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Surface Topography of Composite Coatings Based on Ti-Al_3

Abstract: currently, the development of biomaterials that have the required characteristics for various tissues is one of the main problems in medicine and engineering. Obtaining calcium-phosphate coatings on metal surfaces is one of the urgent problems in materials science. Nevertheless, there is no unambiguous data on what physicochemical properties (phase and elemental composition, structure, crystallinity, roughness, solubility, etc.) should have an implant surface that ensures its osseointegration. In this work, to obtain calcium-phosphate coatings, we used the method of detonation-gas spraying of powder materials on a Grade2 titanium base. A $TiAl_3$ mixture with the addition of calcium hydroxyapatite was used as powder materials. The mass percentage of $TiAl_3$ in the mixture was 40%, 55%, 65%, 80%. The topography of the coating surface was determined by computer three-dimensional modeling based on data obtained using a Zygo New View 7300 interferometer-profilometer. It was found that with an increase in the intermetallic content in the $TiAl_3$ mechanocomposite with hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), the layer roughness of the surfacing of composite materials increases from Ra = 2.628 μm to Ra = 3.490 μm. At the same time, the roughness of the layer has an important role in the growth of bone tissue. Comparative analysis of the dependence of bone tissue growth on the roughness of coatings showed that layers obtained by detonation-gas spraying based on hydroxyapatite have a higher efficiency of about 100% of bone tissue growth at roughness values Ra = 2–4 μm.

Keywords: calcium phosphate coatings, bioactive coatings, implant, detonation gas spraying, roughness, the surface topography of coatings, powder materials.

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Introduction. In modern medical practice, titanium or titanium alloy implants are widely used to replace damaged or defective areas of bone tissue. But the use of implants with a significant difference in the physicochemical and mechanical properties of bone tissue and alloy causes active rejection in the human body and, as a result, further complications in treatment. The skeletal system of a living organism is formed and maintained as a result of complex biochemical reactions. To reduce the negative influence of such factors, it is necessary to create a transition zone between the implant and the bone, which can have a strong bond with the implant material, as well as a macro and microstructure acceptable to the body. Such a zone can be obtained in the form of a coating, which has a developed morphology, porosity for more effective engraftment of implants. Currently, there are several methods for obtaining such coatings. Thus, the method of plasma electrolytic oxidation makes it possible to obtain coatings on titanium alloys characterized by high physical and mechanical properties [1, 2]. The wear resistance of PEO coatings can be increased by a factor of 15 in comparison with an uncoated sample [1]. In this case, the porosity of the coatings can be different depending on the composition of the electrolyte solutions and be 6.2% – 12.2% [2]. Various materials can be introduced into the pores of the coating to increase its biocompatibility.

The method of magnetron sputtering is widely used to obtain biocompatible coatings on implants [3-6]. By applying various modes of magnetron sputtering, one can influence the structure and chemical composition of the surfaces of titanium and its alloys used for intraosseous implantation [6]. It was found that an increase in the sputtering power (from 200 to 300 W) leads to a significant change in the structure, which is accompanied by a change in the grain size and, as a result, in the surface roughness. Magnetron treatment of a pure titanium product with chemically pure titanium makes it possible to create a nanostructured surface connected to the substrate at the atomic level. The morphology of this surface at the nanoscale changes depending on the radiation power [6].

Surface modification of titanium alloys using biocompatible hydroxyapatite (HAP) coatings is of great interest. Sol-gel is a potential method for the precipitation of biocompatible HAP [7-10]. With the help of sol-gel technology, homogeneous, homogeneous HAP coatings can be obtained. In this case, an important role is played by the preliminary treatment of the titanium substrate surface and the optimization of the sol-gel processing parameters [7]. Also, the sol-gel technique makes it possible to synthesize organic-inorganic hybrid systems consisting of a silica matrix [8]. The studies carried out in this direction suggest that hybrid coatings can be used to modify the surface of titanium implants to improve the process of their osseointegration.

