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Development of biocompatible coatings for orthopedic joint implants

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This article presents both theoretical and experimental approaches to the development of biocompatible coatings based on hydroxyapatite modified with titanium dioxide for orthopedic implants made from Ti-13Nb-13Zr titanium alloy. The primary objective was to enhance the adhesion, mechanical strength and antibacterial properties of the coatings by employing a combined technique: micro-arc oxidation followed by gas-thermal spraying. The influence of electrolyte composition and micro-arc oxidation parameters on the coating's morphology, surface roughness and adhesion strength were systematically investigated. The highest values of hardness and adhesion were achieved using electrolyte containing Na_2SiO_3, NaOH and $Na_2S_2O_3$ in conjunction with detonation sputtering method. Morphological and elemental analyses confirmed the density, uniform elemental distribution and minimal porosity of these coatings. Mechanical stability was verified through Rockwell B scale and Martens tests. The proposed dual-step surface treatment strategy offers a promising route for tailoring implant surfaces with multifunctional properties. The obtained results demonstrate that the proposed method can significantly improve the durability and performance of orthopedic implants by producing biocompatible, corrosion-resistant, and mechanically robust coatings.

Key words: Biocompatible coatings, micro-arc oxidation, surface modification, mechanical properties, surface roughness, coating adhesion.
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1 Introduction

Total knee and hip arthroplasty are some of the most successful and common surgical procedures. Implants are made of polyetheretherketone, UHMWPE, cobalt and titanium alloys, and ceramics that mimic the natural joint and are biocompatible [1,2]. Titanium alloys are widely used [3-8], but their implants do not always provide effective osseointegration. To improve bioactivity, a porous layer is created or porous structures are developed [9].

The application of hydroxyapatite (HAp) coating was a solution to this problem; however, due to the brittleness of HAp, the coating can peel off, leading to metal ion release, infections, bleeding, and bacterial growth [10-12]. In mobile implants (e.g., hip implants), HAp particles cause fretting corrosion. Another problem is bacterial infection leading to implant failure [13]. Pure HAp does not prevent bacterial adhesion because organic substances are easily absorbed by its surface, creating favourable conditions for their growth [14].

Thus, reinforcing nanoparticles such as Al O_2, TiO_2, etc. can be added to HAp to solve this problem. The development of HAp-based composites with TiO_2, yttrium-stabilized zirconium dioxide, Al O_2, nanodiamond, magnesium or natural fiber is actively investigated to improve the mechanical properties of HAp [15-18]. Several methods for antibacterial coatings have been proposed [19, 20], including the addition of Ag ions [21], antibiotic peptides [22] and organic compounds [23]. In [24], the concept of a multifunctional coating with both the necessary functionality and biocompatibility was proposed.

From the literature analysis, it can be seen that among the various enhancers, TiO_2 has been most widely used to improve the adhesion and cohesive strength, anti-wear properties, hardness and biological performance of the coating [25, 26]. Moreover, it has been reported that TiO_2 reinforcement in HA significantly improved the corrosion resistance [27, 28].

HA-TiO_2 based coatings are usually produced by plasma spraying (PS). In fact, PS is the most conventional commercial method and is approved for the fabrication of hydroxyapatite coatings by the US Food and Drug Administration (FDA) [29]. However, plasma spraying of HAp is very sensitive to crystalline conditions. Excessive heating of the plasma leads to low crystallinity of HAp and disintegration into more soluble phases (β-TCP) [30,31]. An amorphous phase is also observed in plasma spraying of HAp. This can lead to a decrease in coating strength and jeopardizes the long-term stability of the coating [32].

In the last decade, various authors have proposed to fabricate HAp coatings using alternative methods. Currently, methods that are free of the disadvantages of plasma spraying, such as vacuum plasma spraying [33], cold spraying [34], micro-arc oxidation (MAO) [35], high-velocity oxygen-fuel (HVOF) spraying [36], detonation spraying [37, 38], etc., are being intensively studied.

Thus, HVOF, detonation spraying and MAO methods represent a good prospect for obtaining HA-TiO_2 based coatings on titanium alloy. The above methods are further developed by scientists by optimizing the deposition parameters of the coatings and by combining several methods, etc. Thus, the properties of the coatings are related to the deposition parameters, which need to be optimized to control and analyze the effect of deposition parameters on the coating characteristics.

The role of pretreatment in enhancing the performance characteristics of biocomposite coatings was identified in this phase of work. HAp-TiO_2 biocomposite coatings were obtained using a combined technique of MAO and subsequent gas-thermal spraying detonation spraying, HVOF and cold spraying of calcium-phosphate ceramics based on hydroxyapatite. Comprehensive studies of theoretical and practical aspects of the formation of biocomposite coatings based on various combinations of nonmetallic structures have been carried out.

