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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
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## NEWS

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## **SURFACE HARDENING OF THE ALUMINUM ALLOYS AL3 BY ELECTROLYTIC-PLASMA TREATMENT**

**Abstract.** The article presents the research results of the effect of electrolytic-plasma processing on the structural-phase transformation of aluminum alloy samples. The discharge was ignited from the DC source. When the voltage is switched on, ionization and boiling of the electrolyte occurs. When bubble boiling occurs around the active electrode, large current pulsations are observed. Due to the formation of a gas-vapor jacket and the passage of electric current through it, a low-temperature plasma is formed, which has the characteristic blue color of the glow of the shell around the part. Electric micro-arc plasma is excited on the surface of the product, in which an intense heating of the workpiece occurs from heat generation. After microarc oxidation, the microstructure of quenching and artificial aging in the electrolyte flow is observed on the sample surface. As a result of quenching in the electrolyte stream, solid copper solution in aluminum and pinpoint fine inclusions dissolve from the microplasma temperature, the phases oxidize to form aluminum corundum. X-ray analysis of the samples after electrolytic-plasma processing revealed an increase in the intensity and broadening of the diffraction lines relative to the initial state, which indicates the residual surface stress, which during operation provides an increase in the wear resistance of the part. The average microhardness, after electrolytic-plasma treatment, is 746 MPa, which is about 2.5 times higher than that of the starting material.

**Key words:** aluminium, electrolyte-plasma treatment, micro-arc oxidization, microstructure.

**1. Introduction.** As it is known quenching is used for hardening aluminum alloys. Tempering is heating alloy to a temperature at which excessive intermetallic phases completely or predominantly soluble in aluminum. Exposure at this temperature and rapidly cooled to normal temperature allows obtaining a supersaturated solid solution. The heating temperature for quenching is selected depending on the nature of the alloy satisfying the mechanical property requirements for the part. Temperature of quenching alloy system Al-Cu (figure 1) defined by a line abs limiting solubility line passing above for alloys containing less than 5,7% Cu, and below the eutectic line (548 °C) for alloys containing greater amounts of copper [1].

High temperatures cause burnout (melting along the grain boundaries), which leads to cracking, reduced corrosion resistance, mechanical properties and resistance to brittle fracture. Quenching is followed by aging, where the alloy is kept at normal temperature for several days (natural aging) or for 10-24 hours at 150-200 °C elevated temperature (artificial aging) [2].

However, surface oxidized with aging after the quenching, it requires machining and as a result, coatings do not provide high surface wear resistance.

**2. Research method.** Micro plasma coating is applied to a very narrow range of materials [3]. And overwhelmingly is directed to obtaining a durable and wear resistant oxide layer on the anode surface of aluminum and its alloys [4]. At the same time, the potential of this method has not yet been disclosed, due to the poor knowledge of laws of formation of micro plasma discharges on the surface of the anode and the cathode in different electrolytes and their impact on the structure and properties of the surface of the

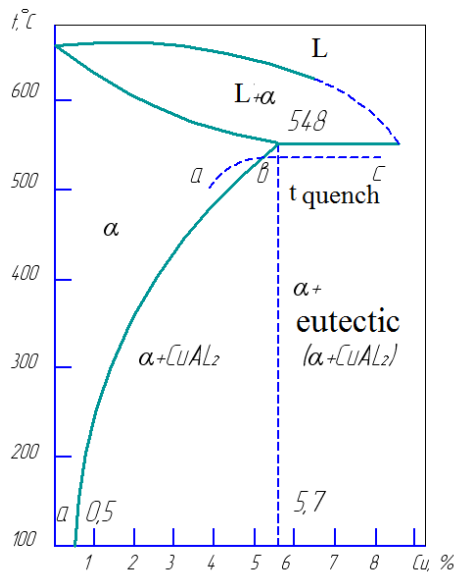


Figure 1 – The phase diagram of Al-Cu: dashed line is quenching temperature

electrodes. To develop the technology of applying various coatings on the surface of the work piece using micro plasma discharges in electrolytes requires studying the patterns of their formation and effects on the surface of the cathode [5]. It is known alternative device for micro-arc oxidation (MAO) wells gear pump housing made of aluminum alloy. The strongest structural hardening of aluminum alloys is shown at micro-arc oxidation [6].

