# Methods of Structural Engineering of Surface in Solving the Problems of Multifactorial Increase of the Level of Operational Characteristics of Materials

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Abstract. In the course of the study, several different methods of surface structural engineering are reviewed. The methods described in this paper are characterized by different process physics on the way to obtaining the result, but they are aimed at modifying the structure and properties of the surfaces to which they are applied. Among them, two different technological directions are considered. The first area involves technologies that include a friction component, namely thermofriction treatment (TFT) for thermofriction strengthening (TFS), additional thermofriction strengthening (ATFS) or thermofriction welding (TFW). The second direction is a technology that involves the use of an anode-cathode electrolysis mode in an alkaline-silicate electrolyte - microarc oxidation (MAO). The paper describes the features and results of the application of such technologies and the feasibility of using this or that method for materials of different classes, and presents schemes of the corresponding installations. The result of additional hardening of the surface of U8A steel from a microhardness level of 7.2 GPa to 14.7 GPa using the ATFS method after its thermal hardening to almost the maximum possible level is shown. The microstructure of the cross-section of a prehardened specimen of U8A steel after ATFS is presented, where the degree and nature of surface hardening are reliably visible. It is emphasized that in previous studies, consistently effective hardening of steels of various classes has been achieved, even up to the level of 22 GPa in 65G steel. Regarding the method of microarc oxidation, the structure and properties of coatings on low-alloy aluminum alloys AB and AD1 formed in an alkaline-silicate electrolyte in the anode-cathode MAO mode were investigated. It is shown that the method of MAO in alkalinesilicate electrolyte allows to obtain a coating thickness of up to 300 µm, a coating growth rate of  $\sim 2 \mu m/min$ , and a coating hardness of 10-20 GPa. The coatings have high adhesion to the substrate; they have a layered structure. The properties of the coatings are determined by the properties of the base layer. The coatings have a crystalline structure and consist of the following phases:  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>), the ratio between the phases depends on the electrolysis conditions. It has been established that phase formation begins with the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase, which in the process of further coating growth turns into the α-Al<sub>2</sub>O<sub>3</sub> phase or interacts with silicon oxide to form the mullite phase.

## **1** Introduction

The properties of products made of different materials and the condition of their surfaces are closely related, especially when they are operated with friction [1, 2]. And here it should be emphasized that achieving high levels of functional properties of materials, respectively, ensures an increase in the performance characteristics of machine parts, tools, and structural elements [3, 4, 5]. An

effective way to improve the operational properties of surfaces is also the use of chemical and thermal methods of saturating surface layers in products made of various materials [5, 6, 7]. The processes of surface layer modification usually occur under non-equilibrium conditions and are a component of structural surface engineering. In most cases, the use of appropriate technologies of this orientation leads to a decrease in the size of crystalline grains to the nano level [8, 9].

Therefore, a large number of surface treatment methods have been developed to date that allow controlling the composition, structure, and properties of surface layers. Structural surface engineering encompasses a set of scientific disciplines and technological methods for targeted changes in the physical and chemical properties of the upper layers of materials by modifying, deforming, applying films, coatings, and protective layers, using various combined methods. This makes it possible to design surface layers of materials and control their properties.

The analysis of the proposed methods of structural engineering of the surface of materials and products made of them shows that the improvement of the operational properties of many types of machine parts, tools, and structural elements is determined mainly by the properties of the surface layers of the parts (hardness, wear resistance, friction coefficient, endurance limit, contact stiffness, corrosion resistance, etc.) There are various approaches to the implementation of structural surface engineering that differ from each other, but all of them are capable of radically changing the properties of the surface of the objects to which they are applied.

### 2 Experimental Part

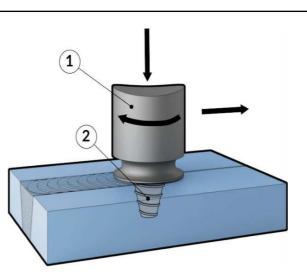
Friction welding and surfacing is characterized by the fact that the main processes take place in the surface layers of the structural elements that are joined together. Such welding processes are aimed at producing permanent joints and can be classified, depending on the type of friction used:

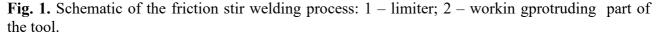
- linear friction welding, i.e., as a result of the rotational action of the contacting parts or one of them under a certain pressure, which contributes to rapid local heating, melting and formation of a permanent joint;

- radial friction welding, when the joints of the parts to be joined are heated by a rotating ring, which is worn externally and generates a melting point temperature under the action of friction with the formation of a permanent joint after cooling;

- friction stir welding, when a rotating pin heats the parts at the joint and, under the influence of high temperature, local melting occurs with the pin penetrating the parts to be joined. When cooled, a permanent connection is formed.

