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Investigation of the influence of electrolyte composition on the structure and properties of coatings obtained by microarc oxidation

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Aluminum alloys, along with positive properties - low density, high specific strength, electrical conductivity, ductility, viscosity and others, have disadvantages: low hardness, modulus of elasticity, wear resistance and high chemical activity in many inorganic acids.

The method of microarc oxidation (MAO) can eliminate these shortcomings. The transformation of the surface layers of the workpiece into high-temperature oxides of aluminum will strengthen the surface and increase its protective properties.

The properties of MAO coatings depend on many factors, one of which is the composition of the electrolyte. According to the literature, the most widely used alkaline-silicate electrolytes, which have shown the greatest efficiency in microarc oxidation of aluminum alloys. The data available in the literature on the use of multicomponent electrolytes containing sodium aluminate NaAlO_2 and sodium hexametaphosphate $\text{Na}_6\text{P}_6\text{O}_{18}$ do not allow to elucidate their role in the formation of MAO coatings.

Research of the use of hexametaphosphate to alkaline silicate electrolyte have shown that $\text{Na}_6\text{P}_6\text{O}_{18}$ promotes the formation of a thicker coating. The rate of thickness formation in the absence of hexametaphosphate is $0,5 \div 0,7 \mu\text{m}/\text{min}$, and at a hexametaphosphate content of $10 \text{ g/l} - 0,9 \div 1,1 \mu\text{m}/\text{min}$. As for the effect on the phase composition, the effect was not detected.

It was investigated that the addition of aluminate-silicate electrolytes of sodium aluminate in the amount of up to 13 g/l does not have a significant effect on the thickness of the coating, but affects the phase composition of the coating.

Keywords: microarc oxidation, morphology of the coating surface, phase-structural state, phases $\gamma\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$, $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$, sodium hexametaphosphate, sodium aluminate, alkali, liquid glass.

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Introduction

Aluminum alloys, along with positive properties - low density, high specific strength, electrical conductivity, ductility, viscosity and others, have disadvantages: low hardness, modulus of elasticity, wear resistance and high chemical activity in many inorganic acids [1].

The method of microarc oxidation (MAO) can eliminate these shortcomings. The transformation of the surface layers of the machined aluminum part into high-temperature aluminum oxides will strengthen the surface and increase its protective properties [2-3].

MAO is an electrochemical process that is carried out in electrolytes of different composition and with different electrical parameters of formation [3]. The properties of MAO coatings depend on many factors, one of which is the composition of the electrolyte. According to the literature [4-6], the most widely used alkaline silicate electrolytes containing $(1 \div 3) \text{ g/l KOH} + (3 \div 12) \text{ g/l Na}_2\text{SiO}_3$ (liquid glass), which showed the greatest efficiency in microarc oxidation of aluminum alloys.

Attempts have been made to use multicomponent electrolytes containing sodium aluminate NaAlO_2 [7] and sodium hexametaphosphate $\text{Na}_6\text{P}_6\text{O}_{18}$ [8]. The data available in the literature do not allow us to determine

the role of these soluble inorganic compounds in the formation of MAO coatings.

I. Goal

The goal of this work is to study the influence of electrolyte composition on the kinetics of coating thickness formation, their phase composition and hardness.

II. Experimental methods

The study was carried out on samples of AV alloys. The chemical composition of the alloys is given in table 1.

Microarc oxidation was carried out in the anodic-cathodic mode on an installation with a capacitor-type power supply. The duration of treatment was 1 hour at a current density of 20 A/dm².

The phase composition of the coating was determined from the diffractograms obtained on a DRON-3 diffractometer in $K\alpha$ -Cu radiation. The survey was carried out in a point-by-point mode with a step of $2\theta = 0.1^\circ$. The quantitative content of the phases was determined by the method of quantitative X-ray analysis using a previously constructed calibration curve according to the data of standard mixtures.

The microhardness was determined using a PMT-3 device. The coating thickness was determined using a VT-10 NTs.