To study the micro-arc oxidation samples measuring  $10 \times 10 \times 20$  mm (figure 2) was cut out of aluminum alloy A3 GOST2685-75, diamond disc, 1 mm thick, which is immersed in the coolant. At small cutting speed  $n = 350$  r / min, and low load,  $m = 250$ g, the sample does not undergo thermal deformation [7].

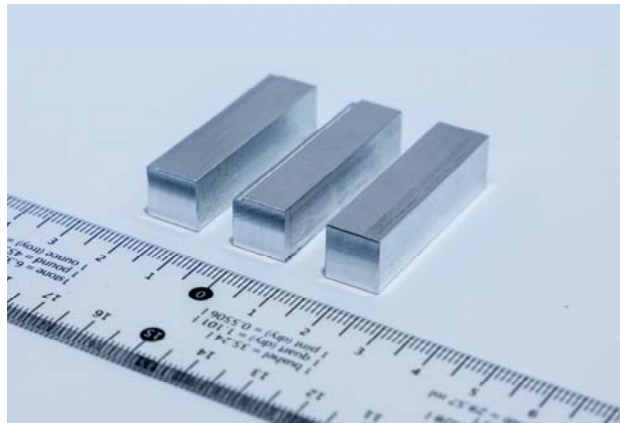


Figure 2 – Pieces for micro arc oxidation

Experimental studies and mechanical tests were carried out in the Regional university laboratory of engineering profile "IRGETAS" D.Serikbaev EKSTU (Ust-Kamenogorsk), Science, and Technology Park "Laboratory of engineering profile". K.I. Satpayev KazNTU (Almaty) [8]. The elemental composition of the aluminum piece was examined in a scanning electron microscope JSM-6390LV - company JEOL (Japan), prefixed energy dispersive microanalysis INCA Energy company «OXFORD Instruments» [9]. Defined elements from boron to uranium. The qualitative and quantitative phase analysis of the structure of aluminium alloy piece was carried out on the X-ray diffractometric DRON-3 in the filtered radiation of a copper anode, as «X'Pert PRO» company «PAN analytical», using Cu-K $\alpha$  radiation [10].

**3. Research results and discussion.** The discharge was ignited by a constant current source. Scheme of constant current source (figure 3a), which consists of push-button station, the starter, the diode

bridge, throttle, machine, ammeter, voltmeter, and other electrical appliances [11]. Cathode is a piece of aluminum (AL 3: 1,5-3,0 Cu; 0,35-0,6 Mg; 4,5-5,5 Si; 0,6-0,9 Mn), submerged to a depth of 4-6 mm in the electrolyte - 10% aqueous  $\text{Na}_2\text{CO}_3$  solution [12].

It should be noted that the quality of the surface layer, which was obtained using micro-arc oxidation, depends on the process conditions largely. The power of the spark discharges and accordingly energy impact on reinforcing surface are depending on them. When the voltage is turned on the surface of the exciting electric micro-arc (figure 3b), in which heat comes from the intense heating of the work piece.

As the applied constant voltage smoothly increases the salt is electrolyzing, with this process, according to Ohm's law, amperage increases too (section 0-A, figure 4). For this section: with increasing of the voltage, amperage increases proportionally. With this process electrolyte's temperature is increasing too, which is the consequence of the current passing through the electrolyte.

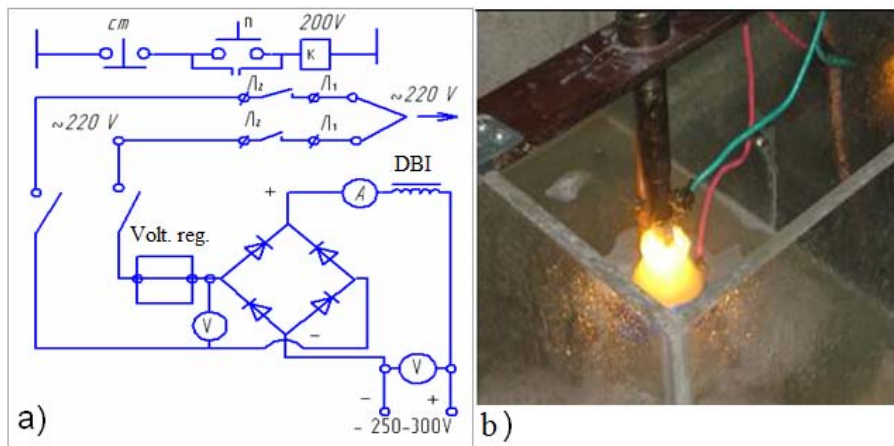


Figure 3 – Scheme of a constant current source for micro arc oxidation of aluminum alloys:  
a - electro scheme of constant current source; b - micro arc aluminum oxidation

