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Metallurgy

Effect of micro-arc oxidation on the properties of aluminum alloy samples

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ABSTRACT
Currently, modern manufacturing industries impose special requirements on structural materials such as aluminum, titanium, and their alloys. Various methods are used to improve the physicomechanical and corrosion properties of these materials. One of the promising ways to modify the surface in order to give it multifunctional properties is the treatment of micro-arc oxidation. A distinctive feature of the process is the formation of the oxide coatings on valve metals because of exposure to micro-arc discharges. At the same time, coatings with unique properties are formed. However, the effect of the micro-arc oxidation process on the properties of the base material has been little studied. The purpose of this work is to study the effect of the micro-arc process, implemented in pulsed mode, on the properties of oxide layers, and the base material. Modification of the alloy surface was carried out in the anode mode, with small values of the duration of the anode current pulse. An alkaline electrolyte solution was used as the electrolyte. Studies of the microhardness of the oxide layer, as well as the metal layer from the interface – oxide layer /metal deep into the metal, have shown that micro-arc discharges affect not only the properties of the oxide layer but also structural changes in the thickness of the metal. It is shown that the formed oxide coating is characterized by high microhardness. The oxide coatings obtained at the duration of the anode current pulse of 100 μs – 200 μs are wear-resistant, the coatings do not collapse, and do not wear to the ground under the accepted test conditions.

Keywords*:* valve metals, plasma electrolytic oxidation, microplasma discharges, oxide coating, microhardness, transition layer.

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Introduction

Structural materials are widely used in many industries. Improvement of the physical and mechanical properties of materials can be carried out in various ways. The method of plasma spraying (PS) is used to create multifunctional coatings in various industries [[1], [2]]. With PS, the coating is formed by the impact of molten particles on the surface of the material. As a result, there are changes in the structure of the surface layers of the metal, and its mechanical characteristics [[3], [4]]. *However, a low adhesive strength of the coating is observed while using this method. Currently, the micro-arc oxidation (MAO) method is increasingly used to modify the surface of various alloys.* A distinctive feature of this process is the formation of many microplasma discharges on the surface of the processed material, which have a significant effect on the properties of the coating [[5], [6], [7]]. The oxide-containing coatings formed during MDO are characterized by high wear resistance [[8], [9]], corrosion resistance [[10], [11], [12], [13]], heat resistance [14]. Biologically active coatings obtained by this method are also well known [[15], [16], [17]]. When applying this method, close attention is paid to the effect of microplasma discharges on the formed oxide coating. At the same time, it should be noted that microplasma discharges can affect the processed material from the interface of the oxide layer-metal deep into this material. It is known that high temperatures of 6000 °C and higher can develop in the area of the base of the combustion of micro-discharges [[18], [19]].

The MAO method, which is carried out in pulse mode, is also energy-saving. At the same time, the properties of oxide layers in their physicomechanical and other characteristics are not inferior to the properties of coatings obtained under stationary conditions [[20], [21]]. This work aims to study the effect of the micro-arc process, implemented in pulsed mode, on the properties of oxide layers, the base material.

Experimental part

Flat samples made of aluminum alloy A0 were used for the research. The samples were processed in pulse mode. The duration of the anode current pulse was: 50 microseconds, 100 microseconds, 150 microseconds, and 200 microseconds. The frequency of the anode pulses was maintained at 50 Hz. The polarizing voltage was equal to 300 V; the current density was about 115 A/dm^2. Pretreatment of samples included the following operations: grinding, degreasing, and drying. Since the MAO process proceeds with the release of heat, it is necessary to ensure the cooling of the electrolyte solution. Various types of cooling are used for this purpose. In this work, during the research, the electrolyte in the bath was cooled by a water cooling jacket (Figure 1). Running cold tap water flowed through the cooling jacket.

