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Obtaining functional-gradient Ti-HA coatings by detonation spraying

Functional-gradient titanium/hydroxyapatite (TiHA) coatings were obtained using detonation spraying technology to improve the structure and mechanical properties. To obtain functional-gradient coatings, pulsed energy sources are best suited, namely, detonation spraying, in which the energy of the explosion of gas mixtures is used as a source of pulsed action. By controlling the modes of detonation spraying, it is possible to vary the temperature and rate of coating deposition; accordingly, it is possible to obtain a certain structural-phase structure of the coatings. The structural-phase state and tribological properties of TiHA detonation coatings were investigated by modern materials science methods: X-ray phase analysis (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDX-mapping), profilometry and ball-disk wear-resistance test. The results showed that the coatings had a continuously gradient elemental composition across the cross-section of the coatings with no boundary between the elemental layers of the coatings. The amount of Ti gradually decreased and the amount of hydroxyapatite gradually increased in the direction from the substrate to the surface of the coatings, which allows to expand the possibilities of using TiHA-coatings for bone implants. Since the surface layer is composed of HA, the resulting functional-gradient coating demonstrates excellent biocompatibility and the ability to create new bone tissue. The excellent mechanical strength of the functionally graded coatings is ensured by the Ti phase.

Keywords: detonation spraying, functional gradient coatings, microstructure, phase composition, mechanical properties, hydroxyapatite, titanium, wear resistance, roughness.

Introduction

The development and production of biomaterials for bone replacement are one of the highly technological sectors of the economy, but the type and quality of implant materials and manufacturing technology currently available require further improvement. The “bottleneck” is not the medical technology associated with implant placement in the body, it is the engineering and materials science problems of producing an implant, with a specific chemical and phase composition and a specific morphological architecture [1]. One of the most promising solutions to these problems is the various combinations of metallic and non-metallic structures where the components gradually change in the materials. In particular, functional-gradient materials are new materials for both orthopaedic and dental applications. Currently, such materials are used for orthopaedic prostheses because functional-gradient materials can be adapted to reproduce the local properties of the original bone, which helps to minimise the effects of stress protection [2]. In stomatology, functional-gradient materials are used in dental crowns to imitate the connection of enamel and dentin of natural teeth and to avoid peeling and delamination between the layers [3, 4]. Various techniques such as PVD, CVD, and powder metallurgy have been used to produce functional-gradient materials [5–8]. In addition, functional-gradient materials have also been produced by gas-thermal spraying [9, 10]. Among the gas-thermal spraying methods, plasma spray technology has been used to produce various functional-gradient coatings suitable for biomedical applications. These include Ti/HA systems [11], HA/β-TCP [12], and HA/TiO_2 [13]. It is important to emphasise that in the widely used plasma spraying method for the application of coatings a continuous flame or plasma sputtering is used [14, 15]. This can lead to undesirable overheating or melting of the particles and a significant substrate temperature increase, which is a major limitation of this method. Pulsed energy sources like detonation spraying, which uses the energy of an explosion of gas mixtures as a pulse source, are best suited for obtaining functional-gradient coatings [16]. This technology, which operates in pulsed mode, is better for obtaining functional-gradient coatings. On one side, it allows for minimising the above-mentioned negative effects. For another, the particle velocity in the detonation spraying method is much higher than in plasma spraying methods, which positively influences important coating parameters such as adhesion strength.

Functional-gradient materials are peculiar and promising composite materials whose composition/components and/or microstructure gradually change in space according to a given profile or sequence, along one or more space directions [10, 16–19]. Due to the gradual change in composition and/or microstructure, the physical and mechanical properties change in space according to the specific requirements for the prescribed application, which improves the operational characteristics of the material. Hydroxyapatite and Ti can be combined to create a perfect functional-gradient material. As the surface layer consists of HA, the resulting functional-gradient coating demonstrates excellent biocompatibility and the ability to create new bone. The excellent mechanical strength of the functional-gradient coatings is provided by the Ti phase.