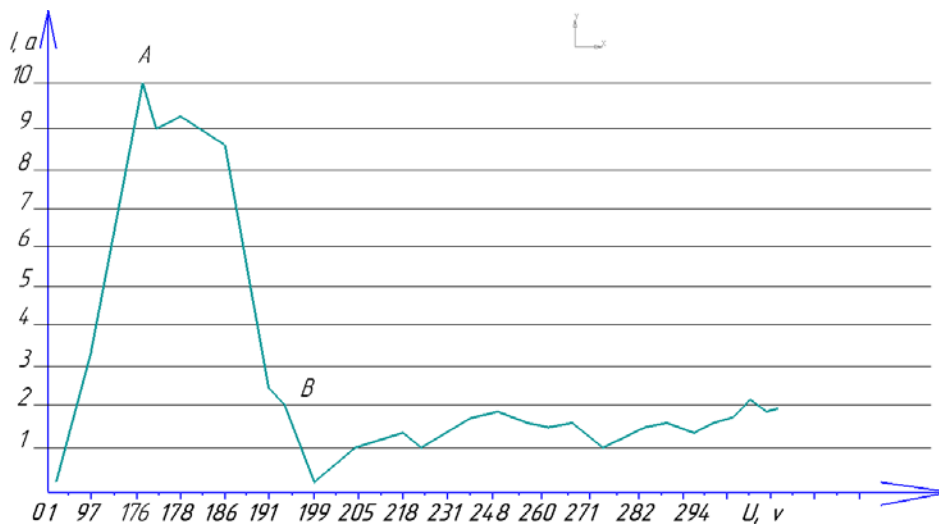


Figure 4 – Voltage-current characteristic of the electrolytium-plasma treatment

After reaching the particular value of voltage (100-180V) on the surface of detail's cathode the electrolyte starts boiling, blisters start actively appear on the surface (nucleate boiling). Nucleate boiling is type of boiling, when detail's temperature is close to the boiling point of water. When this process occurs, the high ripple current around the active electrode is observed. Their amplitude is significantly reduces, when detail heats up to 470°C. With further increasing of rectified voltage film boiling occurs (point A, figure 4), for which it is significant that nucleate boiling is vanishing and quick amperage slump, because



formed gas-steam envelope has higher resistance than liquid electrolyte (section A-B, figure 4). Since gas-steam envelope is less electroconductive, main voltage drop occurs in the zone, where the most heat is released. Because of the forming of gas-steam envelope and current passing through it, low temperature plasma is formed, which has blue envelope glow around the detail. The brighter blue glowing is, the more ions it has, including ion modifiers. With the further increasing of voltage the forming of anomalous discharge is observed.

Experiments have established the optimal modes of micro-arc oxidation of aluminum: voltage  $U = 200$  V, the current  $I = 10$  A, the heating time  $t = 4$  s., quenching time  $t = 4$  sec., the total number  $n = 15$  cycles [13].

An anode having a disk shape 50 mm diameter, 2 mm thick, with holes drilled  $\text{Ø}4$  mm 12X18H10T made of stainless steel [14]. Micro arc occurs between the cathode and a liquid electrolyte [15]. Of particular influence on the structural transformation has intermittent fever, when you connect the power supply, which rises above the phase  $\alpha \rightarrow L$ -conversion ( $548^\circ\text{C}$ ). After the power supply goes off micro-arcs that provides access to the electrolyte reheat alloy and rapid cooling (quenching).

The aluminum alloy structure present in equilibrium grain which is a solid solution (figure 5 a), which is composed of copper, manganese and magnesium, disperse the inclusion  $\text{Mg}_2\text{Si}$ .