Based on the above, our aim is to study theoretical and practical aspects of formation of biocomposite coatings based on hydroxyapatite with TiO_2 addition, as well as to analyse methods of their deposition. Special attention is paid to combined technologies, including micro-arc oxidation and subsequent gas-thermal spraying, in order to optimise deposition parameters and improve the performance characteristics of the coatings.

2 Materials and methods

In accordance with the set objectives, Ti-13Nb-13Zr alloys were chosen as the object of the study. The choice of the research material is justified by the fact that Ti-13Nb-13Zr alloys are used for the manufacture of medical implants, such as joints and bone fixators, due to their biocompatibility and corrosion resistance, operating in temperature ranges from -50°C to 300°C.

The influence of microarc oxidation on the formation of calcium-phosphate coatings during gas-thermal spraying has been investigated. Samples from titanium alloy Ti13Nb13Zr (⌀ 25 mm) underwent mechanical treatment, cleaning and ultrasonic treatment (20-80 kHz) in distilled water or cleaning solution, providing effective removal of contaminants without damage to the material.

Electrolyte compositions were individually selected for each of the material samples prior to MAO coating:

1) 20 g/L Na_2SiO_3, 10 g/L KOH;
2) 20 g/L H_3BO_3, 10 g/L KOH;
3) 20 g/L Na_2SiO_3, 10 g/L NaOH,10 g/L $Na_2S_2O_3$.

The parameters of the microarc oxidation process were set within the following limits: pulse duration – 100-500 μs, pulse frequency – 50-100 Hz, current density – 0.13-0.35 A/cm², process duration – 5-20 minutes, electrical voltage – 50-100 V. These parameters, as well as electrolyte compositions and process mode were specially developed for the formation of calcium-phosphate coating with optimal properties. The MAO device includes a programmable AC source Guintek “APS-77300”, a galvanic cooling bath, a set of electrodes, software for controlling and monitoring the electrophysical parameters of the process, as well as a digital oscilloscope that provides registration of kinetic dependencies during treatment. The formation of calcium-phosphate coating was carried out in alternating mode.

The schematic diagram of the device for carrying out MAO is presented in Figure 1.

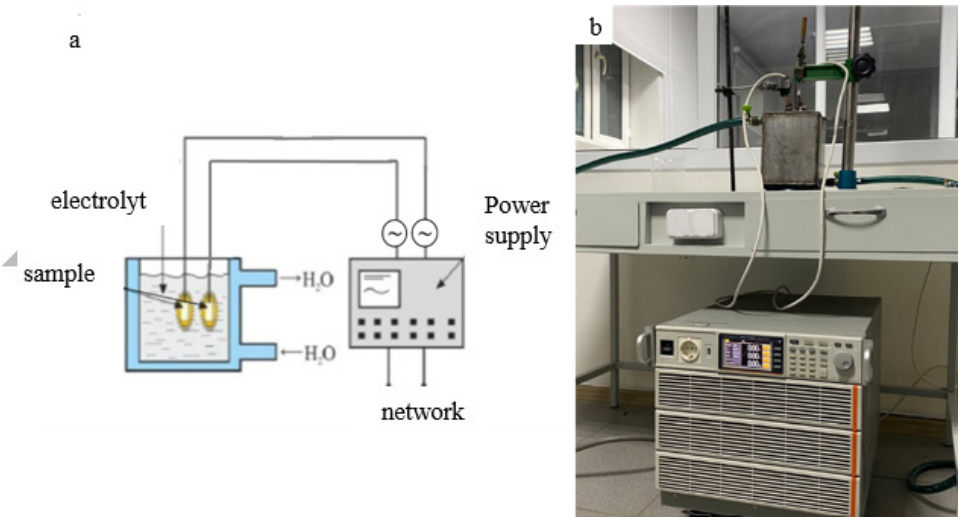

Figure 1 – Schematic diagram of the device (a) and experimental setup with power supply (b) for microarc oxidation.

The surface microstructure and cross-sectional morphology of the coatings were studied by scanning electron microscopy (SEM) on a Vega 4 (Tescan, Czech Republic). A tungsten filament was used as a cathode. To study the elemental composition of the obtained coatings, the Xplore 30 energy dispersive analysis SEM attachment (Oxford Instruments) was used.

Rockwell B hardness (HRB) measurements were made using a 1/16 inch (1.5875 mm) diameter steel ball under a 100 kgf (980.7 N) load. A preload of 10 kgf ensured stable contact with the specimen surface. The depth of the indentation was recorded automatically by the device TR 5006M, and the results were determined according to GOST 9013. The method was used to evaluate soft metals and titanium alloys [39] .