There are no studies in the literature aimed at obtaining functional-gradient coatings using the detonation spraying method. Given the above, the purpose of this work is to obtain composite coatings having a gradient structure by changing the technological parameters of detonation spraying, where the HA ratio is characterized by a smooth change in the chemical composition, structure, and properties over the thickness of the coating.

Experimental

Composite HATi coatings with a thickness of about 60 μm were applied to a Ti6Al4V substrate using a CCDS2000 detonation complex (CCDS-2000, developed by Siberian Protective Coating Technologies LLC, Novosibirsk, Russia), the operation principle of which is described in detail in [20, 21]. Detonation spraying is performed by feeding combustible and oxidizing gases (propane, oxygen) into the channel in a ratio close to stoichiometric. The gas mixture is ignited in the channel by an electric spark. The ignition process is completed by the creation of a detonation wave, in the immediate vicinity of which the powder is injected into the channel by a dispenser device. During detonation combustion of a mixture of gases, propane and oxygen, the powder particles are affected by elevated temperatures, pressures and acceleration. The calculated temperature in the channel reaches 3000 K and the pressure is 5 MPa [22, 23]. At the beginning of the process, the gas powder cloud reaches a velocity of 1500 m/s, and then slightly melted powder particles are moved to the substrate at a velocity of up to 1000 m/s. Figure 1 demonstrates a general view and a schematic diagram of the detonation spraying process.

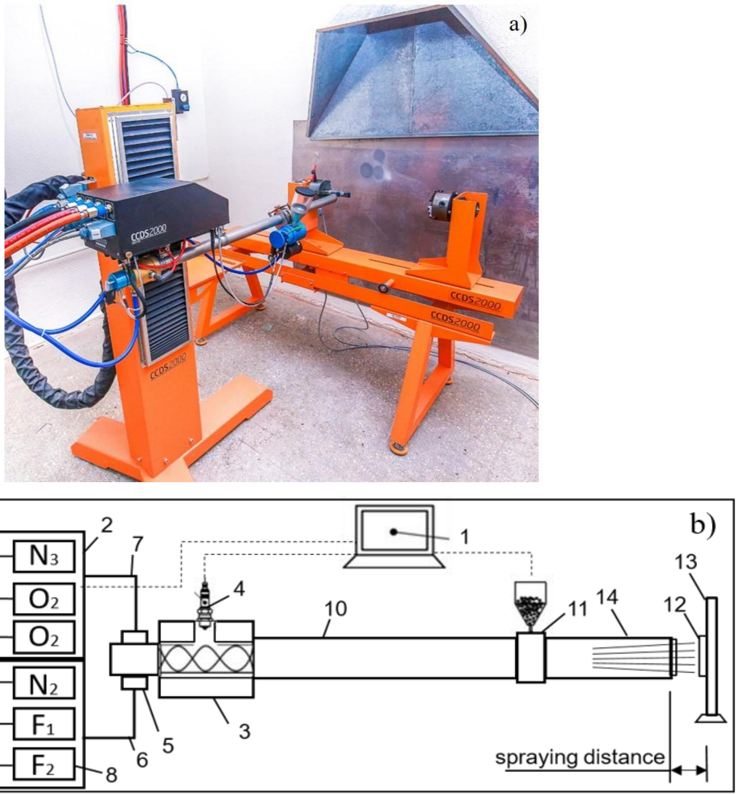

Figure 1. Computerized detonation complex CCDS2000: general view (a) and schematic diagram of the installation (b): 1 - control computer, 2 - gas distributor, 3 - mixing-ignition chamber, 4 - spark plug, 5 - barrel valve, 6 - fuel line, 7 - oxygen line, 8 - gas valves, 9 - gas supply unit, 10 - breech, 11 - powder dispenser, 12 - workpiece; 13 - manipulator, 14 - muzzle of the barrel

Ti6Al4V titanium alloy was used as the substrate material. The sample used to observe the microstructure was a rectangular size of 30 mm × 30mm × 3 mm. Table 1 presents the composition of the Ti6Al4V titanium alloy. The samples were sanded (using SiC paper with a grain size from 100 to 2000). Before coating, the substrates were sandblasted with a grain size of 250–300 microns of aluminium oxide and treated with an ultrasonic bath.