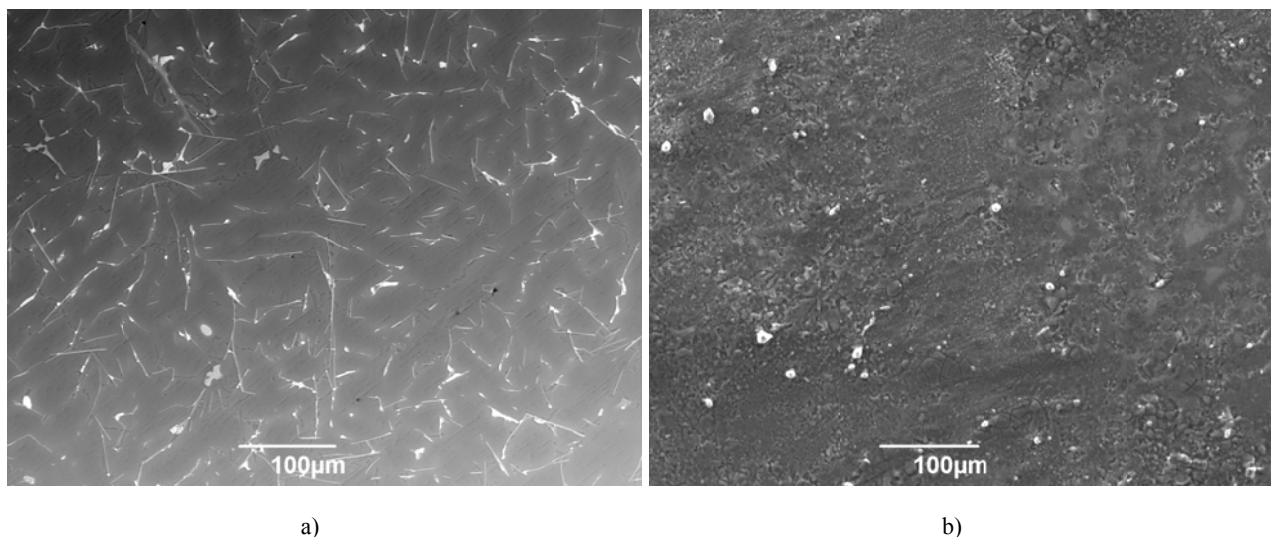


Figure 5 – The microstructure of aluminum alloy AL3:  
a - in the initial state; b - after the micro arc oxidation

After the micro-arc oxidation observed microstructure of hardening and artificial aging of the electrolyte stream (figure 5 b). As a result of quenching the solid  $\alpha$  - a solution of copper in aluminum and spot finely divided inclusions are dissolved by micro plasma temperatures, these phases are oxidized to form alumina corundum [16]. The results of elemental analysis after the micro-arc oxidation (table 1) indicate the appearance of oxygen and the inclusion of the relevant elements of the reinforcing aluminum alloy. Inclusions were allocated from  $\alpha$ -solid solution during artificial aging. After aging, the aluminum surface layer is oxidized. Elements standing out in a dispersed form strengthens the alloy. It can be detected  $\text{CuAl}_2$  particles and magnesium particles.

Table 1 – The elemental composition of the aluminum after MAO (to figure 5 b)

Spectrum	O	Na	Al	Si	Cu	Total
Spectrum 1	2.58	–	97.42	–	–	100.00
Spectrum 2	4.95	0.32	93.49	0.76	0.48	100.00

(All results in weight %)

To identify the structural and phase transformations of aluminum as a result of thermal effects micro-arc oxidation held X-ray analysis [17]. X-ray diffraction analysis of samples of aluminum in the initial state and after the delivery of micro-arc oxidation (figure 6) revealed the presence of lines  $\alpha$  - phase based on Al.