The proposed technologies and methods for creating biocompatible coatings for dental implants do not always fully meet modern medical requirements, and therefore, there is a search for new technological solutions for the formation of a biocompatible rough surface on dental implants, which ensures reliable integration of the implant with bone tissue. As a result of the approximation of the phase-structural state and the properties of the obtained coatings on implants to the parameters of the bone tissue, it is possible to achieve improved compatibility between them. When forming biocompatible coatings, special attention is paid to the creation of a certain relief (roughness) on the surface of the implant, which has a positive effect on osseointegration. So, in the world of a dental practice, implants with an SLA surface obtained by sandblasting and acid etching are used; implants with Osseotite surface obtained by double etching; implants with anodized TiUnite surface, etc.

Field of study. The main requirements for biocoatings are chemical and phase stability, high adhesive strength to a titanium substrate, porosity that promotes integration with bone tissue, and improved biocompatibility.

Studies have shown that hydroxyapatite coated titanium implants, compared to pure titanium implants, promote improved bone growth. In addition, biocompatible coatings on the titanium implant surface have a significant effect on the anchorage of loaded implants, both under stable and unstable conditions. Titanium implants with biocompatible coatings increase the degree of osseointegration. Such coatings prevent the penetration of titanium ions into the living tissues of the human body surrounding the implant. Biocompatible coatings applied to the metal implant, including those based on calcium hydroxyapatite, ensure reliable and fast filling of bone fractures. As a result, the area of contact with living bone tissue increases to almost 100%. The bio-coated implant is covered with dense bone tissue, preventing micromovements and stresses at the bone/implant interface.

Data and methodology. For the deposition of calcium-phosphate coatings on a titanium base, detonation-gas spraying (DGN) unit for powder materials "Katun-M" was used [11, 12].

Installation DGN "Katun-M", consists of the following parts (Figure 1) and operates in a pulse mode. The signals are generated by the control unit - 5. Each cycle begins with the supply of a combustible mixture of propane-butane oxygen in certain proportions for a specified time interval into the detonation chamber - 2. The explosive mixture is diluted with nitrogen or air to reduce the heating temperature of the particles of the sprayed material. The sprayed powder enters from the dispenser - 9, which is fixed on one of the holes - 11, into the barrel - 10, after loading the sprayed powder, a combustible mixture of candles is ignited - 8, while synchronization occurs with the digital prechamber - 15, which receives a signal from multichannel optical attachment, the signal from the attachment is fed to a computer via fiber-optic cables for further processing of the flow characteristics.

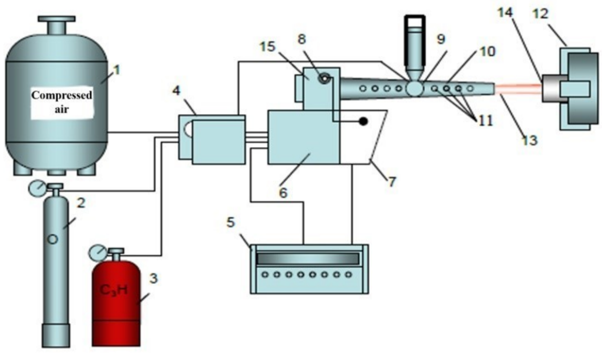

Рисунок 1 – Installation for detonation gas spraying "Katun - M": 1 – receiver, 2 – oxygen, 3 – propane, 4 - gas distribution system, 5 - control unit, 6 - injection mixer, 7 - electronic ignition unit, 8 - spark plug, 9 – dispenser, 10 – trunk, 11 - holes for installing the dispenser, 12 – manipulator, 13 - gas-dispersed flow, 14 – substrate, 15 – prechamber

When spraying pure HAP, the roughness of such coatings is 3.5 μm , and the thickness is up to 20 μm . The coatings practically do not contain pores; single pores are observed, which are formed when particles are melted. Coatings uniform in thickness and phase composition are formed by spraying hydroxyapatite particles with a size in the range of 50–300 microns. The roughness of the coating is 5.5–6.5 microns, and the thickness reaches 100 microns. At the same time, the results of the study of the adhesive strength of coatings showed that it does not exceed 5 MPa, which is insufficient for their operation. Pretreatment of the titanium surface before coating deposition, including sandblasting and chemical etching, as in the case of microarc coatings, makes it possible to increase the adhesion strength of the coatings to the substrate up to 20 MPa. The Ca/P ratio = 1.67 and is close to the ratio in bone tissue. To increase the strength of the coating, powders from composite materials of the composition $TiAl_3$ (X%)+ГАП33, obtained by the method of mechanically activated treatment, were also used. HAP ($Ca_{10}(PO_4)_6(OH)_2$) is a solid inorganic substance. In nature, this substance is part of rocks (together with chlorine and fluorapatites) and makes up the bulk of the bones of animals and humans. There are stoichiometric HAP $Ca_{10}(PO_4)_6(OH)_2$ and non-stoichiometric.