Hardness and modulus of elasticity of the samples were measured according to the Martens method (ASTM E 2546) on a hardness tester FISCHERSCOPE HM2000S ("Fischerscope", Germany), at indenter load F = 245.2mN and dwell time 20s. Surface roughness was determined according to GOST 25142-82 using a profilometer model 130.

Adhesion was measured using a tensile testing machine according to ASTM C633 standard specification. A two-component epoxy system (Adhesive 2214) was used as adhesive and the pulley diameter was 25 mm. After bonding, the pulleys were incubated at 24 °C and 50% relative humidity for 24 hours until the adhesive was fully cured.

To improve adhesion, a preliminary sandblasting with dry corundum was carried out (at air pressure of 0.3-0.6 MPa, the distance from the nozzle shear of the jet-abrasive gun to the treated surface is 80-200 mm).

3 Results and discussion

Surface analysis of the samples revealed differences in the morphology and porosity of the coatings depending on the electrolyte composition (Figure 2). The obtained coatings have rounded porosity, which indicates favorable conditions for osteointegration. The average pore size of MAO coatings of titanium alloy Ti13Nb13Zr samples is 12-23 μm, and the coating thickness varies within 3-8 μm.

Hydroxyapatite powder with a dispersity of 20-60 μm was used as the spraying material (Figure 3).

Figure 4 shows the values of surface roughness R_a depending on the modes of microarc oxidation (MAO) and the applied spraying method: detonation, cold and HVOF spraying. The first mode of MAO shows moderate roughness (R_a 1.8-2.0 μm) for all spraying methods. In the second mode, a maximum increase in roughness is observed, reaching R_a 2.8 μm when HVOF is used, which is due to the enhancement of the porous surface structure that promotes coating adhesion. In the third mode, the roughness decreases, especially in cold spraying (R_a about 1.8 μm), which may be related to the densification effect of the coating.

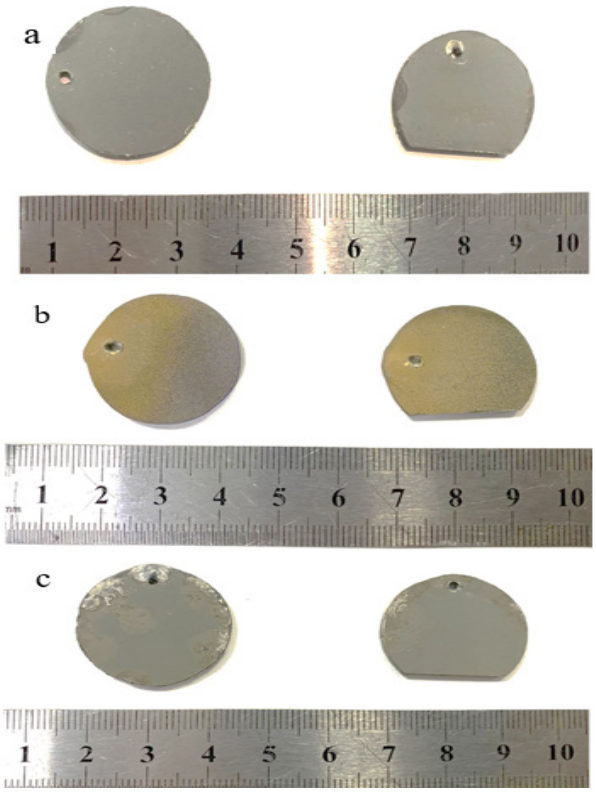

Figure 2 – Samples of titanium alloys after microarc oxidation:
Electrolyte 1: 20 g/L Na_2SiO_3, 10 g/L KOH; b) Electrolyte 2: 20 g/L H_3BO_3, 10 g/L KOH;
c) Electrolyte 3: 20 g/L Na_2SiO_3, 10 g/L NaOH, 10 g/L $Na_2S_2O_3$.