Table 1

Chemical composition of Ti6Al4V alloy (weight percent)

Ti	Al	V	Fe	C	O	N	H
88,5-92,5	5,5-6,5	3,5-4,5	<0,25	<0,08	<0,13	<0,05	<0,012

By characterizing the phase composition of the resulting HA coatings deposited at different spraying parameters, it is possible to determine suitable spraying conditions in DS. Our previous studies showed [24, 25] that by controlling the detonation spraying modes (fuel/oxidizer ratio, spraying distances) it is possible to control the temperature and speed of the coatings, respectively, which significantly influenced the melting and decomposition of HA. Preliminary studies of the microstructure, phase composition and chemical structure of DS HA coatings deposited at different spraying regimes were carried out, and the optimum spraying conditions for depositing composite HA coatings without a thermally decomposed HA phase were determined (Table 2).

Table 2

Spraying conditions

Parameters	Values
Fuel/oxidiser ratio	1,856
Spraying distance, mm	100

Based on this, Ti-HA composite coatings were obtained at different values of the barrel filling volume and different exposure times between shots to study the effect of detonation spraying process parameters on the chemical composition, structure, and properties of the coatings. The volume of the explosive gas mixture of the detonation gun barrel varied from 30% to 60% and the exposure time between shots varied from 0.25 s to 1 s. Table 3 shows the modes of production of Ti-HA-based coatings.

Table 3

Technological parameters for obtaining TiHA coatings

Name of the coatings	Barrel filling volume, %	Time between shots, s	Number of shots
TiHA-1	60	1	15
TiHA-2	45	0,5	15
TiHA-3	30	0,25	15

On this basis, Ti-HA-based coatings were obtained at different barrel filling volumes and different exposure times between shots to obtain coatings having a gradient structure. The HA ratio in these coatings is characterised by a smooth change in chemical composition, structure, and properties over the thickness of the coating (Table 4).

Table 4

Technological parameters for obtaining gradient coating

Name	Layers	Volume of barrel filling, %	Time between shots, s	Number of shots
Gradient	Top layer	30	0,25	5
	Middle layer	45	0,5	5
	Bottom layer	60	1	5

Angular hydroxyapatite (HA) powder (99.95%, produced by Sigma-Aldrich, Steinheim, Germany) with a diameter of 5-25 microns and spherical titanium powder (CL42TI) (made by Concept Laser, Germany) with a diameter of 15-45 microns were used as feedstock. HATi composite powders were obtained by mechanically mixing HA powder with Ti powder for 0.5 x using a PULVERISETTE 23 planetary ball mill. The mass ratio HA toTi for composite powders HA-Ti was 50:50. The phase composition of the coatings was studied using X-ray diffractometer X'PertPRO (Philips Corporation, Amsterdam, the Netherlands) with Cu-Kα radiation (λ = 1.5405 Å), voltage 40 kV and current 30 mA. The diffractograms were interpreted using HighScore software and measurements were taken in the 2θ range of 20°-90° in 0.02 step and 0.5 s/step counting time. The coating structure was analysed by scanning electron microscopy (SEM) using a MIRA 3 TESCAN microscope. Sliding friction wear was evaluated on a TRB^3 tribometer (Anton Paar Srl, Peseux, Switzerland) using the standard ball-and-disk technique (ASTM G 133-95 and ASTM G99 international standards), where a 6.0 mm diameter ball of SiC coated steel was used as a counterbody, at 6 N load and 15 cm/s linear speed, 5 mm radius of curvature of wear, 200 m friction path. The surface roughness of the coatings was evaluated using a profilometer model 130 (OAO Zavod PROTON, Moscow, Russia).