III. Results and Discussion

The formation of MAO coatings leads to the formation of a developed surface relief, which has a significant roughness. The relief and surface roughness are approximately the same for all coatings with a thickness of $\sim 50 \mu\text{m}$ studied. Typical surface morphology is shown in Fig.1.

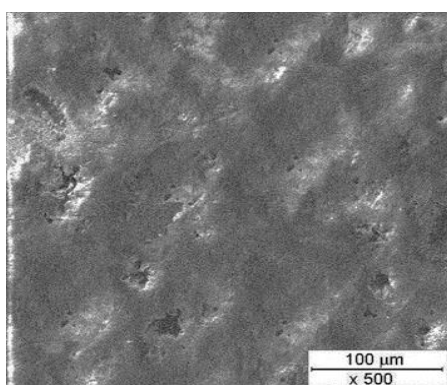


Fig. 1. Morphology of the surface of MAO coatings.

The study of transverse sections of coated samples (Fig. 2) revealed a layered structure of coatings - the technological layer and the main. The basic layer which is monolithic, firm and wearproof has practical value. Technological layer - highly porous, with low hardness, used as corrosion and electrical protection, as well as a sublayer when applying additional protective and decorative coatings. When high hardness and wear resistance are required from the surface, then the technological layer is removed.

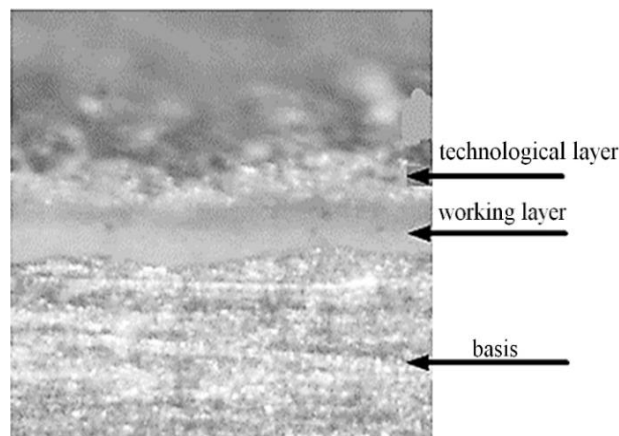


Fig. 2. Transverse section with coating (x 200).

The results below (hardness and phase composition) refer to the base layer.

The process of microarc oxidation was performed in alkaline-silicate electrolyte (base electrolyte) and with the addition of NaAlO₂ (sodium aluminate) and Na₆P₆O₁₈ (sodium hexametaphosphate).

The results of the study of coatings formed in the base electrolyte are given in table. 2 and in Fig. 3 - 4.

The kinetics of formation of the coating thickness and their microhardness are shown in Fig.3

As can be seen from the above data, increasing the concentration of Na₂SiO₃ increases the thickness of the coating, but the hardness of the main coating layer decreases (Fig. 3). This result can be explained by the change in the phase composition of the coating.

Thus, the results of deciphering the diffraction patterns of the base layer coatings showed that the diffraction maxima belong to the phases γ - Al₂O₃ and mullite (3Al₂O₃ · 2SiO₂) (Fig. 4), the phase α -Al₂O₃ (corundum) is absent. The presence of clear diffraction peaks on the diffraction pattern indicates the crystal structure of the main coating layer. A clear texture of the detected phases was not detected, which indicates the chaotic orientation of the crystals of the main coating layer.

The formation of the mullite phase 3Al₂O₃·2SiO₂ (Fig. 5), which has a relatively low hardness, and its increase to 85% reduces the hardness of the coating. However, certain adjustments must be made to the porosity of the coatings.