$Ca_x(HPO)_x(PO_4)_{6-x}(OH)_{2-x}$ of variable composition [13]. Bone biomineral is nonstoichiometric [11, 13]. Some brands of synthesized HAP correspond to the stoichiometric form, which seems to be quite acceptable, based on the chemical formulas of both forms [13-15]. The nomenclature name for stoichiometric HAP is dihydroxide - tricalcium hexaorthophosphate [16]. In terms of chemical composition, this is a polyorthophosphate compound, which suggests the presence of properties like those of other calcium polyorthophosphates (low solubility, relatively high decomposition temperature). This is confirmed by some of the sources indicated below [11, 13, 17, 18].

Mechanical activation (MA) of the initial reaction mixtures $TiAl_3$ (%) + HAP was carried out in an AGO-2 planetary ball mill with water cooling. The volume of each of the two steel drums of the mill is 160 cm . The diameter of the balls is 8 mm, the weight of the balls in each drum is 200 grams, the weight of the sample is 10 g. Centrifugal acceleration of balls 400 and 600 m s^{-2} (40 and 60 g). To prevent oxidation during MA, drums with samples were evacuated and then filled with argon to a pressure of 0.3 MPa. After MA, the samples were unloaded from drums in a box with an argon atmosphere.

Pretreatment of the titanium surface before coating deposition, including sandblasting and chemical etching, improves the adhesion strength of the coatings to the substrate. The processing quality was determined visually by the degree of surface mattness.

Materials obtained using the technology of self-propagating high-temperature synthesis (SHS) can be classified as materials with a high range of strength and tribotechnical properties. The use of powders obtained by the SHS method makes it possible to synthesize composite materials [19].

Detonation gas spraying (DGN) was carried out on titanium plates Grade2 20×20 mm thick 3 mm, with a composition containing a mixture:

1 – $TiAl_3$ (40 wt %.) + HAP - SHS mechanocomposite;
2 – $TiAl_3$ (55 wt %)+ HAP - SHS mechanocomposite;
3 – $TiAl_3$ (65 wt %) + HAP - SHS mechanocomposite;
4 – $TiAl_3$ (80 wt %) + HAP - SHS mechanocomposite;

Surface topography was determined by 3D computer modeling based on data obtained using a Zygo New View 7300 interferometer-profilometer.

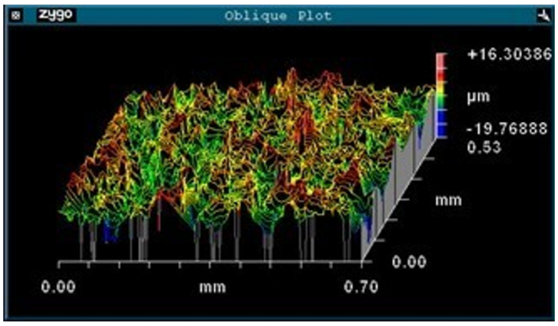

Рисунок 2 – Three-dimensional modeling of the surface of the intermetallic layer (40 wt%) and hydroxyapatite (60 wt%)

It can be seen from the diagram (Figure 3) that the average roughness of composite materials is based on a mixture of $TiAl_3$ (40%) + HAP Ra = 2,628 μm .

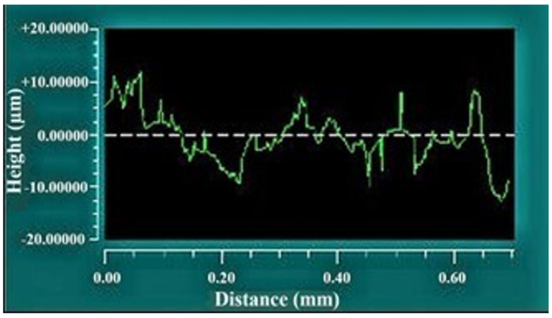

Рисунок 3 – Diagram of the distribution of the surface roughness of the surfacing layer of composite materials based on a mixture of $TiAl_3$ (40%) + HAP

It can be seen from the diagram (Figure 4) that the average roughness of composite materials is based on a mixture of $TiAl_3$ (40%) + HAP Ra = 3,022 μm .