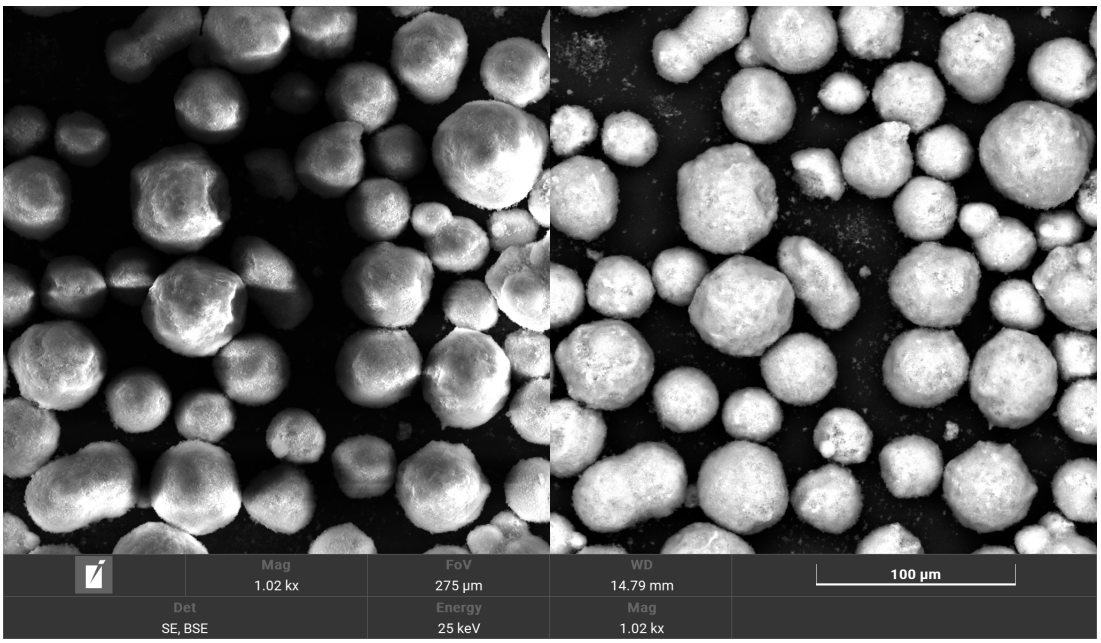

Figure 3 – SEM images of hydroxyapatite powder
with particle sizes in the range of 20-60 µm.

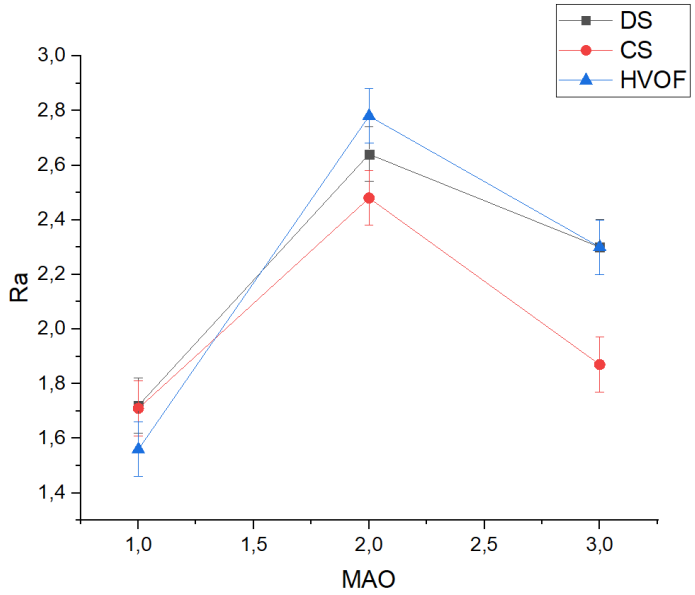

Figure 4 – Dependence of surface roughness parameter on microarc oxidation modes for different spraying methods: detonation (DS), cold (CS) and HVOF spraying.

Based on the presented data, the second electrolyte (20 g/L H_3BO_3, 10 g/L KOH) provides the highest surface roughness (R_a) after microarc oxidation, which is attributed to the synergistic effect of boric acid and potassium hydroxide. Boric acid (H_3BO_3) promotes active pore formation and microstructural rough texture, whereas potassium hydroxide (KOH) accelerates the oxide layer formation process by increasing the ionic conductivity of the electrolyte. Compared to the other compositions, the first electrolyte (Na_2SiO_3, KOH) exhibits lower roughness due to the formation of a dense structure, while the third electrolyte (Na_2SiO_3, NaOH, $Na_2S_2O_3$) creates a layered surface with less porosity.

Adhesion strength plays a key role in evaluating the performance of coatings, as it determines their resistance to mechanical loads and durability under service conditions. The results of adhesion tests for these samples are presented in Figure 5. The analysis has shown that the choice of electrolyte composition and micro arc oxidation (MAO) modes has a significant influence on the substrate surface characteristics and adhesion of coatings applied by different spraying methods. Electrolytes based on sodium silicate (Na_2SiO_3) in combination with potassium hydroxide (KOH) or sodium hydroxide (NaOH) provide the formation of porous and rough layers that are optimal for adhesion in high velocity HVOF and detonation spraying. Boric acid electrolytes (H_3BO_3) promote smooth and dense layers, which improves adhesion in cold spraying, where mechanical bonding is limited by low process temperatures. Maximum adhesion of the coatings is observed at an optimal combination of high porosity and surface roughness, which is achieved by using electrolytes with $Na_2S_2S_2O_3$ addition and MAO modes that promote active growth of the oxide layer.