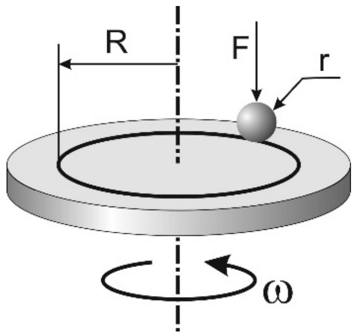

Figure 2. Experiment a schematic of the TRB^3 tribometer.

Results and Discussion

Figure 3 shows the diffractograms of the HATi composite coatings. It can be seen that the HATi composite coatings showed complex phases consisting of HA, Ti, TiO_2. HA, $Ca_3(PO_4)_2$ (TCP), and CaO thermal decomposition phases were not detected in any of the coatings. The peak intensities of HA phase decreased and the peak intensities of Ti and TiO_2 phases increased with increasing detonation spraying barrel filling volume. These results showed that the detonation spraying barrel filling volume is strongly influenced the phase composition. In addition, the diffraction patterns of the HATi composite coatings showed a decrease in the intensity of the Ti diffraction peaks indicating TiO_2 formation [26].

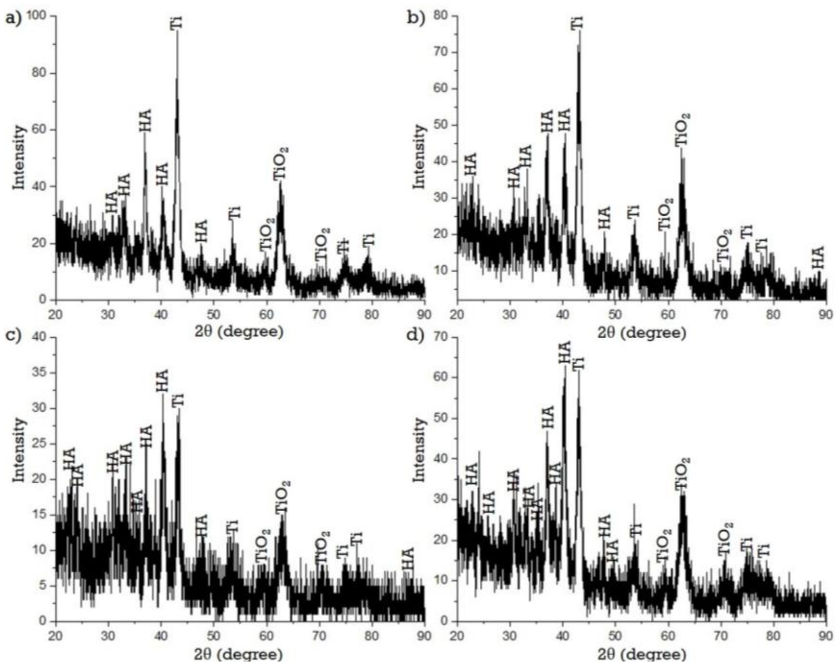

Figure 3. Diffractogram of coating TiHA: a) TiHA-1; b) TiHA-2; c) TiHA-3; d) gradient

Table 5 demonstrates the results of roughness measurements of composite and gradient coatings obtained by the detonation spraying method. The surface of all coatings has heterogeneous structure with pores, typical layered, wavy arrangement of structural components. The surface roughness of the composite coatings was measured using a model 130 profilometer on a 7 mm length segment on the sample surface. It follows from the obtained data that the roughness of composite coatings according to Ra parameter changes from 5.44 to 8.61 μm with change of technological parameters of detonation spraying. Comparison of these dependences allows to conclude that the coatings obtained with increase of detonation spraying barrel filling volume increase the roughness of coatings surface.