Table 1

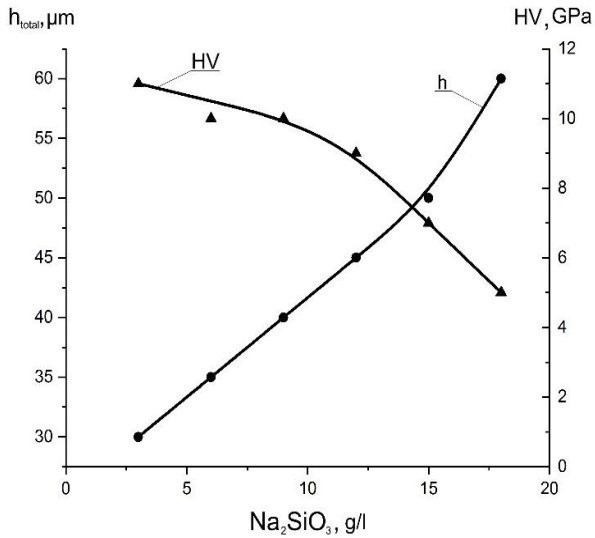
Chemical composition of AV alloys.

Material	Al	Cu	Mg	Fe	Si	Mn	Ti	Zn
AV	95.95- 98.8	0.1-0.5	0.45-0.9	0.5	0.5-1.2	0.15-0.35	0.15	0.2

Table 2

Properties of MAO coatings (AV alloy, $j = 20\text{A/dm}^2$, $\tau = 1$ hour).

Sample №	Electrolyte	$h_{\text{total}}, \mu\text{m}$	Phase composition, %		HV, GPa
			$\gamma\text{-Al}_2\text{O}_3$	$3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$	
1	1 g/l KOH + 3 g/l Na_2SiO_3	30	92	7	11
2	1 g/l KOH + 6 g/l Na_2SiO_3	35	87.5	12.5	10
3	1 g/l KOH + 9 g/l Na_2SiO_3	40	80	20	10
4	1 g/l KOH + 12 g/l Na_2SiO_3	45	58	42	9
5	1 g/l KOH + 15 g/l Na_2SiO_3	50	26	74	7
6	1 g/l KOH + 18 g/l Na_2SiO_3	60	15	85	5



1 - microhardness; 2 - coating thickness

Fig. 3. Dependence of the thickness and microhardness of the coating on the concentration of liquid glass (KOH - 1 g/l, current density $j = 20\text{A/dm}^2$, oxidation time $\tau = 1$ h).

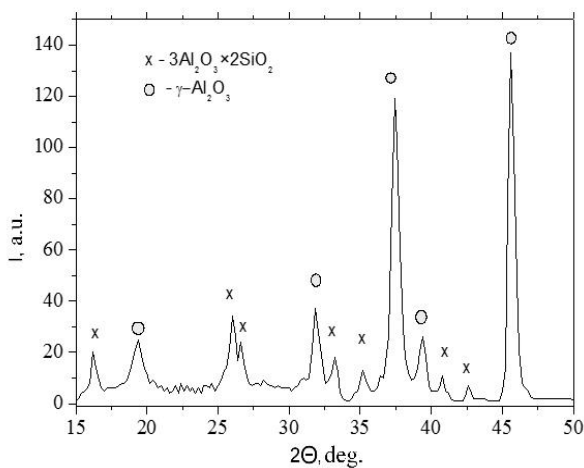
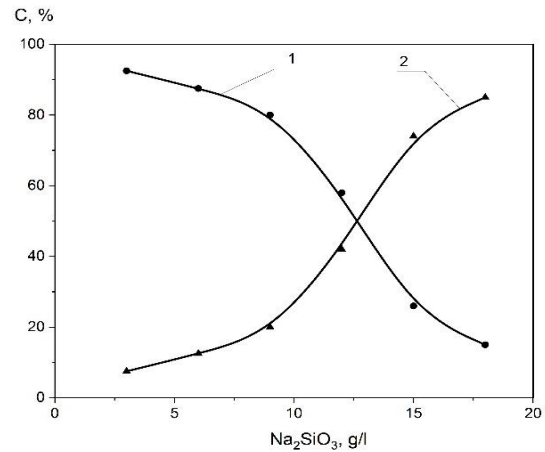


Fig. 4. Fragment of the diffraction pattern of the coating (shooting in monochromatic radiation: $\text{K}\alpha\text{-Cu}$).