Figure 6 shows the results of analysis of coatings deposited by different methods on a substrate treated with a solution of Na_2SiO_3 (10 g/L) and KOH. Figure 6a of the presented micrograph of the coating obtained by high-speed gas thermal spraying method shows the structure, the zone (~0-16 μm) calcium (Ca) and phosphorus (P) concentration reaches a maximum at a depth of ~12-16 μm, indicating the formation of calcium-phosphate compounds. In Figure 6b, it can be seen that the coating is virtually absent, as evidenced by the following facts: the distribution of elements such as calcium (Ca) and silicon (Si) remains almost unchanged and uniform, indicating their association with the substrate material rather than the spraying layer (Figure 6c). The hydroxyapatite coating obtained by the detonation method has a thickness of ~30 μm and is characterized by a stable structure and good adhesion to the substrate. The presence of key elements (Ca, P, O) confirms the successful formation of the functional layer, although porosity may affect the mechanical properties of the coating.

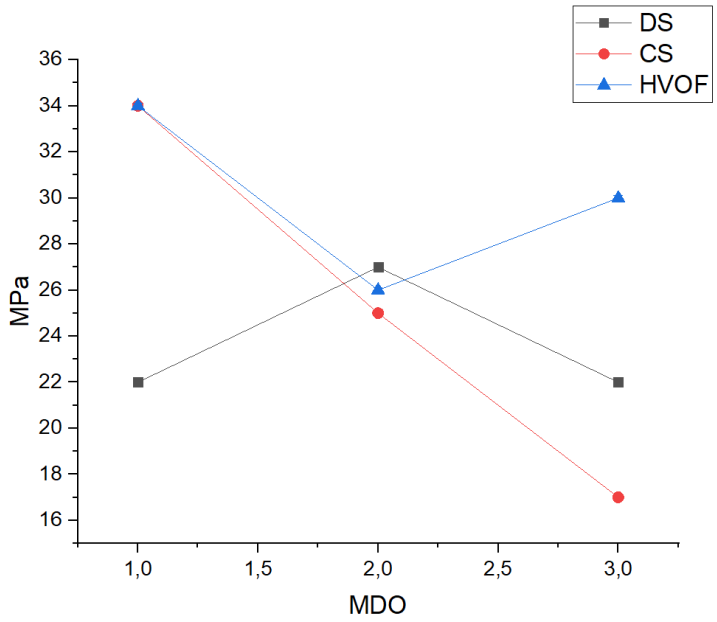

Figure 5 – Dependence of coating adhesion strength on microarc oxidation modes for different spraying methods: detonation (DS), cold (CS) and HVOF spraying.