Table 5

Roughness measurement results for composite and gradient coatings

Name	Ra (μm)	Rz (μm)	Rt (μm)	Rq (μm)	Rv (μm)
TiHA-1	8,61	51,1	55,9	10,4	28,1
TiHA-2	6,58	44,3	46,6	8,42	27,6
TiHA-3	5,79	39,3	47,9	7,40	23,4
Gradient	5,44	39,9	48,5	6,51	22,6

Figure 4 shows a polished cross-section of the gradient coatings obtained by detonation spraying. The gradient coating consists of plates formed from molten particles during impact, some molten particles and small cracks. The presence of small cracks is beneficial in relieving thermal stresses during cooling. The total thickness of gradient coatings is about 60 μm. No obvious cracks appear at the interface between the coating and the substrate, which means that there is excellent adhesion between the coating and the substrate.

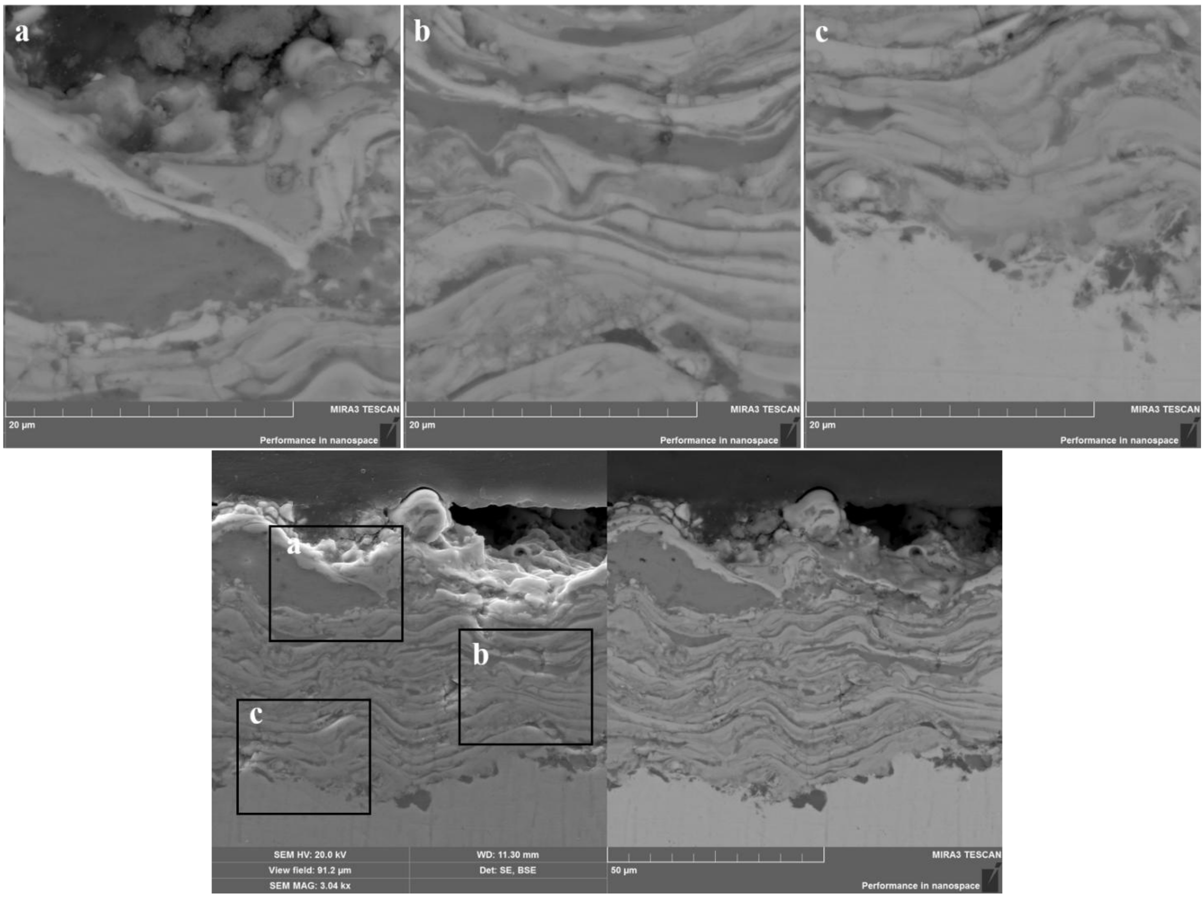

Figure 4. Cross-sectional microstructure of the HATi gradient coating.