1 - $\gamma\text{-Al}_2\text{O}_3$; 2 - $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$
(KOH - 1 g/l, $j = 20\text{ A/dm}^2$, $\tau = 1$ hour)

Fig. 5. Influence of liquid glass content Na_2SiO_3 on the phase composition of the coating.

The obtained data indicate that the phase formation of the coating in the alkaline-silicate electrolyte occurs from the formation of the $\gamma\text{-Al}_2\text{O}_3$ phase. In the process of increasing the coating and increasing the power of micro-discharges, the $\gamma\text{-Al}_2\text{O}_3$ phase interacts with the electrolyte components. When Na_2SiO_3 is dissolved in water, hydrolysis occurs by the reaction: $\text{Na}_2\text{SiO}_3 + \text{H}_2\text{O} \rightarrow 2\text{NaOH} + \text{SiO}_2$. The existing phase of $\gamma\text{-Al}_2\text{O}_3$ interacts with SiO_2 with the formation of the mullite phase: $3\text{Al}_2\text{O}_3 + 2\text{SiO}_2 = 3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$.

Thus, the increase in the electrolyte of liquid glass (Na_2SiO_3) promotes the formation of mullite, which does not provide the formation of a coating with high hardness. According to the obtained results, the content of liquid glass should be 3 - 6 g/l (see Fig. 3), which will ensure maximum hardness of the coating.

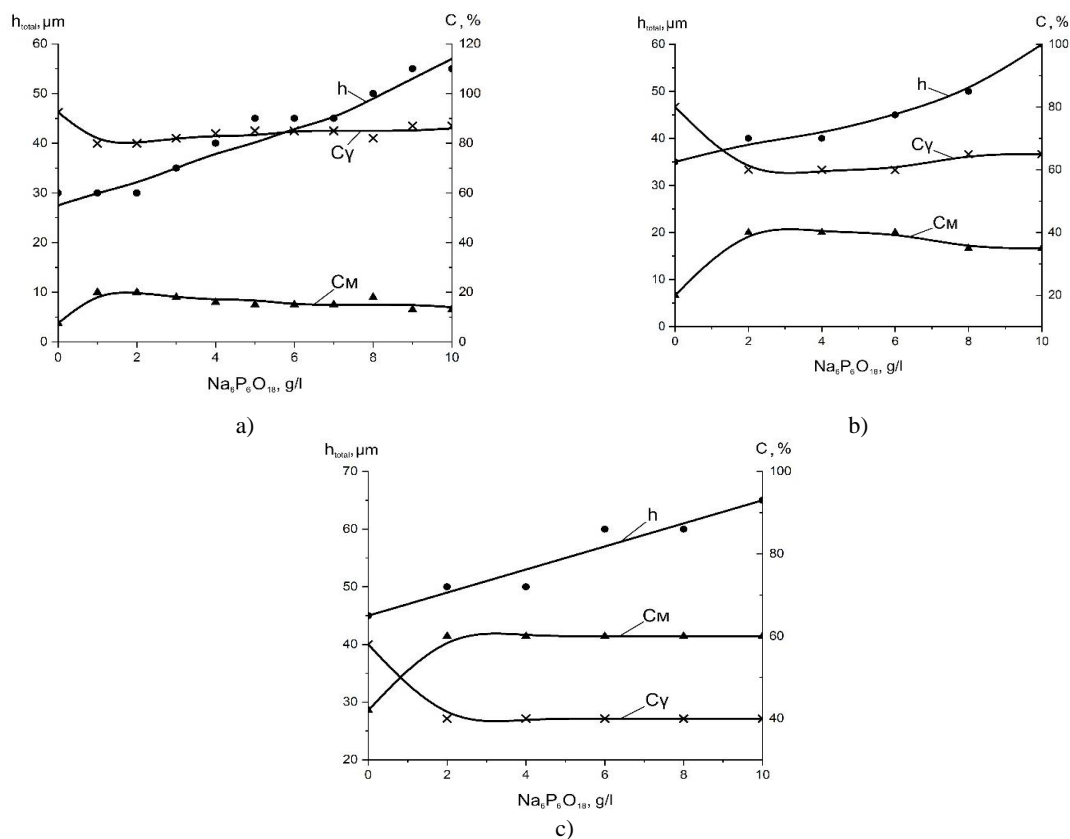
The study of the influence of the content of sodium hexametaphosphate $\text{Na}_6\text{P}_6\text{O}_{18}$ was carried out in the basic electrolytes of the composition: 1 g/l KOH + 3 g/l Na_2SiO_3 , 1 g/l KOH + 6 g/l Na_2SiO_3 , 1 g/l KOH + 12 g/l Na_2SiO_3 , to which was added $\text{Na}_6\text{P}_6\text{O}_{18}$ in an amount of 1 to 10 g/l.