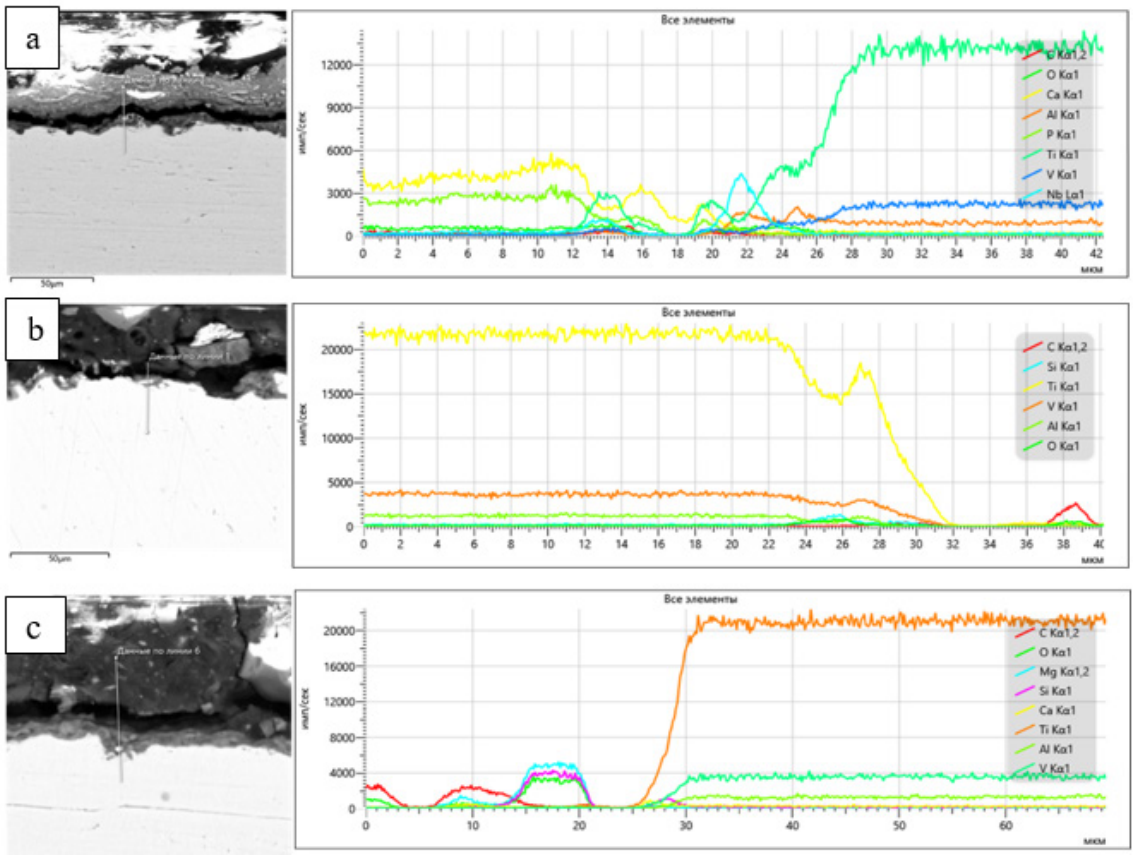

Figure 6 – Morphology and distribution of elements in coatings obtained by different methods: (a) HVOF, (b) cold spraying, (c) detonation spraying.

Figure 7 shows the analysis results of coatings deposited on a substrate with a pre-formed MAO layer in a solution of 20 g/L H_3BO_3 and 10 g/L KOH using three methods: (a) high-speed gas-thermal spraying, (b) cold spraying, and (c) detonation spraying

High-speed gas-thermal spraying formed a dense coating ~18 μm thick with a uniform distribution of calcium and phosphorus (Figure 7a), and a high oxygen content indicating a protective oxide layer. In Figure 7b, it can be seen that the distribution of hydroxyapatite is heterogeneous as evidenced by a peak of calcium (Ca) at ~18-22 μm depth and a rapid decrease in its concentration indicating localized deposition of material. The coating has a porous structure and a maximum thickness of about ~18 μm, beyond which the titanium (Ti) substrate begins, indicating an insufficient thickness of the functional layer. The low oxygen (O) concentration and the absence of significant phosphorus (P) content indicate poor formation of the hydroxyapatite layer.

Figure 7c shows that the coating is characterized by a dense structure and thickness up to ~30 μm, after which the titanium substrate begins, confirmed by a sharp increase in the concentration of titanium (Ti). The main component of hydroxyapatite, calcium (Ca), is uniformly distributed in the working layer (~10-30 μm), but the concentration of phosphorus (P), characteristic of hydroxyapatite, is either too low or not detected in the spectrum. Oxygen (O) is detected in significant amounts, indicating the presence of oxide phases, while magnesium (Mg) and silicon (Si) are present in trace amounts. Despite the high density and minimal porosity of the coating, the insufficiently uniform distribution of hydroxyapatite may limit the biocompatibility and protective properties of the coating, requiring further optimization of the spraying process.