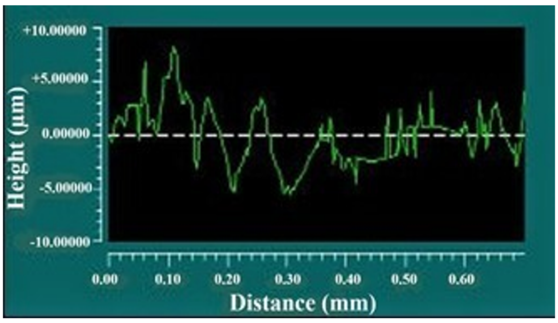

Рисунок 4 – Diagram of the distribution of surface roughness of a layer of the surfacing of composite materials based on a mixture of $TiAl_3$ (55%) + HAP

It can be seen from the diagram (Figure 5) that the average roughness of composite materials is based on a mixture of $TiAl_3$ (40%) + HAP Ra = 3,017 μm .

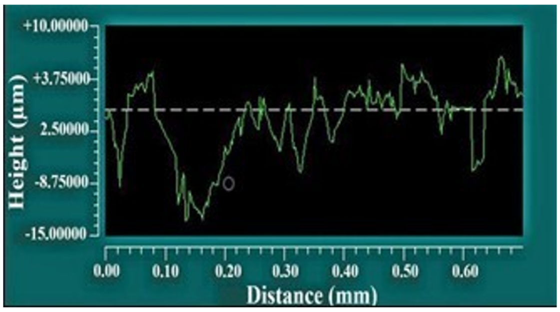

Рисунок 5 – Diagram of the distribution of surface roughness of a layer of the surfacing of composite materials based on a mixture of $TiAl_3$ (65%) + HAP

It can be seen from the diagram (Figure 6) that the average roughness of composite materials is based on a mixture of $TiAl_3$ (40%) + HAP Ra = 3,490 μm .

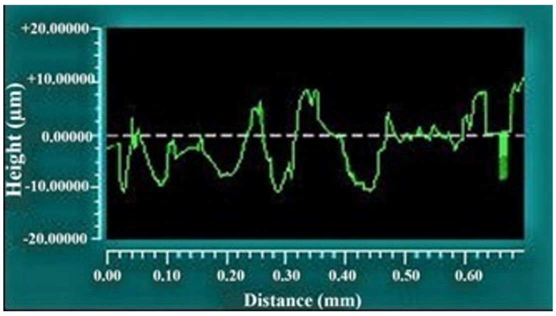

Рисунок 6 – Diagram of the distribution of surface roughness of a layer of the surfacing of composite materials based on a mixture of $TiAl_3$ (80%) + HAP

The roughness of such coatings is Ra = 2,628–3,490 microns. It can be seen from the diagram (Figure 7) that the roughness (Ra) increases with an increase in the percentage of intermetallic compounds in the mechanocomposite of composition $TiAl_3$ +ГАП ($Ca_{10}(PO_4)_6(OH)_2$).

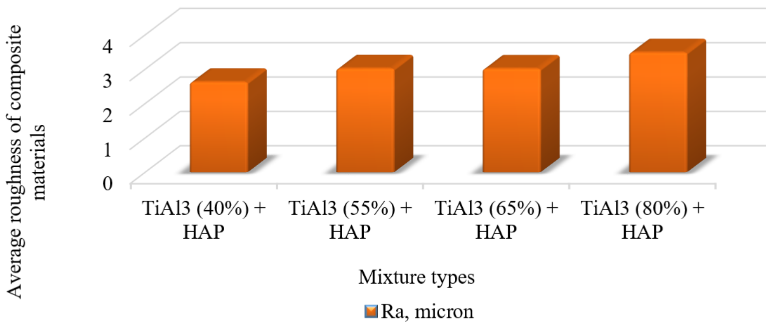

Рисунок 7 – Diagram of roughness (Ra) in a mechanocomposite of the composition $TiAl_3$ + HAP ($Ca_{10}(PO_4)_6(OH)_2$)

Results and a brief discussion. The biological properties of hydroxyapatite coatings were investigated in the test for ectopic bone formation [20]. The study of tissue response to subcutaneous implantation of titanium implants with calcium-phosphate coatings in mice showed that after 1.5 months of the experiment, no signs of inflammatory processes and infectious infections were detected, which determines their high biological compatibility.