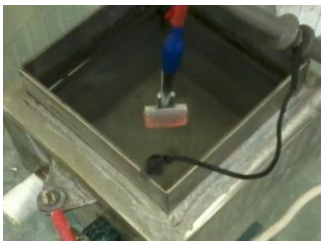


Figure 1 - Electrochemical bath with water cooling jacket

The bath body was used as the cathode, the anode served as the processed samples. Processing time 1200 sec. The electrolyte temperature was maintained within 20 °C – 25 °C. A solution of the composition was used as an electrolyte solution: sodium phosphoric acid 2- substituted, 12 aqueous (40 g/l), sodium tetraborate 10 aqueous (30 g/l), boric acid (22 g/l), ammonium fluoride (10 g/l). To prepare the electrolyte solution were used chemicals of the brand "chemically pure", and "pure for analysis". Electrolytes were prepared in distilled water.

The thickness of the oxide coatings was measured using a NOVOTEST TP-1 thickness gauge with an NF2 sensor. This work shows the arithmetic mean value of the coating thickness based on the results of seven measurements on both sides of the sample.

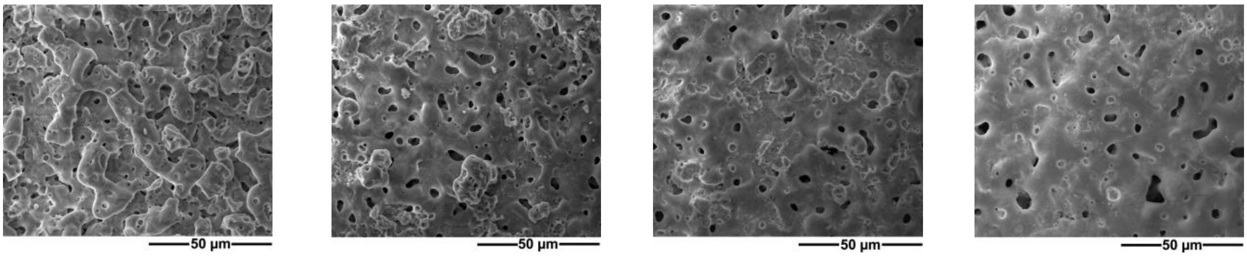
The microhardness of the oxide layers and the transition layer deep into the metal was studied on a NanoHardness Tester on pre-prepared grinds. When testing samples with an oxide coating, indentation of the indenter with a diamond tip occurred at a load of 20.0 mN. After the load reached the maximum value, the indenter began to unload. That is, the load acting on it was gradually reduced to zero, and it returned to its original position. The experimental data were processed based on the measurement results of at least 3 prints obtained under the same experimental conditions.

To evaluate the tribological characteristics were obtained the friction quotient curves on the THT-S-AX0000 tribometer. Conditions for tribological tests: load 1.0 N; number of revolutions 1000; linear velocity 2.5 cm/s; temperature 25°C; air humidity 50%.

Morphological studies were carried out on a Quanta 200 3D scanning electron microscope. The porosity of the coatings was determined by the number of pores per surface unit, and the planimetry method was used to evaluate their shapes by processing micrographs of the surface of oxide coatings [22].

Results and discussion

The structure of the oxide layers formed at different durations of the anode current pulse is porous (Figure 2). The pores are predominantly rounded and oval in shape. The thickness of the oxide layers is 8.0 microns, 11.0 microns, 20.0 microns, 27.0 microns, obtained respectively at the duration of the anode current pulse of 50.0 microseconds, 100.0 microseconds, 150.0 microseconds, 200.0 microseconds. The coatings are dense, evenly distributed in thickness over the entire surface of the sample.



50.0 microseconds 100.0 microseconds 150.0 microseconds 200.0 microseconds

Figure 2 – Microstructure of the surface layer of coatings at different values of the duration of the anode current pulse

Table 1 - Microhardness measurement data for samples processed at different durations of the anode current pulse

