials have technological and instrumental differences, but at the same time they have much in common, which is determined by the final structural state of the material. In general, this directed modification of the structure of the surface and near-surface layers has received a name – structural surface engineering [8]. The possibilities of structural surface engineering in recent years have significantly expanded as a result of using plasma technologies with a high degree of disequilibrium in the process of material formation. This made it possible to obtain new phase states [9],

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# THE INFLUENCE OF THE CONDITIONS OF MICROPLASMA PROCESSING (MICROARC OXIDATION IN ANODE-CATHODE REGIME) OF ALUMINUM ALLOYS ON THEIR PHASE COMPOSITION

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to specify the necessary structural stress state [10], and also to achieve high performance properties by creating multi-period structures [11] or special mutually-oriented multiphase materials [12].

In recent years, there has been a steady tendency towards tightened operating conditions, increased aggressiveness of the technological means used and, as a result, a general increase in requirements for structural materials [13]. This became the determining factor of intensive (large-scale) development of new environmentally clean surface modification technologies for highly effective and reliable protection and hardening of metal products. The development of such highly effective technologies for surface modification and control of surface properties on the basis of structural engineering results is today one of the most urgent tasks of modern science and technology.

## 2. Literature review and problem statement

The non-equilibrium plasma processes of surface modification include the technology of microplasma oxidation, which has been actively developed in recent years. Low-temperature microplasma treatment (also known as microarc oxidation [14], microplasma oxidation [15] or anode-spark electrolysis [16]) is an electrochemical method based on the use of a spark discharge phenomenon. The process of structural surface engineering in microarc oxidation consists in forming a protective ceramic coating on the surface of valve metals (Al, Ti, Mg, Zr, Nb, etc.) and their alloys [17]. It is noteworthy that valve metals are those whose oxide films (formed electrochemically) have unipolar or asymmetric conductivity in the metal-oxide-electrolyte (MOE) system [18]. In this case, the positive potential of the metal (on which the anodic oxide film is formed) corresponds to the locking or reverse direction, i. e., the system works analogously to a semiconductor valve.

A characteristic feature of microarc oxidation (MAO) is the appearance of plasma discharges in the dielectric breakdown in a highly passivated layer performed on the anode surface [19]. The electrolytes used are alkaline [20], so they are environmentally safer than chromium or sulfuric acid, which are commonly used in the anodizing process [21]. Microarc oxidation allows obtaining multifunctional ceramic-like modified layers with a wide range of properties, including wear-resistant [22], corrosion-resistant [23], heat-resistant [24], electrical insulating, and decorative [25].

A distinctive feature of microarc oxidation is the participation in the process of modifying surface microdischarges (Fig. 1), which have a very significant effect on phase and structure formation. As a result, the composition and structure of the resulting oxide layers are nonuniform [26], and their properties are significantly higher compared with conventional anodizing [27].

Thus, MAO simultaneously contains features of two different groups of methods for modifying such as coating deposition (i. e., incremental thickness modification) and changes in the state, structure and properties of surface and near-surface layers (i. e., without increment of thickness).

As a rule, the phase composition of coatings varies throughout the thickness of the MAO layer [28]. With MAO of aluminum-based alloys, it has been found that high-temperature modifications of aluminum oxide are in its internal layers. Towards the outer surface of the modified

layer, the number of low-temperature modifications of aluminum increases. It is assumed that the latter is related to the conditions of electrolysis at the initial stages of anodic spark treatment, since the temperature of the sections of the metal adjacent to the discharge craters is only 400–500 °C [29].



Fig. 1. The type of microdischarges in microarc oxidation of aluminum surface

The highest physicochemical properties and hardness of about 24 GPa are observed in the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase. The  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase has a hardness of about 14 GPa, and mullite has about 10 GPa. In this regard, the production of coatings with high hardness in most cases involves the need to ensure a high percentage of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> (corundum) in the coating.

