STUDY OF STRUCTURE AND MICROHARDNESS OF PROTECTIVE OXIDE-CERAMIC COATINGS OBTAINED BY MICRO-ARC OXIDATION ON DEPOSITED SURFACES

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Abstract:
The paper is devoted to studying the structure and microhardness of protective oxide-ceramic coatings obtained by micro-arc oxidation on the deposited surfaces of parts. Experimental researches were carried out with the use of the commonly used perfected methods and up-to-date measuring instruments and equipment. AlSi7Mg aluminum alloy was used to conduct the research and was deposited with various wires. A welding machine UDG-180 was used to surface the samples. Hardened layers were formed on the deposited surfaces in the developed electrolyte in an installation for micro-arc oxidation, working in the anode-cathode mode. It has been established that the hardened layer formed by micro-arc oxidation is characterized by high microhardness. The increased microhardness of coatings is explained by the presence in their structure of high-temperature solid-phase modification α-Al_2O_3. Introducing electrolytes of composite materials 1.5 to 1.8 times allows for shortening the oxidation duration. Coatings obtained by micro-arc oxidation are heterogeneous in thickness and physical properties. Considering mechanical processing, the thickness of the hardened layer formed on the deposited surface of the part must be at least 90-120 μm.

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1. INTRODUCTION

Among the known methods of obtaining composite nanostructured coatings on machine parts, micro-arc oxidation (MAO) has exceptional prospects. The coatings obtained by MAO are characterized by high physical and mechanical properties [1-5].

The discovery of the phenomenon of galvanoluminescence during electrolysis should be considered the starting point of research in the field of micro-arc oxidation [6]. The sparking effect itself was described as follows. At a specific voltage, there is a sharp heating of a thin pore channel in the oxide layer, and the current there is interrupted due to the formation of a vapor-gas bubble due to evaporation and electrolysis of the electrolyte. An electrical breakdown of the bubble with gas discharge ignition occurs at further voltage increase, accompanied by a sharp thermal increase

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in its volume and, consequently, in the interelectrode distance in the discharge channel. At some moment, the voltage to maintain it becomes insufficient, and the discharge goes out. As a result, the bubble sharply cools and shrinks, accompanied by a crackling characteristic of spark discharge anodization. Galvanoluminescence, valve effect, spark discharge on the anode, and electrical breakdown continue to be investigated even nowadays [7-9].

A further stage in the development of research in this direction was the work of Sofronov et al. [10] on the practical use of reactions occurring in the anodic spark to synthesize complex oxide coatings from substrate and electrolyte components, published in the 50-60s of the last century. At the same time, the first patents were obtained, in particular, for the process of obtaining silicate coatings on aluminum from weakly alkaline solutions [11-12].

The beginning of the modern stage of research in the field of MAO and its practical application can be considered the 70s of the twentieth century, when a significant number of publications and patents appeared by researchers of different schools, the number of which continues to grow today. Among them, there are the works of Brown et al. [13] on anodic spark deposition, Nikolaev et al. [14] on micro plasma treatment, and Chernenko et al. [15] on anode spark electrolysis.

A great burst of publications on MAO has been observed in the last twenty years, indicating the method's active development – the work aimed to study the mechanisms of the MAO and refine the process technology. The contemporaries pay special attention to the development of current sources for plasma electrolytic oxidation [16-19]. The works of scientists who work in the direction of the formation of functional coatings in electrolytes with polyphosphate complexes of metals, with iso- and heteropolyoxoanions, in electrolyte-suspensions, electrolyte-emulsions are of interest [20-22].

In the work of Suminov et al. [23], the process of micro-arc oxidation of valve metals is systematically described.

In the paper [24], correlations were established between the discharge current, the light emission of the discharge channel, and the size of the gas bubbles formed during MAO. This, to a certain extent, makes it possible to explain the mechanism of micro plasma discharge formation and substantiate the stages of oxide-ceramic coating formation.

Rogovab et al. [25] evaluated the role of cathode current in aluminum micro-arc oxidation. Researchers have noted insufficient clarity of the mechanism of the flow of reactions at MAO caused by the cathode current. A quantitative approach to the numerical assessment of the changes occurring in the oxide layer under negative polarization is proposed.

