Investigation of coating thickness obtained by plasma electrolytic oxidation on aluminium alloys in electrolytes of type «KOH-H₃BO₃»

A Kosenko¹, A V Kolomeichenko², Yu A Kuznetsov³, N V Titov², R Yu Solovyev³, S N Sharifullin⁴

¹Ariel University Center, Izrael
²Orel State Agrarian University named after N.V. Parakhin, Russia
³Federal Scientific Agroengineering Center of All-Russian Institute of Mechanization, Russia
⁴Kazan (Volga region) Federal University, Russia

E-mail: Saidchist@mail.ru

Abstract: Plasma Electrolytic Oxidation (PEO) is one of advanced methods of obtaining thin layer oxide ceramic coatings on machine elements of different functional purpose providing considerable increase their resource. The work is devoted to the investigation of coating thickness formed by Plasma Electrolytic Oxidation on different aluminium alloys in electrolyte of type «KOH-H₃BO₃». Plasma Electrolytic Oxidation is recommended to be implemented at different engineering and repair and technical enterprises, dealing with production, restoration and hardening of elements.

1. Introduction

The method of obtaining oxide coatings on aluminum products has been known for a long time [1]. This is chemical and electrochemical oxidation. Disadvantages of this technology: the use of such harmful acids as phosphoric, sulfuric and hydrochloric, the possibility of obtaining only decorative and slightly wear-resistant coatings of several micrometers in thickness. These and other shortcomings did not allow widespread use of this technology.

To date, existing traditional technologies do not allow the production of coatings with such physical and mechanical properties as high hardness, strength, wear resistance, corrosion resistance. It is these coatings that make it possible to increase the service life of both manufactured and restored parts and assemblies of technical products. Only highly efficient technologies based on the use of concentrated energy flows are able to realize this task. In this paper, one of the technologies for using concentrated energy flows is proposed to increase the life of parts made of aluminum alloys. This is the technology of hardening by the method of plasma electrolytic oxidation (PEO) or as it is sometimes called the micro arc oxidation (MDO) method.

Increasing the resource of parts by restoring them and hardening them is the main reserve of material saving, reducing costs for repairing machinery and one of the most important problems in conditions of rising raw material costs and energy resources. One of the promising ways, which allows to significantly increase the life of parts made of aluminum alloys (pistons of internal combustion engines) is the method of plasma electrolytic oxidation (PEO).
The essence of PEO is the formation of a thin-layer high-strength wear-resistant ceramic coating on the surface of the workpiece under the action of microplasma discharges, consisting primarily of solid-phase aluminum oxides [2-13].

The main advantages of PEO can be attributed [3-9, 14, 15]: the possibility of coatings on complex-shaped products, internal surfaces and hidden cavities; Obtaining coatings up to 0.3 mm thick with adhesion comparable to that of the base material; Cheapness and availability of chemical reagents and materials; Environmental safety.

At present, many aspects of the mechanism of the PEO flow remain insufficiently studied: there is no systematic information about the influence of internal and external factors on this process, many new electrolytes have not yet been tested.

Below are the results of studies of the wear resistance of coatings obtained by PEO on various aluminum alloys in the electrolyte of the "KOH-H₃BO₃" type.

2. Methods of conducting research

Aluminium alloys of trade marks AMg2 (1520), AD1 (1235) and D16 (2024) were considered as material for sample production. They are widely used for production of elements of different nomenclature in processing industry. Aluminium alloys chemical content is presented in Table 1.

Plated samples of size 50x15x5 mm were used to carry out the investigations.

Table 1 – Mass fraction of elements in the alloy content (base – aluminium), %

|        | Mg  | Si   | Mn  | Cu  | Ti  | Fe  | Cr  | Zn   |
|--------|-----|------|-----|-----|-----|-----|-----|------|
| AMg2 (1520) | 1.7-2.4 | 0.4 | 0.1-0.5 | 0.15 | 0.15 | 0.5 | 0.05 | 0.15 |
| AD1 (1235)  | 0.05 | 0.3 | 0.025 | 0.05 | 0.15 | 0.3 | -   | 0.1  |
| D16 (2024)  | 1.2-1.8 | 0.5 | 0.3-0.9 | 3.8-4.9 | 0.15 | 0.5 | 0.10 | 0.25 |

It is suggested that the aluminium alloys data application provides the realization of method of installation of the hardened extra repair element (ERE) at the restoration of elements produced from different materials (for example, made of different materials (for example, bronze, corrosion-resisting steel) and contacting with liquid food mediums (milk, fruit extracts, etc). The main task being solved in this case is obtaining of hardened oxide ceramic layers, formed inward of base of oxidized aluminium alloy.

To form coatings on above mentioned aluminium alloys electrolyte of type «KOH-H₃BO₃» with the following components concentration for liter of distilled water: KOH – 5 g/l, H₃BO₃ – 15-30 g/l was used.

The sample oxidation was done on the Plasma Electrolytic Oxidation installation functioning in anode-cathode mode. Current density was 10…30 A/dm²; processing time – 120 min; electrolyte temperature was supported in the range of 30…35°C.

Coating thickness was determined on disks by means of device PMT-3.

3. Research results and discussion

The results of the researches showed that thickness of oxide ceramic coating obtained in electrolyte of type «KOH-H₃BO₃», depends on the modes of Plasma Electrolytic Oxidation and trade mark of aluminium alloy.

Figure 1 presents morphology and typical micro structure of coatings being formed by Plasma Electrolytic Oxidation on aluminium alloys.