Tests have shown that using additives in the electrolyte makes it is possible to lower the temperature of completing the conversion of low-temperature modifications of aluminum oxides to a stable  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase. However, the data published to date do not give complete and clear representations, which is explained by the small volume of the accumulated factual material. This primarily concerns data on the nature of the effect of various additives on the mechanism and rate of  $\gamma \rightarrow \alpha$  alumina transformations.

Since MAO is a relatively new method of plasma surface treatment, an understanding of the processes underlying it is still in its initial stage. This emphasizes the great need for obtaining and processing systematized empirical data on the effect of the parameters of the MAO process on the structural state and properties of the materials being processed (structural engineering).

## 3. The aim and objectives of the study

The aim of this work was to study the effect of additives in the electrolyte composition during microplasma treatment on the process of phase transformation of aluminum oxide. The additives used were cations of the processed alloy and cations that were part of the electrolyte.

To achieve this aim, the following objectives were accomplished:

- to study the stages of phase formation in the process of forming coatings on aluminum alloys in the alkali-silicate electrolyte and the anode-cathode mode of MAO;
- to determine the influence of adding liquid glass in the microarc oxidation process on the phase formation process and the efficiency of microplasma oxidation;
- to determine the nature of the effect of the additives on the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>  $\rightarrow$   $\alpha$ -Al<sub>2</sub>O<sub>3</sub> conversion process and their efficien-

cy for optimizing the technological regimes of microplasma treatment of various types of aluminum alloys.

**4. Materials and methods for obtaining and studying the coatings obtainable with MAO treatment**

Microplasma oxidation was applied to the following:

- the high purity aluminum A99 as well as;
- deformable aluminum alloys (D16, which is a composition of the main elements such as Al (90.9–94.7 %), Cu (3.8–4.9 %), and Mg (1.2–1.8 %); AMg6, which is a composition of the main elements such as Al (91.1–93.7 %), Mg (5.8–6.8 %), and Mn (0.5–0.8 %); as well as AMc, which is a composition of the main elements such as Al (96.35–99 %), Mg (1.0–1.5 %), and Fe (up to 0.7 %));
- the casting alloy (AL9, which is a composition of the main elements such as Al (89.6–93.8 %), Si (6–8 %), and Fe (up to 1.5 %)).

The process was carried out in an alkaline-silicate electrolyte of various compositions in a condenser-type plant [1] in the anode-cathode regime. The current density was 5–50 A/dm<sup>2</sup>.

The phase composition of MAO coatings was determined based on the results of the X-ray phase analysis. The tests were carried out on the DRON-3 unit (Burevesnik, Russia) under monochromatized K<sub>α</sub>-Cu radiation. The data were recorded in a pointwise mode with a step of 2θ=0.1°. The results obtained in the study refer to the main coating layer (the technological layer was removed by stripping by abrasive paper).

For the quantitative phase analysis, the reference mixture method was used [30].

**5. The results of studying the influence of the MAO treatment modes on the phase composition**

Practical implementation of the microplasma process always requires careful coordination of the metal-electrolyte pair. One of the simplest and most widely used electrolytes for treating alloys based on Al is a diluted (2–8 g/l) KOH solution, which makes it possible to obtain superhard high-quality coatings [31].

However, tests have shown that when coatings are formed in an alkaline electrolyte (2 g/l KOH) on aluminum and its alloys, it is not possible to obtain a coating with a thickness of more than 20 μm. This is due to increased etching of the formed coating. The coating consists of the γ-Al<sub>2</sub>O<sub>3</sub> phase.

To increase the thickness of the modified layer during microplasma treatment, technically liquid glass (Na<sub>2</sub>SiO<sub>3</sub>) is added to the electrolyte.

Our results show that although the addition of Na<sub>2</sub>SiO<sub>3</sub> to the electrolyte provides a significant increase in the thickness of the coating formed, it is accompanied by the appearance of a phase with a low hardness (mullite) – Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub> (Fig. 2).