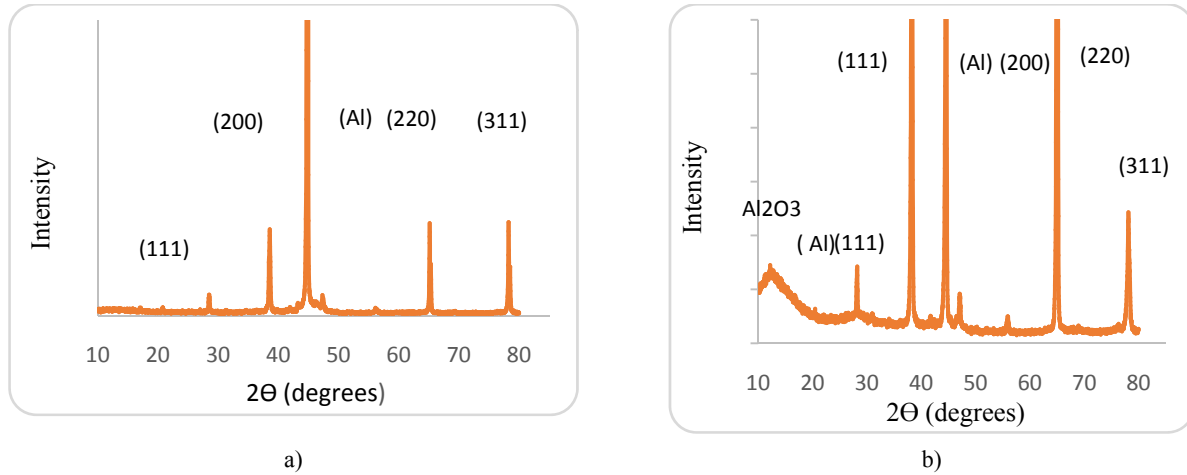


Figure 6 – X-ray diffraction pattern of an aluminum alloy: a) in the initial state, b) after the micro arc oxidation

After the micro-arc oxidation observed increase in the intensity and broadening of diffraction lines (figure 6 b) with respect to the initial state (figure 6 a), which indicates that the surface of the residual voltage, which is in service enhances the quality of the details. To determine the phase composition of the aluminum alloy sample treated with micro-arc oxidation implemented computerized statistical processing of results [18]. X-ray diffraction analysis of the pieces showed the presence of aluminum oxide phases. The values of the interplanar distances show that the angular position of the diffraction lines of samples does not fully coincide, and their intensity are significantly different (table 2).

Table 2 – phase composition of the aluminum piece after mao

№	I, mm	2θ, degree	θ, degree	Sin θ	d/n, Å	I, %	d/n, Å	I, %	Hkl	d/n, Å	I, %	Hkl
1	680	38.64	19.32	0.3308	2.330	100	2.338	100.0	111	2.315	45	401
2	56	44.84	22.42	0.3814	2.021	8.0	2.024	47.0	200	2.019	45	112
3	50	65.3	32.65	0.5395	1.429	7.0	1.431	22.0	220	1.426	10	710
4	11	78.42	39.21	0.6322	1.219	2.0	1.221	24.0	311			
5	10	82.58	41.29	0.6599	1.168	1	1.169	7	222			
6	7	112.2	56.1	0.8300	0.929	1	0.929	8	331			
7	7	116.62	58.31	0.8509	0.906	1	0.905	8.0	420			

Notes: I, mm – intensity of diffraction lines; 2θ – angle according to diffractometer goniometer; θ – angle of diffraction; sin θ – θ angle’s sine; d/n, Å – distance between grids of direct diffraction grating hkl; I, % – intensity of diffraction lines in percents; d – interplanar distance; n – order of reflections from the system of grids (hkl); hkl – indices of diffraction lines (Miller Indices).

This may be due to the fact that internal stress arises in the processing of the high temperature of plasma [19]. The sources of stress are the temperature gradients in the cross section; heterogeneity of the chemical composition; structural imperfections; different crystal orientation in space; different specific volume and different coefficients of linear expansion phases [20].

The micro hardness increased in all modes of processing micro-arc oxidation of aluminum alloy. The average micro hardness reached at micro-arc oxidation is 746 MPa (table 3), which is about 2.5 times higher than that of the starting material (figure 7).