Histological studies of the authors of [20] showed that on the sections of tissue plates, coarse fibrous bone tissue is revealed, in the lacunae of which elements of hematopoietic, adipose, and connective tissues can be located (Figure 8).

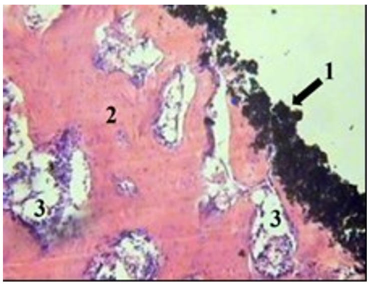

Рисунок 8 – Histological picture of tissue plates grown on the surface of X-ray amorphous calcium phosphate coating in the test of ectopic bone formation [20]: 1 - calcium phosphate substrate, 2 - bone tissue, 3 - elements of hematopoietic and adipose tissue. Hematoxylin-eosin staining

In studies of nanocrystalline calcium phosphate coatings containing β-TCP (β-tricalcium phosphate), it was shown that the growth of bone tissue is influenced by surface roughness [21]. Figure 9 shows that at Ra = 2–3 μm, the structure of tissue plates consisted mainly of bone tissue with holes filled with red bone marrow; the probability of bone tissue growth was about 67%. With an increase in Ra = 7.5 μm, connective and adipose tissue with an admixture of skeletal muscle bundles was revealed, but the probability of bone tissue growth was only 33%.

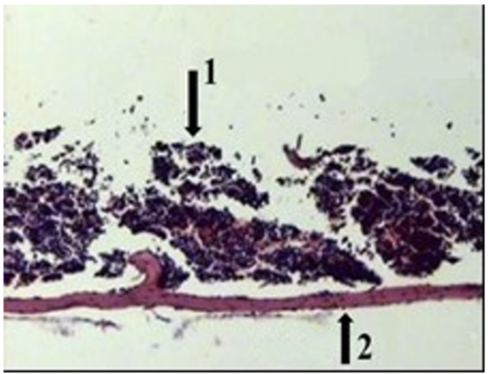

Рисунок 9 – Histological picture of tissue plates grown in the test of ectopic ontogenesis on nanocrystalline calcium phosphate coatings containing β-TCP [21]: 1 - bone marrow, 2 – bone

In comparison with the above coatings, the HAP-based layers obtained by the DGN method showed higher efficiency of about 100% of bone tissue growth in the test of ectopic osteogenesis at roughness values in the Ra = 2–4 μm region (Figure 10).

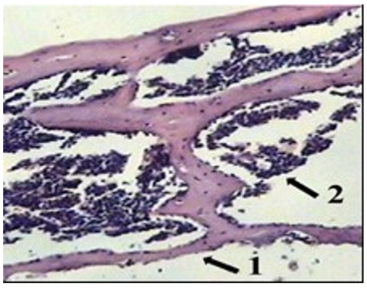

Рисунок 10 – Histological picture of tissue plates grown in the test of ectopic ontogenesis of HAP covering: 1 - bone, 2 - bone tissue

Conclusion. Calcium phosphate coatings (X-ray amorphous, nanocrystalline based on nonstoichiometric calcium phosphates, HA) are biocompatible, promote osseointegration with bone tissue, and can be used as biocoatings on implants for various purposes. These coatings, regardless of the method of obtaining them and their physicochemical properties, induce the growth of tissue plates with a 100% probability. At the same time, the effect of coating roughness on bone growth was revealed. The presence of HAP and TCP in the coating significantly increases the stability of the test of ectopic osteogenesis [20-23].

Thus, based on the data obtained, it can be assumed that, along with the phase composition and crystallinity of the coatings, the relief of the obtained layers is no less significant factor in the control of the bioengineering of the bone tissue. The obtained coatings are of interest for use in medical practice as biocoatings on titanium implants and can be used in the development of dental screw intraosseous implants.