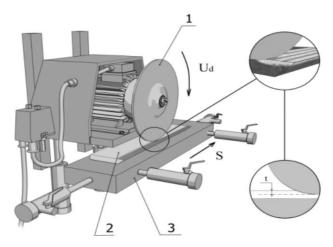
Friction stir welding [10] is gaining considerable scientific and practical interest, being more complex but most interesting in terms of forming properties in the surface of the welded objects. The metals are joined by friction heating in the welding zone. The main tool for such friction stir welding is a metal rod consisting of two parts: a working protruding part (drill) and a limiter (shoulder). When the working protruding part of this rotating tool is deepened into the material, it is heated significantly. The vertical feed of the tool is limited by the shoulder, which does not allow the object to be welded through. In the heating zone, the material significantly increases its plasticity and acquires a state of fluidity and, due to pressure from the limiting shoulder, forms a single mass. This method does not involve the use of various additional media that can release combustion products into the atmosphere during the burning of an arc or gas jet, which is inherent in many standard welding and surfacing methods. Consequently, it is quite environmentally friendly and promising, as it also allows you to control the properties of materials in the resulting joints within a certain range. There are many differences in technological approaches to the realization of such processes, but the basic idea of this method is shown in Fig. 1.



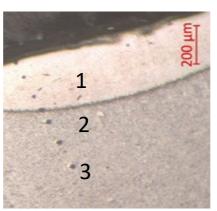


Another method of surface modification is the technology of thermofriction strengthening (TFS), which is aimed at creating surface layers with special properties [11]. Schematically, this method is shown in Fig. 2. TFS does not involve additional alloying of the material to be strengthened, which characterizes this method as environmentally friendly, effective, and promising, including from an economic point of view. It is worth noting that the frictional-strain component of this method can be used for both primary and additional strengthening of materials, depending on the needs for the properties of products made from them. This implies the use of different processing modes for each of these cases. The obtained experimental results showed that the TFS method differs from others in its ability to significantly increase the hardness of materials that have already been thermally hardened to the maximum possible level in the thermal sense.

For example, results have been obtained where the thermofrictional strengthening of samples made of 65 G steel allowed to increase the microhardness of their surface layer to 22 GPa. In addition, there are results confirming that a very significant level of surface hardening can be obtained in this way for a wide range of steels of different classes [7]. At the same time, the stability of the high level of additional hardening is evident. Fig. 3 shows the microstructure of a cross-section of a U8A steel specimen after additional thermofriction strengthening (ATFS), where the degree and nature of surface hardening are clearly visible. Therefore, the result of additional hardening of the surface of U8A steel from a microhardness level of 7.2 GPa to 14.7 GPa using the ATFS method after its thermal hardening to almost the maximum possible level is presented.



**Fig. 2.** Schematic of the process of additional thermofriction strengthening (ATFS): 1 – strengthening disk; 2 – hardening object; 3 – magnetic plate of the machine.



**Fig. 3.** The result of ATFS – the formed surface hardened layer, which is presented in the crosssection of the sample with the prints from the microhardness measurement (Steel U8A): 1 - strengthening surface layer; 2 - weakened subsurface layer; 3 - main part of the sample.

The micro-arc oxidation (MAO) method is quite promising today [12, 13], as it demonstrates high environmental performance and can be quite economical, depending on the electrolysis conditions. The use of this method provides the highest efficiency of obtaining high-hardness protective coatings on valve metals and alloys (Al, Ti, Mg, Ta, Nb, Zr) [14–18]. MAO is an electrochemical method that originates from traditional anodizing, but allows the formation of multifunctional ceramic-like coatings with a wide range of properties, such as hardness, wear resistance, corrosion resistance, electrical insulation and decorativeness, and others, that are fundamentally different in properties. A special feature of micro-arc oxidation is the participation of micro-arc discharges in the coating formation process. This combines plasma and electrochemical mechanisms of oxide layer formation [19], as a result of which the surface layer of the material being treated is converted into high-temperature crystalline oxides that are firmly bonded to the substrate and are characterized by high resistance to corrosion and wear.

