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Analysis Of Technological Operations For The Development Of Aluminum-Based High-Strength Nanostructured Materials (Coatings)

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Abstract: The development of aluminum-based high-strength nanostructured materials and coatings is essential for advanced surface engineering applications. Their functional properties strongly depend on the technological operations employed during fabrication. This study analyzes the key technological stages involved in producing aluminum-based nanostructured coatings, including substrate preparation, thin film deposition, anodization, and post-treatment processes. Particular attention is paid to the influence of processing parameters on the formation of nanoscale structures and columnar morphologies. The analysis shows that optimization of technological operations allows effective control of structural characteristics, leading to improved hardness, elastic modulus, and mechanical stability of the coatings. The results demonstrate the potential of optimized aluminum-based nanostructured coatings for use in high-performance engineering and protective applications.

Keywords: Nanostructured coatings, post-treatment processes, elastic modulus, tantalum oxide.

INTRODUCTION:

In recent years, in the field of modern materials science and engineering modification of surfaces, there has been a significant increase in interest in the development of solid nanostructural materials and coatings with an aluminum base. Aluminum and its alloys are widely used in aviation, automotive, energy, microelectronics and other high-tech industries due to their properties such as low density, high specific strength, good thermal and electrical conductivity, and stability to corrosion [1-6]. At the same time, the mechanical and operational properties of traditional aluminum materials do not fully satisfy some engineering requirements, which makes it necessary to strengthen them at the nanostructural level.

The sequence of technological operations used in the formation of aluminum-based nanostructural coatings and their interrelationships are one of the main factors determining the final structure and functional properties of the coating. In particular, the processes of preparation of a metal base, deposition of thin layers, anodizing and reanodizing, as well as the stages of heat and electrochemical processing, have a direct effect on the formation mechanisms of nanostructure. Through an in - depth analysis of each of these technological operations, it is possible to control the micro-and nanomorphology of the coating, the mechanical strength and the duration of its service life [7-11].

Experimental Studies

The results of investigations into the formation and modification processes of porous anodic aluminum oxide (AAO) matrices, as well as methods for the electrochemical oxidation of tantalum through AAO pores and the formation of columnar nanostructures within these pores, served as the basis for developing a preliminary technological route for producing nanostructured composite coatings with specified structural parameters and geometric dimensions [12-14]. These coatings are intended for strengthening

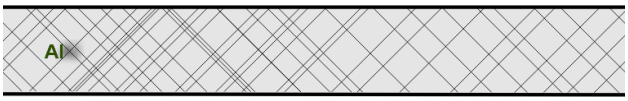
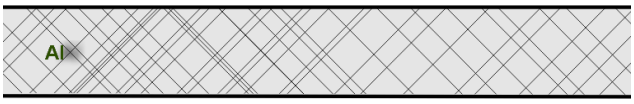
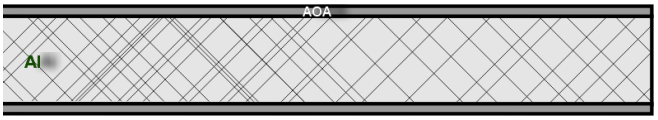
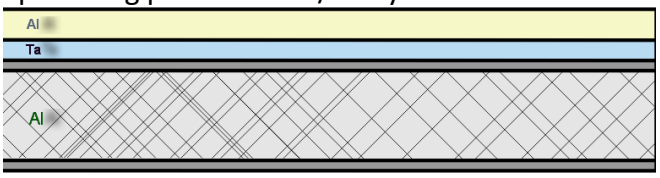
and protecting products made of aluminum and aluminum alloys against external influences [15]. The draft technology was developed and tested on aluminum rolled blanks. The schematic technological operations for producing the new reinforcing nanostructured coatings are presented in Table 1.

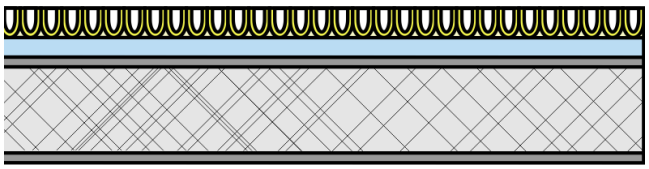
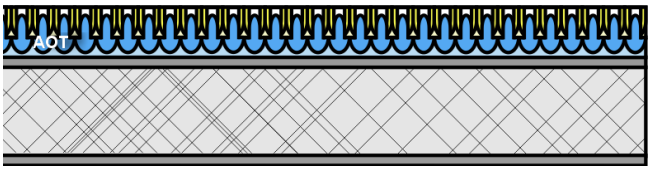
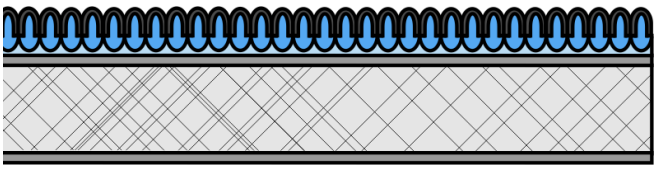
Initially, primary blanks with dimensions of 60 × 48 mm were stamped from aluminum rolled stock with a thickness of 2000 µm (99.95% purity). After multiple polishing steps, thermo-straightening was carried out at 350 °C under a pressure of approximately 10^7 Pa [16]. Subsequently, the blanks were cleaned in a chromium-containing solution (CrO_3:H_2SO_4 at a ratio of 1:100) at a temperature of 18–20 °C for 2–3 min to remove surface contaminants. The samples were then rinsed in distilled water and dried with hot air at 100 ± 5 °C for 5 min [17].