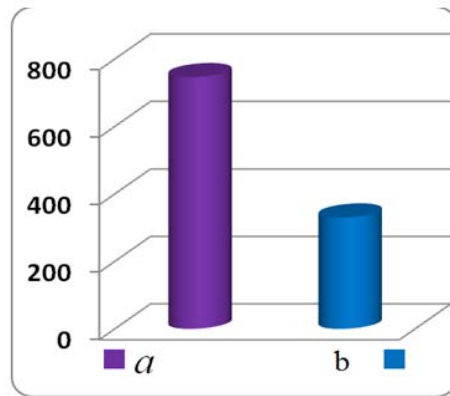


Figure 7 – the average value of micro hardness

Table 3 – Results of micro hardness after MAO

№	z1	z3	z2	z4	zr	zb	z	d	H, MPA	L, mkm	H <sub>avrg</sub>	H <sub>avrg</sub> -H	(H <sub>avrg</sub> -H) <sup>2</sup>
1	250	408	172	325	158	153	155.5	34.21	777.56	0	746	-32.06	1027.89
2	250	411	160	320	161	160	160.5	35.31	729.87	100		15.63	244.33
3	250	412	161	320	162	159	160.5	35.31	729.87	200		15.63	244.33
4	250	407	164	322	157	158	157.5	34.65	757.94	300		-12.44	154.71
5	250	405	163	325	155	162	158.5	34.87	748.41	400		-2.90	8.44
6	250	407	160	324	157	164	160.5	35.31	729.87	500		15.63	244.33
7	250	410	160	323	160	163	161.5	35.53	720.86	600		24.64	607.21
8	250	410	163	320	160	157	158.5	34.87	748.41	700		-2.90	8.44
9	250	409	164	318	159	154	156.5	34.43	767.66	800		-22.16	490.87
10	250	408	164	322	158	158	158	34.76	753.15	900		-7.65	58.51
11	250	411	164	322	161	158	159.5	35.09	739.05	1000		6.45	41.61
12	250	410	163	321	160	158	159	34.98	743.71	1100		1.79	3.22
13	250	407	161	320	157	159	158	34.76	753.15	1200		-7.65	58.51
14	250	409	162	324	159	162	160.5	35.31	729.87	1300		15.63	244.33
15	250	408	163	321	158	158	158	34.76	753.15	1400		-7.65	58.51

Notes: z<sub>1</sub>, z<sub>3</sub>, z<sub>2</sub>, z<sub>4</sub>, z<sub>r</sub>, z<sub>b</sub>, z – alculated parameters for determination of imprint's diagonals; d – length of imprint's diagonal of diamond pyramid on the sample's surface, mkm; h, MPa – microhardness value; L, mkm – distance between imprints of diamond pyramid; H<sub>avrg</sub> – average hardness value; H<sub>avrg</sub>-H – deviation from average microhardness value; (H<sub>avrg</sub>-H)<sup>2</sup> – standard deviation.

After the micro-arc oxidation observed increase in the micro hardness values (figure 7) with respect to the initial state, which certainly improves the performance of aluminum alloy parts [21].

#### 4. Conclusion.

1. Studies show that micro-arc oxidation under certain conditions leads to an increase in the strength properties of aluminum alloys.

2. It is found that the structural phase transformation toughening of aluminum alloys at micro-arc oxidation occurs at significantly lower energy consumption as compared to conventional heat treatment.

3. For hardening an aluminum alloy instead of traditional heat treatment, an alternative electrolytic-plasma hardening technology was proposed. Samples of aluminum alloy AL3 GOST2685-75 were subjected to cyclic heating from the temperature of microplasma and quenched in a stream of electrolyte. Heating, to the temperature of structural phase transformations, occurs at a significantly lower energy consumption compared with traditional heat treatment.

4. Experimentally established the optimal modes of microarc aluminum oxidation: power supply voltage  $U = 200$  V, current  $I = 10$  A, heating time  $t = 2$  sec., Hardening time  $t = 24$  sec., The total number of cycles  $n = 15$ . Pre-established the dependence of the heating temperature on the heating, cooling and voltage times which is expressed by the formula:  $T = 4.5 \times t^2 \text{ nag.} + 4.8 \times U - 18 \times t \times l$ . After microarc oxidation, the microstructure of quenching and artificial aging in the electrolyte flow is observed.

5. As a result of quenching, solid  $\alpha$  is a solution of copper in aluminum and dotted fine inclusions dissolving from microplasma temperature, these phases, when oxidized, form aluminum corundum.