The formation of mullite is determined by the fact that when liquid glass is diluted with water, hydrolysis occurs by the following reaction:

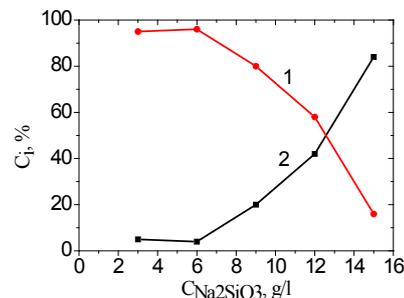
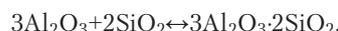


Fig. 2. The influence of the content of liquid glass on the phase composition of C<sub>i</sub> (1 – C<sub>γ-Al<sub>2</sub>O<sub>3</sub></sub>, 2 – C<sub>3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub></sub> (mullite)) of coatings on aluminum A99 (KOH – 1 g/l, j=20 A/dm<sup>2</sup>, τ=1 h, and thickness of about 40 μm)

The interaction of γ-Al<sub>2</sub>O<sub>3</sub> with SiO<sub>2</sub> leads to the appearance of mullite:



Thus, in the case of treating aluminum alloy A99, the presence of liquid glass (Na<sub>2</sub>SiO<sub>3</sub>) in the electrolyte promotes the formation of mullite, which is a negative factor in terms of high mechanical properties of the coating.

The kinetics of phase formation on A99 in an alkali-silicate electrolyte is shown in Fig. 3 (the electrolyte: KOH – 2 g/l, Na<sub>2</sub>SiO<sub>3</sub> – 12 g/l, and current j=10 A/dm<sup>2</sup>).

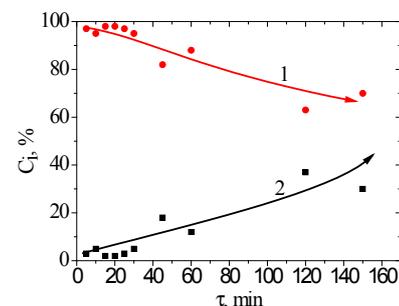


Fig. 3. The kinetics of phase formation (1 – C<sub>γ-Al<sub>2</sub>O<sub>3</sub></sub>, 2 – C<sub>3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub></sub> (mullite)) in coatings on A99 (j=10 A/dm<sup>2</sup>)

The absence of the α-Al<sub>2</sub>O<sub>3</sub> phase in the coating can be explained by the low power of microdischarges, at which the temperature of the polymorphous transformation γ→α is not ensured.

The phase composition during the formation of coatings on the alloys D16 and AL9 is shown in Fig. 4. It is noteworthy that for the AMc alloy at a depth of 100 μm, the composition varies from 30 % of C<sub>γ-Al<sub>2</sub>O<sub>3</sub></sub> plus 70 % of C<sub>α-Al<sub>2</sub>O<sub>3</sub></sub> (with an electrolyte KOH being 2 g/l), up to 70 % of C<sub>γ-Al<sub>2</sub>O<sub>3</sub></sub> plus 30 % of C<sub>α-Al<sub>2</sub>O<sub>3</sub></sub> (with 2 g/l of the electrolyte KOH plus 6 g/l of Na<sub>2</sub>SiO<sub>3</sub>). The coating of the AMg6 alloy consists only of the γ-Al<sub>2</sub>O<sub>3</sub> phase.