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Ti-Al_3 негізіндегі композиттік жабындар бетінің топографиясы

Аннотация. Қазіргі уақытта әр түрлі ұлпаларға тән сипаттамалары бар биоматериалдардың дамуы медицина мен инженерияның негізгі мәселелерінің бірі болып табылады. Металдардың бетінде кальций-фосфат жабындарын жасау материалтану саласындағы өзекті міндеттердің бірі болып табылады. Дегенмен, импланттың остеоинтеграциясын қамтамасыз ететін импланттың беткі қабатының қандай физика-химиялық қасиеттері (фазалық және элементтік құрамы, құрылымы, кристалдылығы, кедір-бұдырлығы, ерігіштігі және т.б.) болу керектігі туралы нақты мәліметтер жоқ. Бұл жұмыста кальций-фосфат жабындарын алу үшін Grade2 титан негізіне ұнтақты материалдарды детонациялық-газды бүрку әдісі қолданылды. Ұнтақ материалдар ретінде кальций гидроксиапатиті қосылған $TiAl_3$ қоспасы қолданылды. Қоспадағы $TiAl_3$ -тің массалық пайызды мөлшері 40%, 55%, 65%, 80% құрайды. Жабын бетінің топографиясы Zygo New View 7300 интерферометр-профилометрінің көмегімен алынған мәліметтер негізінде компьютерлік үш өлшемді модельдеу арқылы анықталды. Гидроксиапатит ($Ca_{10}(PO_4)_6(OH)_2$) бар $TiAl_3$ құрамының механокомпозитіндегі интерметаллид мөлшерінің артуымен композиттік материалдардан жасалған беткі қабаттың кедір-бұдырлығы Ra=2,628 мкм-ден Ra= 3,490 мкм-ге дейін өсетіні анықталды. Бұл жағдайда қабаттың кедір-бұдырлығы сүйек ұлпасының өсуіне әсер етеді. Сүйек ұлпасының өсуі жабындардың кедір-бұдырлығына тәуелділігін салыстырмалы талдау, гидроксиапатит негізінде детонациялық–газды бүрку әдісімен алынған қабаттардың кедір-бұдырлық мәндері Ra=2-4 мкм болғанда, сүйек ұлпаларының өсуі шамамен 100% тиімділігін көрсетті.

Түйін сөздер: кальций-фосфатты жабындар, биоактивті жабындар, имплант, детонациялық-газды бүрку, кедір-бұдыр, жабын бетінің топографиясы, ұнтақ материалдар.

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Топография поверхности композиционных покрытий на основе Ti-Al_3

Аннотация. В настоящее время разработка биоматериалов, которые обладают необходимыми характеристиками для различных тканей, является одной из основных проблем медицины и инженерии. Получение кальций-фосфатных покрытий на поверхности металлов является одной из актуальных задач в материаловедении. Тем не менее нет однозначных данных о том, какими физико-химическими свойствами (фазовый и элементный составы, структура, кристалличность, шероховатость, растворимость и т.д.) должна обладать поверхность имплантата, обеспечивающая его остеоинтеграцию. В данной работе для получения кальций-фосфатных покрытий использовался метод детонационно-газового напыления порошковых материалов на титановую основу Grade2. В качестве порошковых материалов использовалась смесь $TiAl_3$ с добавлением гидроксиапатита кальция. Массовое процентное содержание $TiAl_3$ в смеси составили: 40%, 55%, 65%, 80%. Топография поверхности покрытий была определена путем компьютерного трехмерного моделирования на основе данных, полученных с помощью интерферометра-профилометра Zygo New View 7300. Установлено, что с увеличением содержания интерметаллида в механокомпозите состава $TiAl_3$ с гидроксиапатитом ($Ca_{10}(PO_4)_6(OH)_2$) шероховатость слоя наплавок композиционных материалов увеличивается от Ra = 2,628 мкм до Ra = 3,490 мкм. При этом шероховатость слоя оказывает немаловажное влияние на рост костной ткани. Сравнительный анализ зависимости роста костной ткани от шероховатости покрытий показал, что слои, полученные методом детонационно-газового напыления, на основе гидроксиапатита имеют большую эффективность, около 100%, роста костной ткани при значениях шероховатости Ra = 2–4 мкм.

Ключевые слова: кальций-фосфатные покрытия, биоактивные покрытия, имплант, детонационно-газовое напыление, шероховатость, топография поверхности покрытий, порошковые материалы.

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