Depth along the transverse section, microns	Microhardness of the oxide layer, MPa	Depth along the transverse section, microns	Microhardness of metal, MPa
1. MAO at the duration of the anode current pulse of 50 microseconds (the thickness of the oxide layer is 8.0 microns)			
0	1523.0	15	1899.0
5	1622.0	20	1997.0
10	1032.0	25	1936.0
		30	1636.0
		40	1372.0
2. MAO at the duration of the anode current pulse of 100 microseconds (the thickness of the oxide layer is 11.0 microns)			
0	1894.0	15	1467.0
5	1889.0	20	1470.0
10	1887.0	25	1046.0
		30	964.0
		40	884.0
3. MAO at the duration of the anode current pulse of 150 microseconds (the thickness of the oxide layer is 20.0 microns)			
0	3776.0	25	1997.0
5	3808.0	30	1689.0
10	4891.0	40	1230.0
15	5330.0	55	978.0
20	2863.0		
4. MAO at the duration of the anode current pulse of 200 microseconds (the thickness of the oxide layer is 27.0 microns)			
0	33344.0	30	1914.0
5	30579.0	40	1493.0
10	29661.0	55	1167.0
15	33744.0	60	1004.0

The coatings are characterized by a surface porosity of 6% to 14%. Pores are known to form because of exposure to microarc discharges [23]. Figure 2 shows that as the result of the thickness of the coating increases with an increase in the duration of the anode pulse, some pores become overgrown, and the coating becomes more uniform. This trend is in good agreement with the earlier research conducted by the references [[24], [25]]. When studying the microhardness of the transverse section of the treated samples along the thickness of the coating and from the coating/metal interface deep into the metal, the effect of micro-arc discharges was found not only on the properties of the oxide layers formed but also on structural changes in the thickness of the metal from the oxide/metal interface. Table 1 shows the results of measuring the microhardness of the transverse section of samples from the zero surface (the oxide layer) deep into the metal during the processing of MAO, depending on the duration of the anode current pulse. For comparison, the microhardness of an uncoated aluminum sample was experimentally established, which was 261 MPa.

According to the available model concepts [26], during the microplasma process, both the coating and the metal are exposed to temperature. Structural changes occur in the oxide layer and in the thickness of the metal. High current densities in the micro-arc channel lead to phase structural changes in the surface layers of the metal, to metal melting. At the same time, a part of the metal can be ejected through the channel (pore) to the outside. Then the ejected metal is converted into oxide and embedded in the coating. With the termination of the combustion of the micro-discharge, the oxides formed because of the process fill the breakdown channel. The pores become overgrown, their diameter decreases, and in some cases, they are completely closed by the products of plasma chemical reactions. The molten metal at the bottom of the pores undergoes a crystallization process.

Structural changes also depend on the thickness of the coating in the oxide layer. The thin oxide layers are formed at short durations of the anode pulse. In thin coatings, the cooling rate is higher, so the structural changes under the influence of temperature are less. In thicker coatings, temperature influences act for a longer time. Significant structural changes occur both in the oxide layer and in the metal. Comparative microhardness data (Table 1) confirm this. Thus, at the duration of the anode current pulse of 200 microseconds, the thickness of the oxide layer is 27.0 microns, and the microhardness is significantly higher than that of the oxide layer obtained at the duration of the anode current pulse of 50 microseconds, in which the coating thickness is 8.0 microns.

Part of the heat released because of micro-discharge penetrates deep into the metal. As a result, the tension in the metal is removed and the metal is released. As a result of structural changes in the aluminum alloy, under the influence of MAO, its microhardness from the oxide layer/metal interface deep into the metal is higher than the microhardness of aluminum not treated with MAO. As the distance deep into the metal from the oxide layer/metal interface increases, the microhardness decreases and eventually will be equal to the microhardness of the aluminum alloy (Figure 3).

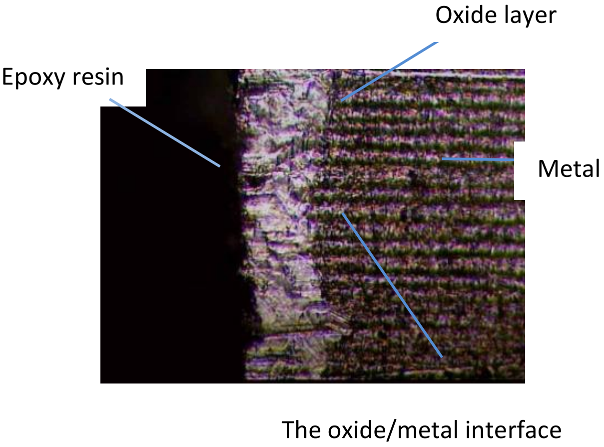


Figure 3 – Microplate of the sample processed by MAO

In previous studies, the effect of the micro-arc process on the crystal structure of the metal from the coating into the depth of the base material was observed [27]. According to the authors, a bath of molten metal is formed in the zone of thermal impact of microarc discharges at the interface of the metal oxide layer. With the termination of the micro-discharge, the process of metal crystallization begins. The metal bath cools down, while the bulk of the heat is withdrawn deep into the metal, affecting its structural changes [27].