The main phases of MAO of the coatings on the aluminum alloys are aluminum oxides γ-Al<sub>2</sub>O<sub>3</sub> and α-Al<sub>2</sub>O<sub>3</sub>. The analysis of the properties of the coatings proves the fact that the coatings on the different aluminum alloys with the same phase composition have different properties, for example, hardness. One of the reasons for this difference may be the features of the crystal structure of the phases that make up the coatings. In this connection, a precise measurement of

the lattice parameter of the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase was carried out. X-ray diffractometry was performed on a DRON-3 unit with a pointwise registration of diffraction profiles. The lattice period of  $\gamma$ - $\text{Al}_2\text{O}_3$  was determined in  $\text{K}\alpha$ -Fe radiation along the line (800) located at an angle of  $\theta \approx 78.5^\circ$ . In order to take into account the influence of macrostresses on the lattice period, inclined surveys were carried out.

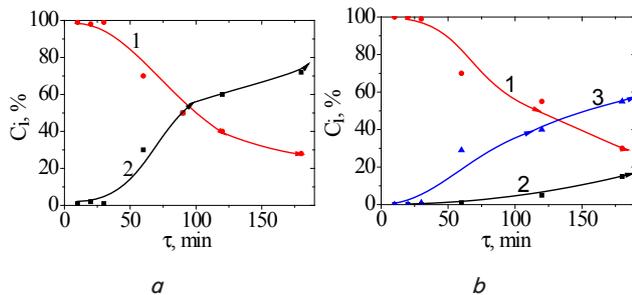


Fig. 4. The phase composition (1 –  $C_{\gamma\text{-Al}_2\text{O}_3}$ , 2 –  $C_{\alpha\text{-Al}_2\text{O}_3}$  and 3 –  $C_{3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2}$ ) of the coatings on the alloys: a – D16 (the electrolyte:  $\text{KOH} - 2 \text{ g/l}$ ,  $\text{Na}_2\text{SiO}_3 - 12 \text{ g/l}$ , and current  $j = 20 \text{ A/dm}^2$ ); b – AL9 (the electrolyte:  $\text{KOH} - 2 \text{ g/l}$ ,  $\text{Na}_2\text{SiO}_3 - 12 \text{ g/l}$ , and current  $j = 20 \text{ A/dm}^2$ )

The data of the X-ray diffractometry tests have revealed the following patterns:

- for all the tested samples, strengthened by MAO, it is typical to have pronounced diffraction peaks. This indicates the crystalline structure of the hardened layer (unlike the anodically oxidized coatings formed in conventional oxidation consisting of X-ray amorphous aluminum);
- a pronounced texture of the crystallites of oxide phases was not detected (this indicates a chaotic orientation of the crystallites of the hardened layer formed in various regimes of MAO).

Precision studies have shown that the lattice period of the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase varies widely (Fig. 5). At the initial moment of forming the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase, its lattice period is 0.79 nm, which corresponds to the tabulated value.

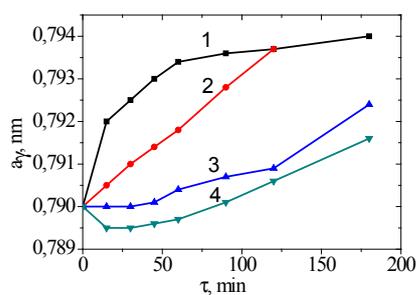


Fig. 5. The dependence of the lattice period of the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase on the time of the MAO process in the coatings of the different alloys: 1 – AMg6, 2 – D16, 3 – A99, and 4 – AL9

With increasing the processing time, as a rule, an increase in the lattice period during the formation of the coating is observed. Only in the case of aluminum A99 and alloy AL9 in the initial period of oxidation there is a slight decrease in the lattice period. With increasing the processing time, the period increases.

The obtained results indicate that during the oxidation process, the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase is alloyed with the base components and the electrolyte components to form solid substitu-

tional solutions. The change in the lattice period in this case will be determined, on the one hand, by the difference in the ionic radii of atoms in the lattice and, on the other hand, by the difference in valence. After comparing the ionic radius of Al with the ionic radius of the additive cations, it can be concluded that all cations of the base and the electrolyte, with the exception of Si, should lead to an increase in the lattice period of the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase. The observed deviation from the general trend is probably due to the absence of isovalence of the cations.