In the paper [26], the kinetics of coating formation during MAO are by existing modern models of discharge phenomena.

Publications on the possibility of micro-arc oxidation of zinc-based alloys have appeared. For example, Fattah-alhosseini et al. [27] used silicate, aluminate, and borate electrolytes to obtain oxide-ceramic coatings on zinc.

The work of Chinese scientists on the effect of electrolyte components during MAO on the microstructure and mechanism of coating formation on aluminum alloy 1060 using silicate, phosphate, and combined electrolytes is very interesting [28].

The paper [29] studied the characteristics of corrosion-resistant coatings obtained by MAO on low-carbon steel in aluminate electrolyte. It was found that the obtained coatings contain polycrystalline aluminum oxide phases (Al_2O_3-α and Al_2O_3-γ), are porous and have a thickness of 3 to 11 μm. The anticorrosive properties of the coatings were limited by through and volumetric porosity.

The paper [30] presents research on the micro-arc oxidation of magnesium alloys. The studies found that the addition of potassium fluoride (KF) to the electrolyte causes significant changes in the structure and properties of the oxide layers. At the same time, fluorine was detected as an amorphous phase near the metal substrate. The presence of fluorine components in the electrolyte contributed to an increase in the thickness of the oxide layers. The obtained coatings significantly improved the corrosion resistance of magnesium alloys.

A brief analysis of literary sources shows that the process of formation of composite protective oxide-ceramic nanostructured coatings on machine parts is relevant and has deep prospects for its development.

The aim of this work was to investigate the structure and microhardness of oxide-ceramic coatings obtained by micro-arc oxidation on deposited surfaces.

2. MATERIALS AND METHODS

Methods of experiments included deposition of the surfaces of the samples and their subsequent micro-arc treatment in a combined electrolyte based on boric acid with additives of finely dispersed metal oxide powders.

The complex studies of coating characteristics included X-ray diffraction analysis and evaluation of the microhardness of the formed coatings along the thickness of the hardened layer.

The research was carried out based on the Department of Reliability and Repair of Machines at the N.V. Parakhin Orel State Agrarian University (Russia).

To conduct research, aluminum alloy AlSi7Mg, was used, deposited with welding wire OK Autrod 18.01. A general view of the samples for research is shown in Fig. 1.

Fig. 1. Samples for research

A welding installation was used to deposit the samples UDG-180 (Fig. 2).

Fig. 2. Installation UDG-180 and torch GDN-201

The hardened deposited surfaces were performed using a micro-arc oxidation unit (Fig. 3) operating in the anode-cathode mode.

The electrolyte was prepared by mixing the components in distilled water, qualification AR (analytic grade). Boric acid was used as an inhibitor that increases the efficiency and durability of the electrolyte, as well as the mechanical properties of the coatings.

a)

b)

Fig. 3. Micro-arc oxidation installation: a) Power unit and b) electrolytic bath

The following electrolyte composition, g/l, was used in the studies:

- *KOH* 4-6,
- H_3BO_3 20-30,
- composite material 20-30.

Finely dispersed additives of aluminum, titanium, iron, chromium, and silicon oxide powders were used as composite materials.

X-ray studies of the coatings were performed on a general-purpose diffractometer DRON-3M (Fig. 4). To determine the phases of metal oxides, copper Cu Kα-radiation with a wavelength λ = 0.154178 nm, Ni-filter was chosen. The scanning angles were 2θ = 5-60°, which allowed us to cover almost all diffraction maxima.

Fig. 4. DRON-3M diffractometer

The ICDD 1997 JCPDS-International Centre for Diffraction Data computer database was used to identify the phases formed in the coating during MAO.

The microhardness of the hardened layers obtained on the deposited surfaces was determined using the PMT-3M microhardness tester according to standard procedures on cross-sectional sections of the samples at an indenter load of 0.981 H.

3. RESULTS AND DISCUSSION

Experiments have shown that the highest quality coatings are formed in silicate-alkali, oxalic acid, and boron-acid electrolytes. The optimum electrolyte is based on boric acid for the developed method of hardening deposited surfaces, as it provides the most complex coatings.