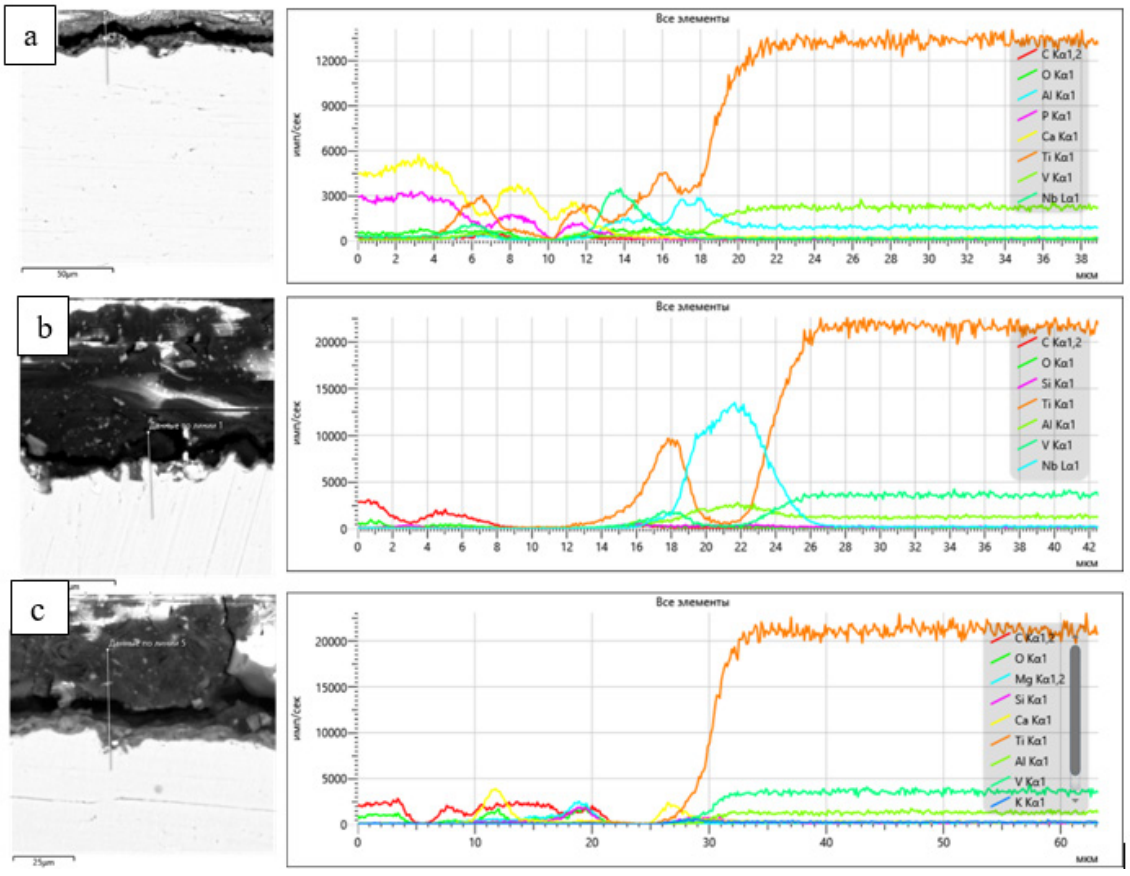

Figure 7 – Morphology and distribution of elements in coatings obtained by different methods: (a) HVOF, (b) cold spraying, (c) detonation spraying.

Figure 8 shows the microstructures and elemental analysis results of coatings deposited on substrates with pre-formed MAO layer in a solution of 20 g/L Na_2SiO_3, 10 g/L NaOH and 10 g/L $Na_2S_2O_3$, using three methods: high-speed gas-thermal spraying (a), cold spraying (b) and detonation spraying (c). The coating obtained by high-speed gas-thermal spraying method has a thickness up to ~12-16 μm, a dense structure and a high concentration of oxygen (O) and silicon (Si), which provides its protective properties. Cold spraying forms a coating up to ~28 μm thick, but its structure is porous and the distribution of calcium (Ca) and silicon (Si) is non-uniform, which reduces the effectiveness of the coating. Detonation spraying produces a ~30μm thick coating with a uniform distribution of calcium (Ca), magnesium (Mg) and oxygen (O), providing density and minimal porosity, making this method the most effective for creating protective layers.