The results of the study of thickness and phase composition are shown in table 3 and in Fig. 6.

Table 3

The results of the research of the $\text{Na}_6\text{P}_6\text{O}_{18}$ effect on the properties of coatings.

Sample №	Electrolyte	$h_{\text{total}}, \mu\text{m}$	$h_{\text{basic}}, \mu\text{m}$	Phase composition, %	
				$\gamma\text{-Al}_2\text{O}_3$	$3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$
1G	1:3 + 1 % $\text{Na}_6\text{P}_6\text{O}_{18}$	30	15	80	20
2G	1:3 + 2 % $\text{Na}_6\text{P}_6\text{O}_{18}$	30	15	80	20
3G	1:3 + 3 % $\text{Na}_6\text{P}_6\text{O}_{18}$	35	15	82	18
4G	1:3 + 4 % $\text{Na}_6\text{P}_6\text{O}_{18}$	40	15	84	16
5G	1:3 + 5 % $\text{Na}_6\text{P}_6\text{O}_{18}$	45	20	85	15
6G	1:3 + 6 % $\text{Na}_6\text{P}_6\text{O}_{18}$	45	20	85	15
7G	1:3 + 7 % $\text{Na}_6\text{P}_6\text{O}_{18}$	45	20	85	15
8G	1:3 + 8 % $\text{Na}_6\text{P}_6\text{O}_{18}$	50	20	82	18
9G	1:3 + 9 % $\text{Na}_6\text{P}_6\text{O}_{18}$	55	20	87	13
10G	1:3 + 10 % $\text{Na}_6\text{P}_6\text{O}_{18}$	55	20	87	13
11G	1:6 + 2 % $\text{Na}_6\text{P}_6\text{O}_{18}$	40	15	60	40
12G	1:6 + 4 % $\text{Na}_6\text{P}_6\text{O}_{18}$	40	15	60	40
13G	1:6 + 6 % $\text{Na}_6\text{P}_6\text{O}_{18}$	45	15	60	40
14G	1:6 + 8 % $\text{Na}_6\text{P}_6\text{O}_{18}$	50	15	65	35
15G	1:6 + 10 % $\text{Na}_6\text{P}_6\text{O}_{18}$	60	15	65	35
16G	1:12 + 2 % $\text{Na}_6\text{P}_6\text{O}_{18}$	50	20	40	60
17G	1:12 + 4 % $\text{Na}_6\text{P}_6\text{O}_{18}$	50	20	40	60
18G	1:12 + 6 % $\text{Na}_6\text{P}_6\text{O}_{18}$	60	20	40	60
19G	1:12 + 8 % $\text{Na}_6\text{P}_6\text{O}_{18}$	60	20	40	60
20G	1:12 + 10 % $\text{Na}_6\text{P}_6\text{O}_{18}$	65	20	40	60



a - base composition of 1 g/l KOH + 3 g/l Na_2SiO_3 , b - base composition of 1 g/l KOH + 6 g/l Na_2SiO_3 , c - base composition of 1 g/l KOH + 12 g/l Na_2SiO_3

Fig. 6. The effect of sodium hexametaphosphate on the thickness and phase composition of MAO coatings (Cm - content of $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$, $C\gamma$ - content of $\gamma\text{-Al}_2\text{O}_3$).