Fig. 5 shows the results of studies of the thickness of the working layer and microhardness of coatings formed in electrolytes with the addition of metal oxide powders. The samples made of AlSi7Mg alloy with a deposited layer of OK Autrod 18.01 were subjected to hardening. The treatment duration was 90 min.

X-ray diagrams taken on the diffractometer DRON-3 confirmed the presence of substances in the electrolytes in the structure of the formed coatings (Fig. 5, Table 1). Fig. 6 also shows the designations of identified *α*- and *γ*- Al_2O_3 phases.

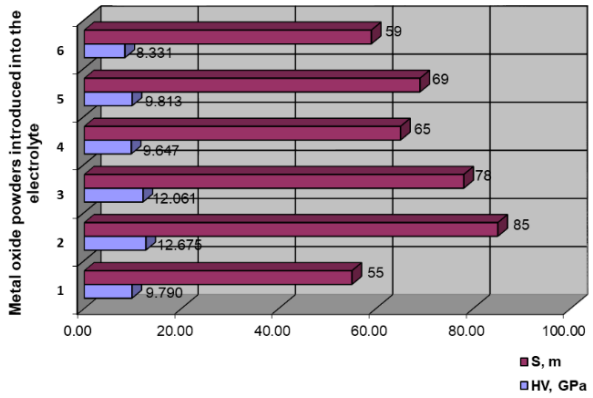

Fig. 5. Comparative data on the microhardness (HV) and thickness of coatings (S) formed in electrolytes with the addition of metal oxide powders:
1 - without adding powders; 2 - Al_2O_3 (20 g/l);
3 - TiO_2 (20 g/l); 4 - Cr_2O_3 (20 g/l); 5 - Fe_2O_3 (20 g/l);
6 - SiO_2 (20 g/l)

Studies have shown that the formation of composite oxide-ceramic coatings with the addition of metal oxide powders leads to an increase in microhardness compared to the standard boric acid-based electrolyte (Fig. 6) and significantly accelerates the process of their formation. At the same time, the coatings' homogeneity largely depends on the dispersity of the powder used and the stability of its maintenance in the electrolyte in a suspended state.