MAO is a complex process in which the properties of coatings depend on many factors, such as electrolyte composition, voltage polarization conditions, current density, processing time, and others. However, many issues remain unclear regarding the impact of the composition of the materials being processed, the composition of the electrolyte, and technological factors on the processes of structure and phase formation and the properties of coatings. Resolving these issues will speed up practical implementation. Let us consider the MAO method in more detail.

The purpose of this part of the study is to develop scientific and technological principles for the formation of multifunctional coatings on valve metals using the micro-arc oxidation method.

The following tasks were solved for this purpose:

- to optimize the electrolysis conditions that ensure the formation of coatings in the microarc discharge mode on aluminum alloys;

- to determine the regularities of the formation of thickness, phase composition and properties of coatings depending on the electrolysis conditions (electrolyte composition, current density, processing time);

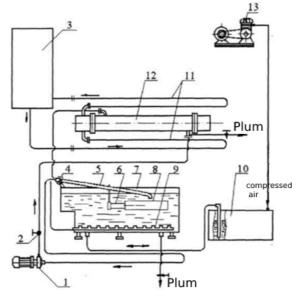
- determine the effect of the phase state of coatings on wear resistance and hardness;

- generalize the possibilities of using MAO coatings and develop recommendations for the use of microarc oxidation technology to increase the durability of products.

The coatings were formed on an installation that provided the process of microarc discharges (Fig. 4). The installation scheme is shown in Fig. 5.



Fig. 4. Micro-arc oxidation process.



**Fig. 5.** Schematic of the micro-arc oxidation plant: 1 - electrolyte pumping pump; 2 - ball valve; 3 - refrigeration machine; 4 - swivel tee; 5 - intake pipeline; 6 - workpiece mounting bracket; 7 - electrolytic bath; 8 - workpiece; 9 - pipe for air supply to the bath; 10 - air filter; 11 - insulating pipelines; 12 - heat exchanger; 13 - compressor.

Aluminum alloys were subjected to microarc oxidation, namely, low-alloy aluminum alloys AD1 and AB (Table 1).

<b>Table 1.</b> Chemical composition of the studied anoys, [76] (0051 4784-97)								
Material	Al	Cu	Mg	Fe	Si	Mn	Ti	Zn
AD1	99.3	0.05	0.05	0.3	0.3	0.25	0.15	0.1
AB	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

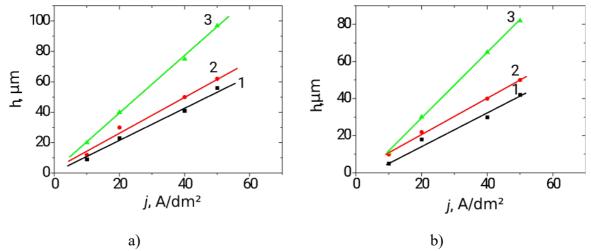
 Table 1. Chemical composition of the studied alloys, [%] (GOST 4784-97)

To ensure the micro-arc oxidation regime, electrolytes consisting of caustic potassium (KOH), liquid glass (Na<sub>2</sub>SiO<sub>3</sub>), and distilled water were used.

MAO treatment was carried out at a current density of 5-50  $A/dm^2$ , and the treatment duration was up to 3 hours.

To study the structure and properties, we used metallography, X-ray diffraction analysis, hardness measurement methods, and coating thickness measurement methods.

The MAO treatment of AD1 alloy was carried out in an alkaline-silicate electrolyte, which made it possible to form a coating with a thickness of up to 100  $\mu$ m, depending on the composition of the electrolyte and the current density. To accelerate the rate of coating growth, it is necessary to increase the current density. An increase in current density from 5 A/dm<sup>2</sup> to 50 A/dm<sup>2</sup> leads to a 5-fold increase in the rate of thickness formation (Fig. 6).