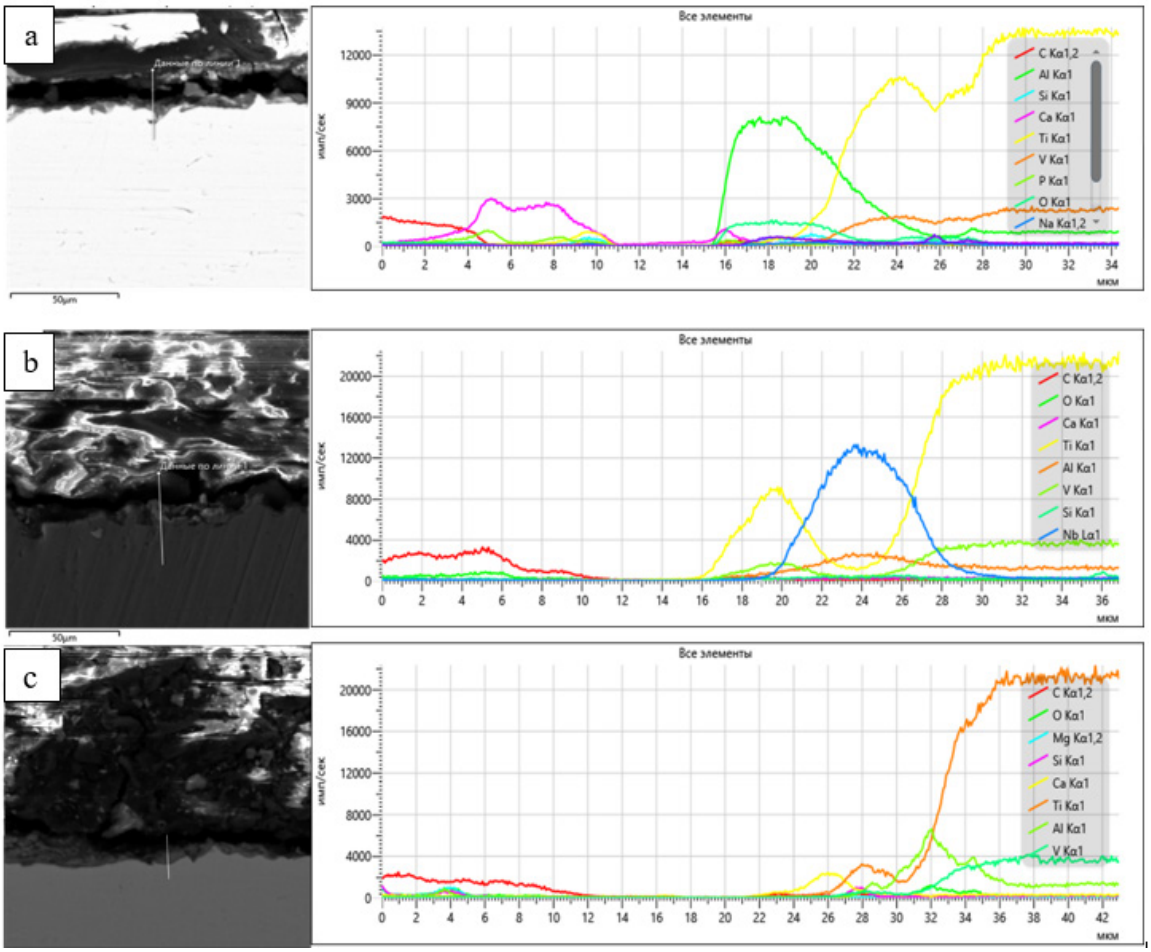

Figure 8 – Morphology and distribution of elements in coatings obtained by different methods: (a) HVOF, (b) cold spraying, (c) detonation spraying.

The study was complemented by measurements of the hardness of the top layer of the coatings on the Rockwell B scale (HRB). The results in Figure 9 show that the detonation sprayed coatings have the highest HRB values due to the dense structure and minimal porosity of the top layer. High-speed gas-thermal spraying shows average HRB hardness values, confirming the balance of mechanical characteristics and uniformity of the coating structure. Cold sprayed coatings have minimal HRB hardness due to high porosity and insufficient layer density. Thus, detonation spraying provides the highest hardness and strength values among the studied methods.

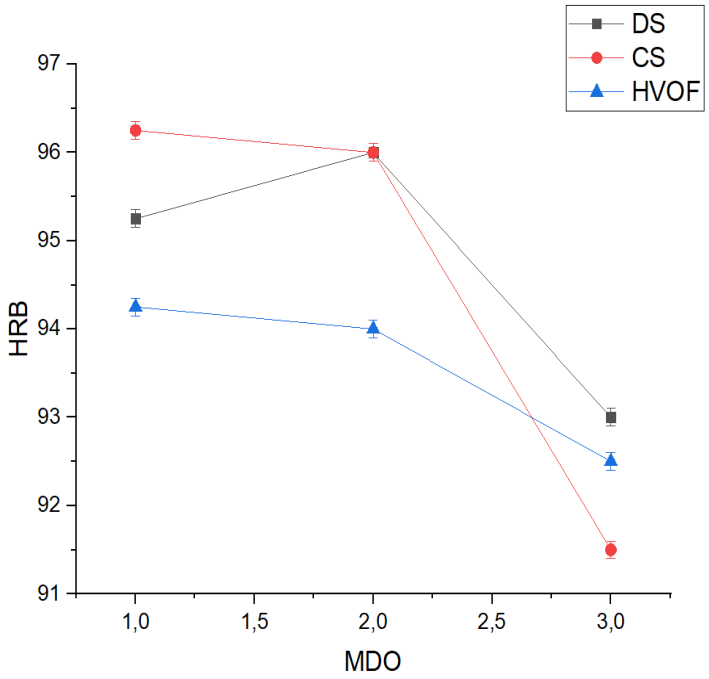

Figure 9 – Results of microhardness study of biocomposite coatings: detonation (DS), cold (CS) and HVOF spraying.

4 Conclusion

An effective technology for forming biocompatible coatings based on hydroxyapatite modified with titanium dioxide (HAp-TiO_2) for orthopaedic implants made of titanium alloy Ti-13Nb-13Zr has been developed and experimentally validated. The combined approach including micro-arc oxidation and subsequent gas-thermal spraying (HVOF, detonation and cold spraying) significantly improved adhesion, density and mechanical characteristics of the coatings.

It was found that both the parameters of micro-arc oxidation and the composition of the electrolyte exert a strong influence on the substrate's morphology and roughness, creating favorable conditions for robust adhesion of the coating layer. The highest values of roughness (up to R_a 2.8 μm) and adhesion strength are achieved using a boron-containing electrolyte and the method of high-speed gas-thermal spraying. The best mechanical characteristics, including maximum hardness and minimum porosity, were recorded for the coatings obtained by detonation spraying.

Overall, the optimal combination of the parameters of micro-arc oxidation and coating method allows to form strong, uniform and functional biocompatible layers capable to increase the reliability and service life of orthopaedic implants.

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