It was investigated that the addition of sodium hexametaphosphate in the amount of up to 2 g/l to the base electrolytes affects the phase formation, at a content of > 2 g/l the phase composition practically does not change. The increase in the thickness of the coating with increasing content of $\text{Na}_6\text{P}_6\text{O}_{18}$ can be explained by the participation of the components of the additive in the

formation of the oxide coating.

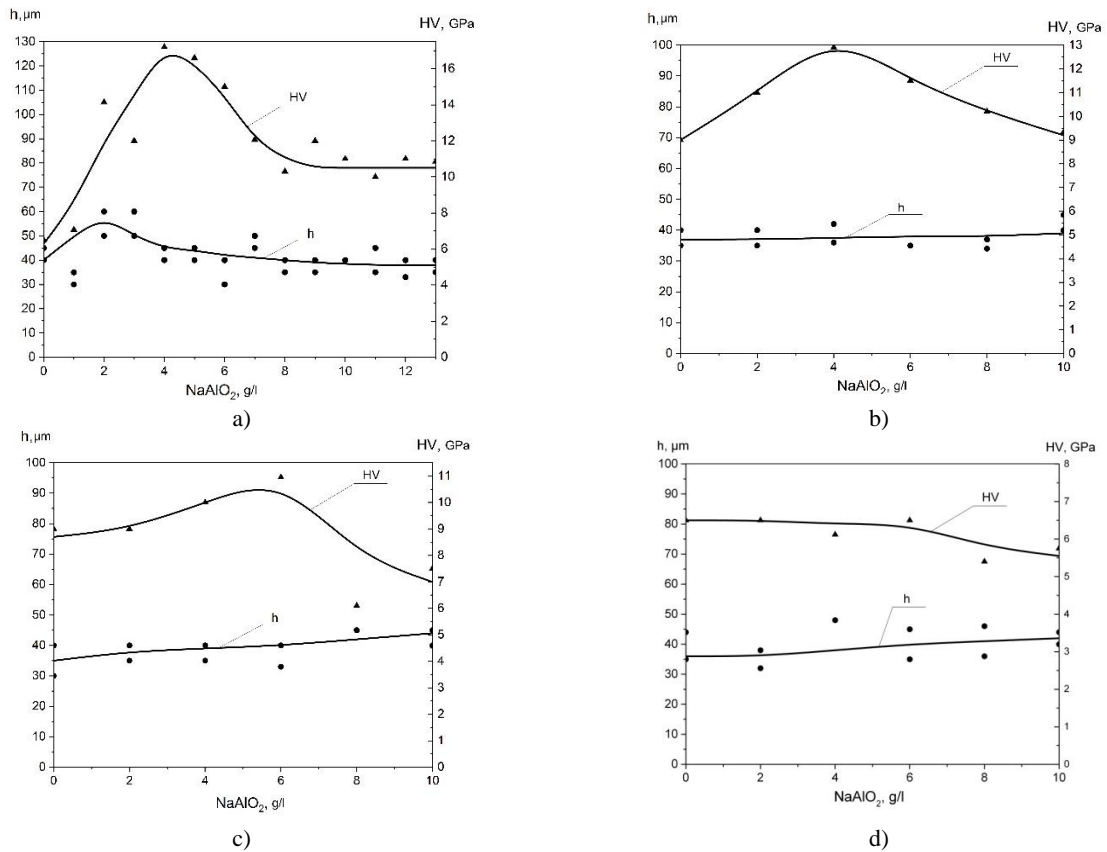
The detected dependences are equally manifested for different composition of the basic alkaline-silicate electrolyte.