**Fig. 6.** Effect of current density on total (a) and working (b) thickness of coatings on AD1 aluminum (duration 30 min):  $1 - \text{electrolyte 1 g/l KOH} + 3 \text{ g/l Na}_2\text{SiO}_3$ ;  $2 - \text{electrolyte 1 g/l KOH} + 6 \text{ g/l Na}_2\text{SiO}_3$ ;  $3 - \text{electrolyte 2 g/l KOH} + 12 \text{ g/l Na}_2\text{SiO}_3$ .

A characteristic feature of MAO coatings is their layered structure. In Fig. 7 clearly shows the technological and working layers. The technological layer is porous, wear-resistant, easily removed with abrasive paper, and makes up 30-40 % of the total thickness.

The working layer is monolithic and wear-resistant. The phase composition of the technological and working layers is different.

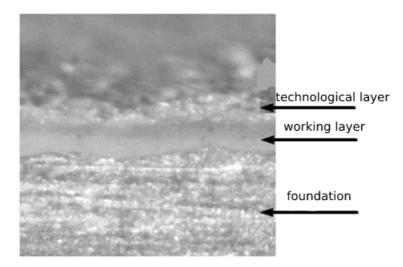
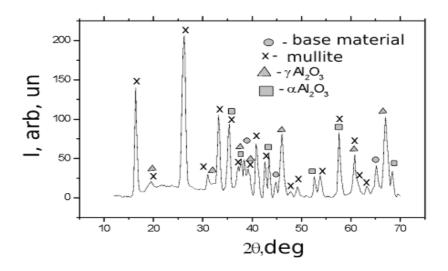


Fig. 7. MAO coating on aluminum alloy AD1.

The X-ray phase analysis showed that the coating on AD1 alloy has a crystalline structure, the main phase being  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> (Fig. 8). With an increase in the concentration of the Na<sub>2</sub>SiO<sub>3</sub> component in the electrolyte, the content of the crystalline mullite phase in the coating increases.



**Fig. 8.** A fragment of the diffraction spectrum of MAO coating on AD1 alloy during oxidation with a current density of 20 A/dm<sup>2</sup> for 60 min (electrolyte: 1 g/l KOH + 6 g/l Na<sub>2</sub>SiO<sub>3</sub>). Shooting in K $\alpha$ -Cu.

Similar studies have been conducted on the aluminum alloy AB. It should be noted that this alloy can also be attributed to low-alloy aluminum alloys, but with a different composition of alloying elements compared to AD1 alloy and a higher level of alloying by basic elements (about 1 % in AB alloy, compared to less than 0.1 % in AD1 alloy).

As in previous studies, the first characteristic to be studied and analyzed in microarc oxidation is the thickness of the formed coatings and its dependence on the main technological parameters of electrolysis (process duration, current density, and electrolyte composition).

Dependences of the thickness of MAO coatings on the process duration are almost linear. For clarity, the kinetic parameters of the coating growth rate depending on the current density on the sample are conveniently presented in the form of graphs for the total growth rate and the growth rate of the working layer.

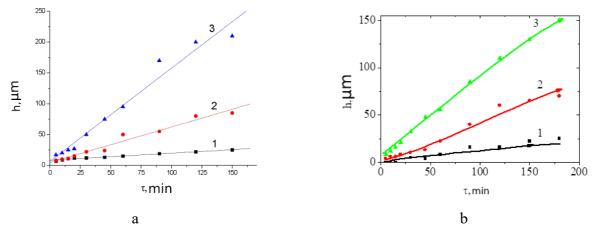
Fig. 9 shows the dependence of the total thickness of the coating formed in the electrolyte of  $1 \text{ g/l KOH} + 6 \text{ g/l Na}_2\text{SiO}_3$  on the AB alloy (Fig. 9, a) and the thickness of the working layer (Fig. 9, b) on the duration of oxidation at different current densities. It can be seen that the identified trends in increasing the thickness growth rate with increasing current density are also manifested in the oxidation of the AB alloy.

For the coatings obtained at different current densities in the electrolyte of a solution of  $1 \text{ g/l KOH} + 6 \text{ g/l Na}_2\text{SiO}_3$ , an X-ray phase analysis of the state of the coatings was performed. Fig. 10 shows the results of this study.