Studies in the field of mechanics of contact interactions in the surface layers of rubbing materials show that the material in these layers changes its physical state during friction, the process of wear occurs. Three stages of wear are known (Figure 4). The first section of the curve

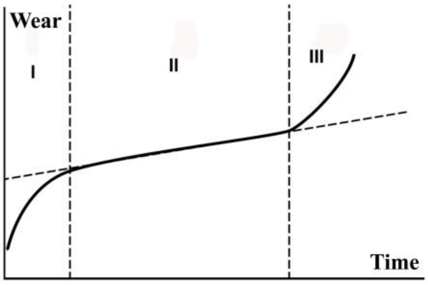


Figure 4 - Stages of the wear process

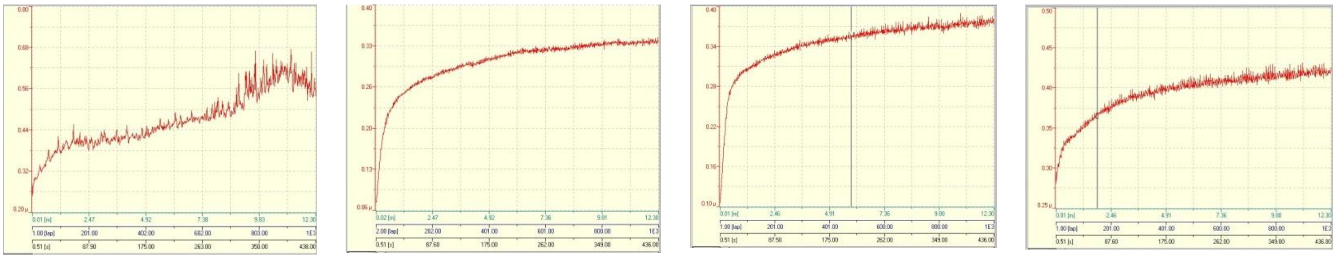

a b c d

Figure 5 – Curves of the quotient of friction of coatings obtained at different durations of the anode current pulse: a - 50 microseconds, b – 100 microseconds, c – 150 microseconds, d – 200 microseconds

(stage I) represents the initial wear period during which the rubbing surfaces adapt to each other, and this stage is called the burn-in stage. At this stage, there is a high intensity of wear. After the run-in stage, there comes a stage of steady wear (stage II), which has the longest duration. This stage is characterized by stable friction conditions and almost constant and relatively low wear intensity. During its development, wear gradually increases, which is accompanied by damage to the surface. Eventually, there is a significant change in the friction conditions, the intensity of wear increases sharply and catastrophic wear occurs (stage III).

Analysis of the curves of the friction quotient of oxide layers on aluminum obtained by the MDO method at different values of the duration of the anode current pulse shows that there are zones of run-in of the tribosystem, as well as a period with a stable friction condition (Figure 5). The oxide layer obtained at the duration of the anode current pulse of 50 microseconds is characterized by all stages of wear up to the wear of the coating (Figure 5 a). For other coatings, there is no sharp change in the quotient of friction characteristic of the destruction of the coating (Figure 5 b, c, d). The coatings don't break down and don't wear to the ground under the accepted test conditions.

Conclusions

The oxide coatings on aluminum alloy A0 were obtained in the pulse mode of the MAO. It is shown that structural changes under the influence of microplasma processes in the oxide layer make it possible to obtain wear-resistant coatings. When analyzing the friction quotient curves, it was found that the oxide coatings obtained at the duration of the anode current pulse of 100 μs – 200 μs are wear-resistant, the coatings don't collapse, don't wear to the ground under the accepted test conditions. The study of microhardness has shown that the microplasma process affects structural changes not only in the oxide layer, but also in the metal thickness. As a result of the thermal effect during the combustion of the microarc, an aluminum layer is formed with excellent physical and mechanical properties from aluminum that wasn't subjected to MAO treatment.