The results of the study of the effect of sodium aluminate on the characteristics of the coating are given in table. 4 and in fig. 7.

Table 4

Characteristics of coatings formed in alkaline-silicate electrolyte with the addition of sodium aluminate.

Sample №	Electrolyte	h_{total} , μm	h_{basic} , μm	HV, GPa	Phase composition, %		
					$\alpha\text{-Al}_2\text{O}_3$	$\gamma\text{-Al}_2\text{O}_3$	$3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$
1A	1:3 + 1 % NaAlO ₂	30÷35	19	7.1	7	93	0
2A	1:3 + 2 % NaAlO ₂	50÷60	42	14.1	7	93	0
3A	1:3 + 3 % NaAlO ₂	50÷70	37	12	8	92	0
4A	1:3 + 4 % NaAlO ₂	40÷45	25	17.2	10	90	0
5A	1:3 + 5 % NaAlO ₂	40÷45	27	16	10	90	0
6A	1:3 + 6 % NaAlO ₂	30÷40	27	15	10	90	0
7A	1:3 + 7 % NaAlO ₂	45÷50	35	12.1	10	90	0
8A	1:3 + 8 % NaAlO ₂	35÷40	33	10.3	10	90	0
9A	1:3 + 9 % NaAlO ₂	35÷40	29	12	10	90	0
10A	1:3 + 10 % NaAlO ₂	40	27	11	10	90	0
11A	1:3 + 11 % NaAlO ₂	35÷45	30	10	10	90	0
12A	1:3 + 12 % NaAlO ₂	35÷40	29	11	10	90	0
13A	1:3 + 13 % NaAlO ₂	35÷40	31	10.9	10	90	0
14A	1:6 + 2 % NaAlO ₂	35÷40	28	11	10	85	5
15A	1:6 + 4 % NaAlO ₂	36÷42	30	12.9	10	80	10
16A	1:6 + 6 % NaAlO ₂	35	20	11.5	12	78	10
17A	1:6 + 8 % NaAlO ₂	34÷37	20	10.2	10	80	10
18A	1:6 + 10 % NaAlO ₂	40÷45	22	9.3	10	80	10
19A	1:9 + 2 % NaAlO ₂	35÷40	23	9	0	80	20
20A	1:9 + 4 % NaAlO ₂	35÷40	20	10	0	75	25
21A	1:9 + 6 % NaAlO ₂	33÷40	14	11	0	75	25
22A	1:9 + 8 % NaAlO ₂	45	20	6.1	0	75	25
23A	1:9 + 10 % NaAlO ₂	40÷45	22	7.5	0	75	25
24A	1:12 + 2 % NaAlO ₂	32÷38	20	6.5	0	58	42
25A	1:12 + 4 % NaAlO ₂	48	28	6.1	0	65	35
26A	1:12 + 6 % NaAlO ₂	35÷45	20	6.5	0	65	35
27A	1:12 + 8 % NaAlO ₂	36÷46	14	5.4	0	65	35
28A	1:12 + 10 % NaAlO ₂	40÷44	21	5.2	0	65	35



a - base composition of 1 g/l KOH + 3 g/l Na₂SiO₃, b - base composition of 1 g/l KOH + 6 g/l Na₂SiO₃, c - base composition of 1 g/l KOH + 9 g/l Na₂SiO₃, d - base composition of 1 g/l KOH + 12 g/l Na₂SiO₃

Fig. 7. The effect of sodium aluminate on the characteristics of MAO coatings.