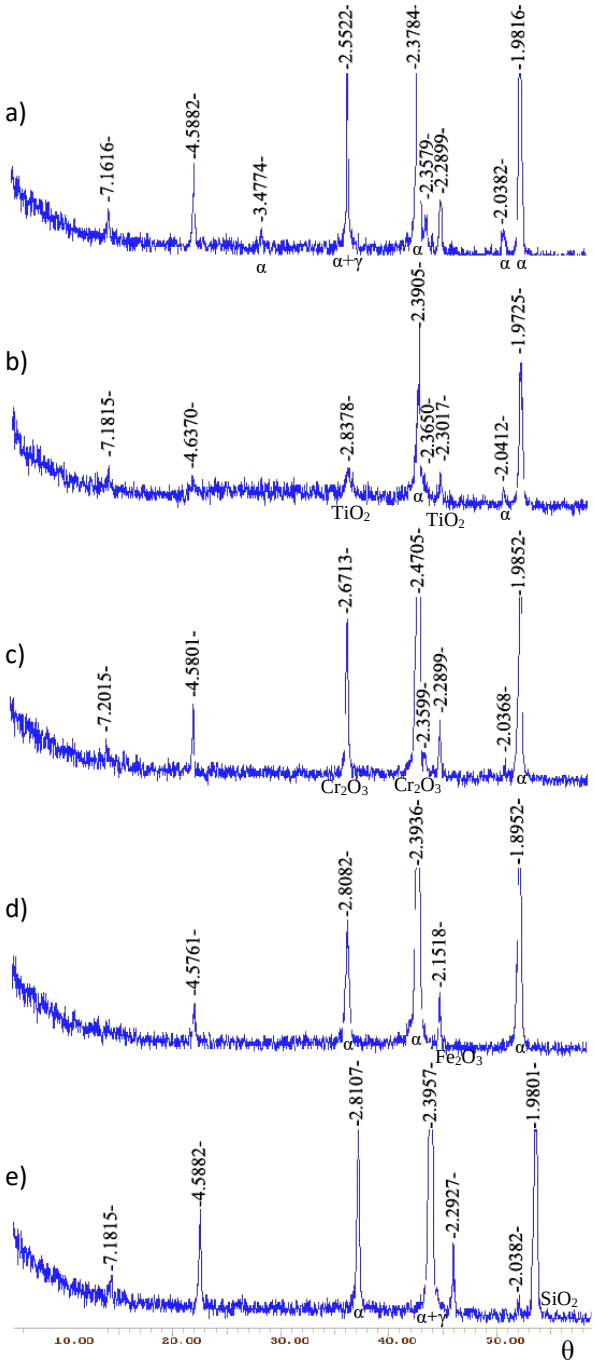

Fig. 6. X-ray diagrams of samples with coatings formed in electrolytes with added oxides:
a) Al_2O_3 (20 g/l); b) TiO_2 (20 g/l); c) Cr_2O_3 (20 g/l);
d) Fe_2O_3 (20 g/l); e) SiO_2 (20 g/l)

Table 1. Results of X-ray studies of composite oxide-ceramic coatings

Sample number	Electrolyte composition	Characteristic X-ray spacing for materials introduced into the electrolyte				
		α-Al_2O_3	**TiO_2**	**Cr_2O_3**	**Fe_2O_3**	**SiO_2**
1	H_3BO_3 (20 g/l)+KOH (5 g/l)+Al_2O_3 (20 g/l)	3.47 2.55 2.37 1.99				
2	H_3BO_3 (20 g/l)+KOH (5 g/l)+TiO_2 (20 g/l)		2.83 2.30			
3	H_3BO_3 (20 g/l)+KOH (5 g/l)+Cr_2O_3 (20 g/l)			2.67 2.47		
4	H_3BO_3 (20 g/l)+KOH (5 g/l)+Fe_2O_3 (20 g/l)				2.15	
5	H_3BO_3 (20 g/l)+KOH (5 g/l)+SiO_2 (20 g/l)					1.98

Of the group of powder materials introduced into the electrolyte, aluminum and titanium oxide powders give the best results. In order to study the properties of composite oxide-ceramic coatings in more detail, an experiment was conducted to reveal the character of microhardness change along the coating thickness.

In the works of many authors [31-34], it is noted that the coating formed by the MAO method can be conditionally divided into several layers. The top coating layer consists mainly of mullite (compounds of aluminum oxide with electrolyte elements), the wear-resistant working layer – of alumina and corundum (γ- and α-Al_2O_3), and the α-Al_2O_3 phase prevails at the border with the base. Differences in these layers' properties are due to their formation's peculiarities [35-38].

Table 2 shows the results of measuring the microhardness by the thickness of the coatings formed by MAO on samples made of AlSi7Mg alloy deposited with OK Autrod 18.01. Measurements were taken from the base (aluminum alloy) through the coating to the surface layer in increments of 10-20±5 μm.

Table 2. Results of microhardness measurements by thickness of oxide-ceramic coatings

Indentation point	Microhardness $HV_{0,1}$, GPa by thickness S, μm (HV/S), (averages of 10 measurements)					
	Electrolytes with metal oxide additives					
	H_3BO_3+KOH	H_3BO_3+KOH+Al_2O_3	H_3BO_3+KOH+TiO_2	H_3BO_3+KOH+Cr_2O_3	H_3BO_3+KOH+FeO_2	H_3BO_3+KOH+SiO_2
Base metal (5-20 μm)	0.995/-20	1.2/-20	1.676/-20	1.2/-20	0.874/-20	1.201/-20
	1.127/-10	1.18/-10	1.757/-10	1.48/-10	1.857/-10	1.631/-10
Metal-coating boundary (3-5 μm)	4.335/5	3.124/5	2.86/5	3.255/5	4.155/5	3.913/5
Basic coating layer (60-100 μm)	12.052/15	15.12/15	15.57/15	14.12/15	11.44/15	11.50/15
	14.66/30	16.76/25	14.07/25	12.76/30	13.97/30	9.78/30
	8.325/40	14.37/35	15.32/35	9.37/40	9.73/40	8.10/40
	7.071/50	13.42/45	14.01/45	7.72/50	9.52/50	6.78/50
	6.825/60	12.24/55	8.89/55	8.04/60	7.38/60	5.50/60
		10.68/65	9.08/65	5.87/70	6.84/70	
		10.76/75	7.50/75			
		7.87/85				
Average microhardness value, GPa	9.79	12.65	12.062	9.65	9.813	8.331
Thickness of the working layer, S working, μm	55	85	78	65	69	59
Surface coating layer (20-40 μm)	4.882/70	6.15/100	5.628/90	4.155/80	5.116/80	4.333/70
	3.735/85	4.628/120	4.833/100	3.555/90	4.04/90	3.453/80