Conflict of interest

The correspondent author declares that there is no conflict of interest on behalf of all authors.

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Алюминий қорытпасы үлгілерінің қасиеттеріне микро доғалық тотығудың әсері

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ТҮЙІНДЕМЕ

Қазіргі уақытта заманауи өндіріс салалары алюминий, титан және олардың қорытпалары сияқты құрылымдық материалдарға ерекше талаптар қояды. Бұл материалдардың физика-механикалық, коррозиялық қасиеттерін жақсарту үшін әртүрлі әдістер қолданылады. Оларға көп функциялы қасиеттер беру үшін бетті түрлендірудің перспективті әдістерінің бірі – микро доғалық тотықтыру арқылы өңдеу. Процестің айрықша ерекшелігі – микро доға разрядтарының әсерінен клапан металдарында оксид жабындарының пайда болуы. Бұл жағдайда ерекше қасиеттері бар жабындар пайда болады. Алайда, микро доғалық тотығу процесінің негізгі материалдың қасиеттеріне әсері аз зерттелген. Бұл жұмыстың мақсаты импульстік режимде жүзеге асырылатын микро доғалық процесінің оксид қабаттарының қасиеттеріне және негізгі материалға әсерін зерттеу болып табылады. Қорытпаның бетін модификациялау анод режимінде, токтың анодтық импульсі ұзақтығының аз мәндерінде жүргізілді. Электролит ретінде электролиттің сілтілі ерітіндісі қолданылды. Оксид қабатының микро қаттылығын, сондай – ақ металл қабатының – оксид қабаты/металлдың ішкі бөлігіндегі металл қабатын зерттеу микро доға разрядтары тек оксид қабатының қасиеттеріне ғана емес, сонымен қатар металл қалыңдығындағы құрылымдық өзгерістерге де әсер ететіндігін көрсетті. Түзілетін оксид жабыны жоғары микро қаттылықпен сипатталады. 100 мкс – 200 мкс токтың анодты импульсінің ұзақтығы кезінде алынған оксидті жабындар тозуға төзімді болып табылады, жабындар бұзылмайды, сынақтың қабылданған жағдайында соңына дейін тозбайды.

Түйін сөздер: вентильді металдар, плазмалық электролиттік оксидтеу, микроплазмалық разрядтар, оксидтік жабын, микроқаттылық, ауыспалы қабат

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Влияние микродугового оксидирования на свойства образцов из сплава алюминия

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АННОТАЦИЯ

В настоящее время современные производства предъявляют особые требования к конструкционным материалам, таким как алюминий, титан и их сплавы. Для улучшения физико-механических, коррозионных свойств этих материалов применяют различные методы. Одним из перспективных способов модификации поверхности с целью придания ей многофункциональных свойств является обработка методом микродугового оксидирования. Отличительной особенностью процесса является образование оксидных покрытий на вентильных металлах в результате воздействия микродуговых разрядов. При этом образуются покрытия с уникальными свойствами. Однако влияние процесса микродугового оксидирования на свойства основного материала изучено мало. Целью данной работы является исследование влияния микродугового процесса, реализуемого в импульсном режиме, на свойства оксидных слоев, основного материала. Модификацию поверхности сплава проводили в анодном режиме, при малых значениях длительности импульса анодного тока. В качестве электролита использовали щелочной раствор электролита. Исследования микротвердости оксидного слоя, а также слоя металла от границы раздела – оксидный слой/металл вглубь металла показали, что микродуговые разряды влияют не только на свойства оксидного слоя, но и на структурные изменения толщины металл. Показано, что сформированное оксидное покрытие характеризуется высокой микротвердостью. Оксидные покрытия, полученные при длительности импульса анодного тока 100 мкс – 200 мкс, износостойкие, покрытия не разрушаются, не изнашиваются до основания при принятых условиях испытаний.

Ключевые слова: вентильные металлы, плазменное электролитическое оксидирование, микроплазменные разряды, оксидное покрытие, микротвердость, переходный слой.

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