The research of the effect of NaAlO_2 on the formation of coatings revealed (see table. 4):

Addition of alkali-silicate electrolyte NaAlO_2 has virtually no effect on the thickness of the coating;

NaAlO_2 affects the phase composition differently depending on the composition of Na_2SiO_3 in the base electrolyte:

in electrolytes 1 g/l KOH + 3 g/l Na_2SiO_3 , NaAlO_2 stimulates the formation of the phase α - Al_2O_3 , mullite is not formed (samples 1A - 13A);

in electrolytes 1 g/l KOH + 6 g/l Na_2SiO_3 addition of NaAlO_2 provides the formation of the α - Al_2O_3 phase (~ 10%), Na_2SiO_3 promotes the formation of mullite (samples 14A - 18A);

in electrolytes 1 g/l KOH + 9 g/l Na_2SiO_3 and 1 g/l KOH + 12g/l Na_2SiO_3 no effect of NaAlO_2 addition on the phase composition of the coating was detected (samples 19A - 23A and 24A - 28A).

Thus, the effect of adding sodium aluminate depends on the composition of Na_2SiO_3 in the alkaline-silicate electrolyte. To ensure maximum hardness, it is recommended to use an electrolyte of 1 g/l KOH + (3 ÷ 6) g/l Na_2SiO_3 with the addition of ~ 4 g/l NaAlO_2 .

Conclusions

1. It is determined that the phase formation of the coating in the alkaline-silicate electrolyte occurs from the formation of the γ - Al_2O_3 phase. The increase in the electrolyte of liquid glass (Na_2SiO_3) promotes the formation of mullite, which does not provide the formation of a coating with high hardness.

2. It is shown that hexametaphosphate promotes the formation of a thicker coating, the addition of sodium hexametaphosphate in the amount of ~ 2 g/l to the base electrolytes promotes the formation of mullite. At a content of 2 to 10 g/l the process of mullite formation is stabilized;

3. It was investigated that the addition of sodium aluminate to the base electrolytes in the amount of up to 13 g/l does not have a significant effect on the thickness of the coating. The effect of NaAlO_2 in different ways affects the phase composition of the coating - it depends on the composition of the alkaline-silicate electrolyte.

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Дослідження впливу складу електроліту на структуру та властивості покриттів, отриманих методом мікродугового оксидування

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Алюмінієві сплави, поряд з позитивними властивостями – низька щільність, висока питома міцність, електропровідність, пластичність, в'язкість та інші, володіють недоліками: низькі твердість, модуль пружності, зносостійкість і висока хімічна активність у багатьох неорганічних кислотах.

Усунути ці недоліки може метод мікродугового оксидування (МДО). Перетворення поверхневих шарів оброблюваної деталі у високотемпературні оксиди алюмінію дозволить забезпечити зміцнення поверхні та підвищити її захисні властивості.

Властивості МДО-покриттів залежить від багатьох факторів, одним з них є склад електроліту. Згідно з літературними даними, найбільш широко застосовуються лужно-силікатні електроліти, які показали найбільшу ефективність при мікродуговому оксидуванні алюмінієвих сплавів. Наявні в літературі дані щодо використання багатокомпонентних електролітів, які містять алюмінат натрію NaAlO_2 та гексаметафосфат натрію $\text{Na}_6\text{P}_6\text{O}_{18}$ не дозволяють з'ясувати їх роль у процесі формування МДО-покриттів.

Дослідження застосування гексаметафосфату до лужно-силікатного електроліту показали, що $\text{Na}_6\text{P}_6\text{O}_{18}$ сприяє утворенню більш товстішого покриття. Швидкість формування товщини за відсутності гексаметафосфату складає $0,5 \div 0,7$ мкм/хв, а при вмісті гексаметафосфату 10 г/л – $0,9 \div 1,1$ мкм/хв. Що стосується впливу на фазовий складу то впливу не виявлено.

Досліджено, що додавання до лужно-силікатних електролітів алюмінату натрію в кількості до 13 г/л не чинить суттєвого впливу на товщину покриття, але впливає на фазовий склад покриття.

Ключові слова: мікродугове оксидування, морфологія поверхні покриття, фазово-структурний стан, фази $\gamma\text{-Al}_2\text{O}_3$ і $\alpha\text{-Al}_2\text{O}_3$, $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$, гексаметафосфат натрію, алюмінат натрію, луга, рідке скло.