To remove micro-asymmetries from the aluminum surface, an electrochemical polishing operation was performed in an electrolyte consisting of 65% perchloric acid and 99.5% acetic acid in a 22%:78% ratio, at an electrolyte temperature of 7–9 °C. The process was conducted under continuous stirring at an applied voltage of 25–27 V and a current d-ensity of 250–300 mA/cm². The anodic treatment time ranged from 30 to 60 s [18-20]. After electrochemical polishing, the samples were thoroughly rinsed in running and distilled water, dried with compressed air, and finally dried in a thermostat at 160 °C for 20 min.

DISCUSSION

Table 1. Sequence of technological operations for the fabrication of aluminum-based high-strength nanostructured materials (coatings).

№	Experimental operation procedure and schematic diagram	Experimental procedure
1	Preparation of an Al-containing experimental sample 	The sample was chemically cleaned under a pressure of 10^7 Pa at a temperature of 350 °C in a chromium-containing solution. The cleaning process (CrO_3:H_2SO_4 in a 1:100 ratio) was carried out at a temperature of 18–20 °C for 2–3 min.
2	Chemical cleaning 	Electrochemical polishing (brightening) was performed using a solution of 65% perchloric acid and 99.5% acetic acid in a 22%:78% ratio at a temperature of 7–9 °C, under an applied voltage of 25–27 V and a current density of 250–300 mA/cm², for a duration of 30–60 s.
3	Formation of an adhesive protective layer 	The anodization process was carried out in a 0.5 M aqueous oxalic acid solution at a temperature of T = 14 ± 0.5 °C, to a depth of 0.5 µm, under a constant voltage of 50 V, with preliminary potential scanning at a rate of 10 V/s.
4	Sputtering process of Ta/Al layers 	Tantalum with a thickness of 0.5 ± 0.05 µm was deposited onto the sample surface by the magnetron sputtering method in the chamber of the “Oratoria-9” setup under a pressure of P = 900 Pa, at a temperature of T = 250 °C, with a

		sputtering current of I = 0.9 A and an accelerating voltage of U = 6 kV.
5	Anodization of Al 	The electrochemical anodization of aluminum was carried out in a 0.4 M oxalate solution at an applied voltage of E_a = 53 V and a current density of j = 10 mA/cm², at a temperature of T = 10–12 °C, until the current density decreased to 20 µA/cm².
6	Anodization and re-anodization of Ta 	Anodization was carried out in a 0.1 M citric acid solution by increasing the voltage at a rate of 1 V/s up to 220 V and maintaining the process for 18 min until the current density decreased to 20 µA/cm². The re-anodization process was performed in a 0.5 M H_3BO_3 + 0.05 M $Na_2B_4O_7$ electrolyte under an applied voltage of E = 420 V, with an anodization current density of j = 500 µA/cm². The voltage was increased at a rate of 2 V/s up to 420 V, and the process was maintained for 30 min until the current density decreased to 40 µA/cm².
7	Application of the PVDF planarization process to the AAO/ATO structure 	The selective removal of the AAO layer from the columnar structures in solution was carried out using an electrolyte containing chromium trioxide (20 g/L) and orthophosphoric acid (60 g/L), diluted with distilled water to a total volume of 1 L, at a temperature of 60 °C. PVDF was applied to the structure with a thickness of 0.2–0.45 µm. The spreading time was 6 min, while the film formation time was 30 s, at a rotation speed of 3200 rpm. Drying of the PVDF layer was performed at a temperature of T = 80–100 °C for 45 min.

The prepared sample blanks were mounted in special holders inside a vacuum chamber and heated to a temperature of 230 ± 20 °C. Subsequently, double-sided tantalum deposition was carried out using the magnetron sputtering method on the production line of the Oratorio-9M system. In the Oratoria-9 setup, a tantalum layer with a thickness of 0.5 ± 0.05 µm was deposited onto the working surface under a chamber pressure of P = 900 Pa, at a temperature of T = 250 °C, with a sputtering current of I = 0.9 A and an accelerating voltage of U = 6 kV. After that, an aluminum layer with a thickness of 1.5 ± 0.05 µm was

deposited onto the tantalum surface.

The deposited aluminum layer was anodized in a 0.4 M oxalic acid solution at an anodization voltage of E_a = 53 V, with an anodization current density not exceeding j = 10 mA/cm², and an electrolyte temperature of T = 10–12 °C. The formation of tantalum columns was achieved by high-voltage re-anodization through the pores of the formed anodic aluminum oxide. The re-anodization process was carried out in a 0.5 M H_3BO_3 + 0.05 M $Na_2B_4O_7$ solution at an applied voltage of E = 420 V and an anodization current density not exceeding j = 500 μA/cm².

After electrochemical anodization, the porous AAO layer was selectively removed from the columnar structures using a solution containing chromium trioxide (20 g/L) and orthophosphoric acid (60 g/L), diluted with distilled water to a total volume of 1 L, at a temperature of 60 °C for 20 min. As a result of this operation, a planar AAO/ATO structure was formed. The total thickness of the coating was approximately 1.2 μm.

CONCLUSION

All the described methods are implemented using specialized equipment, which makes their practical realization relatively simple. This approach enables controlled single-stage or multistage formation of nanostructured coatings. The experimental procedures carried out using this method comply with environmental requirements. In addition to being cost-effective, the proposed approach allows the fabrication of materials with high wear resistance, as well as enhanced thermal and corrosion resistance.

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