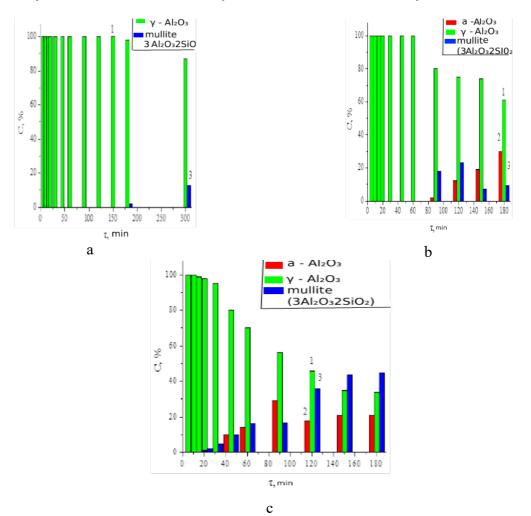
It can be seen that the phase composition of the coatings formed at a current density of 5 A/dm<sup>2</sup> up to almost the largest thicknesses consists of a single-phase modification of aluminum oxide -  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> (Fig. 10, a). At a higher current density and a duration of more than 90 min, a three-phase coating state (Fig. 10, b) is formed from  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, and mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>).

A similar three-phase state (but with a relatively shorter oxidation duration of 40 min and below) is manifested in the MAO coating on the AB alloy when it is oxidized with the highest current density of 50  $A/dm^2$  (Fig. 10, c).

At low current densities, the specific content of the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase increases in AB alloys (up to almost 100 %). In coatings formed at higher current densities, a decrease in the specific content of the mullite component is accompanied by an increase in the content of the hardest polymorphic modification of aluminum oxide, the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase (corundum).



**Fig. 9.** Dependence of the total coating thickness (a) and the thickness of the working layer (b) on the duration of processing of the aluminum alloy AB (electrolyte:  $1 \text{ g/l KOH} + 6 \text{ g/l Na}_2\text{SiO}_3$ ):  $1 - \text{current density 5 A/dm}^2$ ;  $2 - \text{current density 20 A/dm}^2$ ;  $3 - \text{current density 50 A/dm}^2$ .



**Fig. 10.** Dependence of the phase composition of the coating on the duration of oxidation of AB alloy in electrolyte 1 g/l KOH + 6 g/l Na<sub>2</sub>SiO<sub>3</sub> at different current densities:  $a - 5 \text{ A/dm}^2$ ;  $b - 20 \text{ A/dm}^2$ ;  $c - 50 \text{ A/dm}^2$ .  $1 - \gamma - \text{Al}_2\text{O}_3$ ;  $2 - \alpha - \text{Al}_2\text{O}_3$ ; 3 - mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ).

Thus, for different classes of materials, the aspect of the optimal use of certain technologies for modifying surface layers should be taken into account, where different approaches to ensuring their required properties are provided.

#### **3** Conclusion

The paper describes the features and results of the application of technologies for modifying the structures and properties of surfaces and the feasibility of using a particular method for materials of different classes.

The possibility of additional strengthening of the surface of U8A steel from a microhardness level of 7.2 GPa after its thermal hardening to almost the maximum possible level of 14.7 GPa using the ATFS method is shown. The microstructure of the cross-section of a specimen of prehardened U8A steel after additional thermofriction strengthening is presented, where the degree and nature of surface hardening are clearly visible. It is emphasized that in previous studies, consistently effective hardening of steels of various classes has been achieved, even up to the level of 22 GPa in 65 G steel.

The study of coatings on low-alloy aluminum alloys showed that the micro-arc oxidation method in alkaline-silicate electrolyte allows to obtain a coating thickness of up to 300 microns, a coating growth rate of ~ 2 microns/min, coating hardness from 10 to 20 GPa. Coatings have high adhesion to the substrate; they have a layered structure, the properties of coatings are determined by the properties of the base layer. The coating has a crystalline structure and consists of the following phases:  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>), the ratio between the phases depends on the electrolysis conditions. It has been established that phase formation begins with the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase, which in the process of further coating growth turns into the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> phase or interacts with silicon oxide and forms the mullite phase.

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