The character of changes in the coatings microhardness formed in H_3BO_3-KOH type electrolyte for 90 minutes with aluminum oxide additives is shown in Fig. 7.

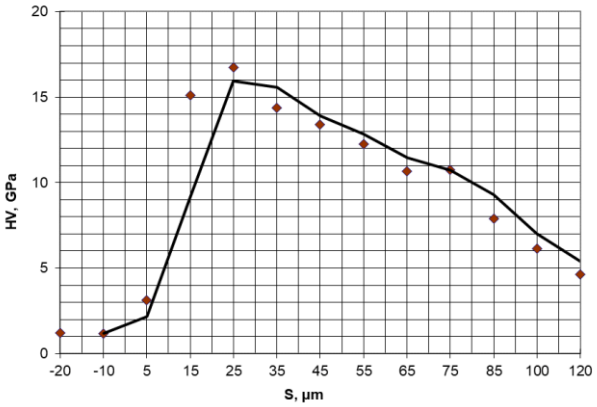

Fig. 7. Change of microhardness by thickness of the coating obtained in the electrolyte H_3BO_3+KOH+Al_2O_3

It was found that the heterogeneity and non-uniform structural distribution over the thickness of the coating lead to the fact that the microhardness changes in accordance with changes in the phase composition of the coating and, to a greater extent, depending on the distribution of the high-hard phase α-Al_2O_3. Indeed, if the ratio between α- и γ-Al_2O_3 in the surface layer does not exceed 0.5, then in the main coating layer, this ratio is already close to 1.0, and in the layer adjacent to the metal, it is 2.5. In the coating, especially in the boundary zone, phases related to the metal structure are distinguished. This explains the high bonding strength of the coating to the base and characterizes the presence of chemically bonded compounds between the base metal and the coating. It is evident that as the thickness of the fundamental (dense) coating layer increases due to changes in its electrophysical characteristics, the front of the maximum temperature of micro-arc discharges shifts deep into the oxide, i.e. their burning zone separates from the base metal surface. This is indirectly confirmed by the fact that there are peaks in the microhardness distribution along the thickness (maximums in the 15-50 µm zone from the base). This is where the area of maximum temperature is concentrated, and consequently, the maximum melting of the coating material occurs. Accordingly, the maximum content of the high-temperature phase is localized in this area - α-Al_2O_3.

4. CONCLUSION

The most important results of this research are:

1. The application of aluminosilicate and rutile increases the microhardness of the coatings with good homogeneity. The application of electrolytes with the addition of composite materials allows reducing the time of formation of MAO coatings by 1.5-1.8 times compared with “pure” electrolytes. The main criterion for obtaining quality coatings is ensuring that the dispersed particles are suspended.

2. Coatings obtained by the MAO method are heterogeneous in thickness and physical properties. Three zones can be distinguished in the coating: zone I - the transition area between the coating and the base material; zone II - the basic coating layer with the highest value of microhardness; zone III - the surface coating layer, characterized by relatively low values of microhardness.

3. Diffusion of elements in the molten substance proceeds more actively than in the solid, so the increase in the content of the high-temperature phase in the coating unambiguously indicates the area with the maximum temperature gradient. The indicated feature allows us to determine the most negligible coating thickness sufficient to ensure high wear resistance of the working joint. Taking into account the subsequent machining (grinding) and removal of the surface layer in the process of lapping, it should be at least 90-120 microns. Increasing the coating thickness over 200 microns is not rational in terms of energy consumption for its formation, as it requires significant time to harden the part.

Conflicts of Interest

The authors declare no conflict of interest.

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