Concentration KOH increase at other constant factors results in deeper penetration into alloy base, increasing thickness. Obviously it can be explained that for the sake of more intensive etching of the oxidized alloy surface, in coating formation the greater number of formed connections that had reacted with alkalis. Concentration KOH increase being more than 6 g/l does not sufficiently influence on coating thickness, because electrolyte aggressiveness increases. Obtained coatings turn out to be soft.
At KOH concentration being less than 2 g/l electrolyte has low dispersive capacity and obtaining of thick uniform coatings is difficult.

Boric acid concentration increase at other constant factors of Plasma Electrolytic Oxidation does not sufficiently influence on change of coating thickness. In our case boric acid is used as anode inhibitor, increasing break down strength. Boric acid concentration increase being more than 25 g/l is not rational from economic point of view. At boric acid concentration being less than 15 g/l to switch into Plasma Electrolytic Oxidation is not possible. The above mentioned is well coherent with the data of other scientists [3, 9].

Current density growth at other constant factors of Plasma Electrolytic Oxidation results in increase of hardened layer thickness (Figure 1). It provides more intensive process of Plasma Electrolytic Oxidation at the account of increase of microdischarges number on the oxidized surface. Nevertheless at current densities being more than 35 A/dm$^2$ micro plasma discharges are changed into arc ones. Deep craters and delaminations are observed on the surfaces of the oxidized samples, coating is destroyed.

Carrying out plasma electrolytic oxidation at low current densities is not rational from technological point of view, because to obtain the required thickness is necessary to have sufficient amount of time.

Coatings obtained on alloy AMg2, have maximum thickness, because magnesium in alloy with aluminium form uniform oxide connections (Figure 1). Coatings obtained on alloys AD1 and D16, do not differ sufficiently.

To maintain the main factors influence on coating thickness change and also taking into consideration their interaction the investigation was done according to the method of full factorial experiment planning $2^3$. Matrix of planning $2^3$ and mean real values of optimization parameter are presented in Table 2.

| Number of plan point | Code factor designations | Geometry parameters (process factors) | Mean real values of optimization parameter h, µm |
|----------------------|--------------------------|----------------------------------------|-----------------------------------------------|
|                      | $X_1$ | $X_2$ | $X_3$ | $D_t$ | CH$_3$BO$_3$ | СКОН | AMg2 | AD1 | D16 |
| 1                    | -     | -     | -     | 15    | 20       | 4    | 106,2 | 104,34 | 97,8 |
| 2                    | +     | -     | -     | 25    | 20       | 4    | 127,8 | 126  | 120,6 |
| 3                    | -     | +     | -     | 15    | 30       | 4    | 113,4 | 108  | 106,3 |
| 4                    | +     | +     | -     | 25    | 30       | 4    | 135   | 126  | 122,4 |
| 5                    | -     | -     | +     | 15    | 20       | 6    | 109,2 | 108  | 104,4 |
The experiment data processing, creation of mathematical model of process and check of its adequacy were done using PC by means of software product MS Excel. As the result the following regression equations were obtained:

For AMg2: \( h = 121,95 + 11,7 X_1 + 2,85 X_2 + 1,75 X_3 + 0,3 X_1 X_2 X_3; \)
\[
(1)
\]

For AD1: \( h = 118,4 + 10,81 X_1 + 0,95 X_2 + 2,26 X_3 + 0,5 X_1 X_2 X_3; \)
\[
(2)
\]

For D16: \( h = 114,16 + 10,49 X_1 + 1,96 X_2 + 2,39 X_3 + 1,06 X_1 X_2 X_3; \)
\[
(3)
\]

Considering their ratios importance for obtaining the required thickness all three factors were controlled.

Then the experiment results were processed using the least square method by means of software product MS Excel. As the result the process mathematical models in the form of linear equations were obtained:

For AMg2: \( h = 54,15 + 2,34 D_t + 0,57 C_{H_3BO_3} + 1,35 C_{KOH}; \)
\[
(4)
\]

For AD1: \( h = 59,04 + 2,16 D_t + 0,19 C_{H_3BO_3} + 2,26 C_{KOH}; \)
\[
(5)
\]

For D16: \( h = 50,45 + 2,09 D_t + 0,39 C_{H_3BO_3} + 2,4 C_{KOH} \)
\[
(6)
\]

For final check of the obtained models adequacy the results of coating thickness measurements obtained in the pilot experiment were used. Substituting into equation \( D_t = 20 \text{ A/dm}^2; C_{H_3BO_3} = 25 \text{ g/l}; C_{KOH} = 5 \text{ g/l}, \) we will get: for AMg2: \( h = 122 \mu m, \) for AD1: \( h = 118 \mu m, \) for D16: \( h = 114 \mu m. \)

Comparison of these calculated values with experimental values points to the mathematical model adequacy.

4. Conclusions

Taking into account that the thickness of coating necessary to provide high wear resistance, corrosion resistance, low porosity must be no less than 90…120 µm, to harden surfaces of extra repair element (ERE) (for example, repair bushings) in electrolyte of type «KOH-H_3BO_3», it is possible to recommend the following:

- to produce repair bushings ERE one should use alloy AMg2;
- to use electrolyte of the following content, g/l: KOH – 4…6, H_3BO_3 – 20…25;
- the recommended electrolyte temperature 30…35 °C;
- the current density during the process one should maintain about 20…25 A/dm²;
- the recommended duration of Plasma Electrolytic Oxidation – 110…130 min.

This method of elements surface hardening can be recommended for implementation at engineering and repair and technical enterprises, dealing with production, restoration and hardening of elements. The expected life growth of hardened elements will correspond to not less than 150-200% in relation to new elements.

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