6. X-ray analysis of the samples after electrolytic-plasma processing revealed an increase in the intensity and broadening of the diffraction lines relative to the initial state, which indicates the residual surface stress, which during operation provides an increase in the wear resistance of the part.

7. The average microhardness, after electrolytic-plasma treatment, is 746 MPa, which is about 2.5 times higher than that of the starting material.

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#### ЭЛЕКТРЛІ-ПЛАЗМАЛЫҚ ӨНДЕУ КЕЗІНДЕГІ AL3 АЛЮМИНИЙ ҚОРЫТПАЛАРЫН БЕТТІК БЕРІКТЕНДІРУ

**Аннотация.** Мақалада электролитті-плазмалық өндеудің алюминий қорытпасының үлгілеріне құрылымдық-фазалық өзгеру әсерінің зерттеу нәтижелері келтірілген. Разряд тұрақты тоқ корегінен тұтандырылған. Кернеуді қосқан кезде электролиттің қайнауы және иондалуы түзіледі. Көпіршікті қайнау кезінде белсенді электрод айналасында үлкен пульсті тоқ күштері байқалады. Газ-бұлық өрісінің пайда болу нәтижесінде және тоқ өту нәтижесінде төмен температуралық плазма түзіледі, ол көк түсті сипатқа ие үлгінің айналасында жалындайды. Бөлшектің бетінде электрлік микроплазма туындайды, жылу бөліну әсерінен дайындама белсенді қызады. Микродоғалық тотығудан кейін үлгінің бетінде шыңдалған микроқұрылым байқалады және электролит ағымында жасанды ескіру жүреді. Электролит ағымында шыңдалу нәтижесінде мыстың алюминидегі  $\alpha$  қатты ертілдісі және ұсақ түйіршікті қосындылар плазма температурасынан еріп тотығады да, алюминий корундын түзеді. Электролитті плазмалық өндеуден кейінгі бастапқы қалпымен салыстырғанда рентгенқұрылымдық талдау дифракциялық сызықтарының ұлғайуының және созылғанын байқатты. Беткі қабатының ішкі кернеуінің нәтижесі эксплуатациялау кезінде беткі төзімділігін қамтамасыз етеді. Электролитті-плазмалық өндеуден кейін орташа микроқаттылығы 746 МПа құрайды, бастапқы қалпынан 2,5 есеге жоғары.

**Түйін сөздер:** алюминий, электролиттік-плазмалық өндеу, микроарктік тотықтыру, микроқұрылым.

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#### ПОВЕРХНОСТНАЯ ЗАКАЛКА АЛЮМИНИЕВЫХ СПЛАВОВ AL3 ПРИ ЭЛЕКТРО-ПЛАЗМЕННОЙ ОБРАБОТКЕ

**Аннотация.** В данной статье приведены результаты исследований влияния электролитно-плазменной обработки на структурно-фазовое превращение образцов алюминиевого сплава. Разряд зажигался от источника постоянного тока. При включении напряжения происходит ионизация и кипение электролита. При возникновении пузырькового кипения вокруг активного электрода наблюдаются большие пульсации силы тока. Вследствие образования газопаровой рубашки и прохождения через нее электрического тока образуется низкотемпературная плазма, которая имеет характерный голубой цвет свечения оболочки вокруг детали. На поверхности изделия возбуждается электрическая микродуговая плазма, в которой от тепловыделения происходит интенсивный разогрев заготовки. После микродугового окисливания на поверхности образца наблюдается микроструктура закалки и искусственного старения в потоке электролита. В результате закалки в потоке электролита, твердый  $\alpha$  раствор меди в алюминии и точечные мелкодисперсные включения растворяются от температуры микроплазмы, фазы окисляясь, образуют корунд алюминия. Рентгеноструктурный анализ образцов после электролитно-плазменной обработки выявил увеличение интенсивности и уширение дифракционных линий относительно исходного состояния, что свидетельствует об остаточном напряжении поверхности, которая в процессе эксплуатации обеспечивает повышение износостойкости детали. Средняя микротвердость, после электролитно-плазменной обработки, составляет 746 МПа что примерно в 2,5 раза выше, чем у исходного материала.

**Ключевые слова:** алюминий, электролитно-плазменная обработка, микродуговое окисление, микро-структура.

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