## 6. Discussion of the results of studying the phase composition and structure of the MAO of the modified surface

Despite the fact that theoretical ideas about the nature of the phenomena on which the MAO technology is based are far from perfect, highly effective ways of obtaining a variety of coatings have been developed empirically.

Determination of the causes of forming and the conditions (possibilities) for controlling the phase composition and the structural state is the main task of structural engineering of the surface of materials subjected to MAO treatment.

The composition of the electrolytes, along with the substrate material, the regime and time of treatment, is the determining factor of the microarc oxidation process, affecting the composition, structure and properties of the coatings obtained. Tests have shown that the composition of the electrolyte has a significant influence on the initial stages of the process during which strong passivating layers are formed on the metallic surface. These layers determine the possibility of spark explosions of sufficient intensity and, thus, the implementation of the MAO process.

With the MAO treatment, the surface layer of the processed aluminum alloy is converted to a coating consisting of aluminum oxides, whereby the coating has very high bond strength with the substrate, which corresponds to the strength level of the alloy.

The phase composition of coatings formed on different alloys is different both qualitatively and quantitatively. For example, the strengthened layers obtained on AMg alloys are predominantly  $\gamma$ - $\text{Al}_2\text{O}_3$ , and on the A99 and D16 alloys, they are  $\alpha$ - $\text{Al}_2\text{O}_3$ . The reinforced layer formed on the casting alloy consists of the phases of  $\gamma$ - $\text{Al}_2\text{O}_3$  and mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ).

The phase composition of the thickness of the hardened layer is also not even. There is a change in the hardness throughout the thickness of the coating, regardless of the chemical composition of the oxidized alloy and the MAO regimes, which is due to a change in the phase composition of the hardened layer.

Thus, the use of alkali-silicate electrolytes allows a significant increase in the time of the MAO process (the microarc discharge mode is implemented within 3–5 hours). Liquid glass increases the degree of reflow of the coating and makes it possible to obtain high quality coatings with a thickness of 300–400  $\mu\text{m}$ . In this case, the phase composition of the coating is determined both by the composition of the electrolyte and by the composition of the alloy being processed (Fig. 2–4).

The mechanism of forming the phase composition should be associated with stabilization and destabilization of the  $\gamma$ - $\text{Al}_2\text{O}_3$  phase.

The coatings obtained by microplasma oxidation have a unique complex of properties, including high wear resistance, antifriction, high hardness, heat resistance, and electrical insulating properties. This has been confirmed by the pilot-industrial tests on a number of products from aluminum alloys: friction pairs, end seals of engine systems, impeller pumps, connecting rods, heating elements, etc. (Fig. 6).



Fig. 6. Details made of aluminum alloys after their pilot-industrial processing by microplasma oxidation

It is planned to develop research further in the direction of optimizing the phase composition to improve the performance characteristics of machine elements and aluminum alloy assemblies operating under the influence of aggressive media.

## 7. Conclusion

1. The multi-stage phase establishment (with the development of intermediate phases such as  $\gamma\text{-Al}_2\text{O}_3$  and  $3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ) was revealed during the formation of coatings on aluminum alloys in the alkali-silicate electrolyte and anode-cathode MAO regime.

2. It has been determined that the use of a liquid glass additive in the process of microarc oxidation leads to an increase in the thickness of the modified layer more than 10 times (from 20  $\mu\text{m}$  without up to 400  $\mu\text{m}$  with the addition of liquid glass). However, the functional properties of the modified layer can be reduced due to the formation of a phase with a relatively low hardness, which is mullite.

3. It has been established that the degree of the effect of additives on the  $\gamma\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$  transformation is determined by their crystal-chemical characteristics (the charge and the ionic radius). Because of this, for each type of alloys, preliminary phase-structural studies are required to optimize the technological regimes of microplasma treatment.

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