EDS scanning light analysis shows that the coatings have a continuous gradient composition across the entire cross section with no distinguishable interface (Fig. 5 a, b, c). The Ca and P concentrations gradually increase from the substrate to the surface and the Ti concentration gradually decreases. The EDS comparisons for the gradient coating (Fig. 5 e, f) further confirm that the coating conforms to the expected gradient composition structure.

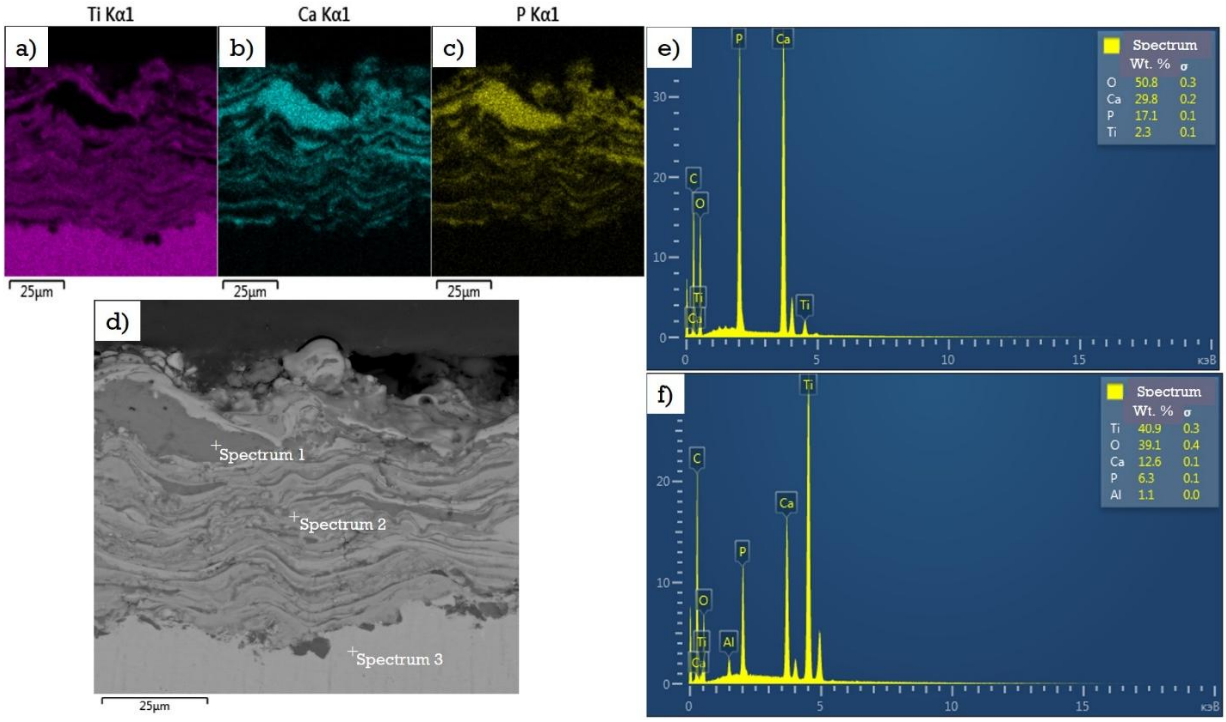

Figure 5. Cross-sectional microstructure and EDS analysis of the TiHA gradient coating.

To determine the wear resistance of the coatings, tribological tests were carried out in the ball-and-disk scheme. Figure 6 represents the friction coefficient of the composite and gradient TiHA coatings. According to the obtained results of tribological test of detonation coatings, TiHA-1 low friction coefficient values of 0.548 are observed, respectively high wear resistance in sliding friction conditions. However, the effect of surface roughness on the initial friction of the coatings is relatively higher compared to other coatings. An increase in barrel filling volume leads to an increase in the coefficient of friction of the coatings.

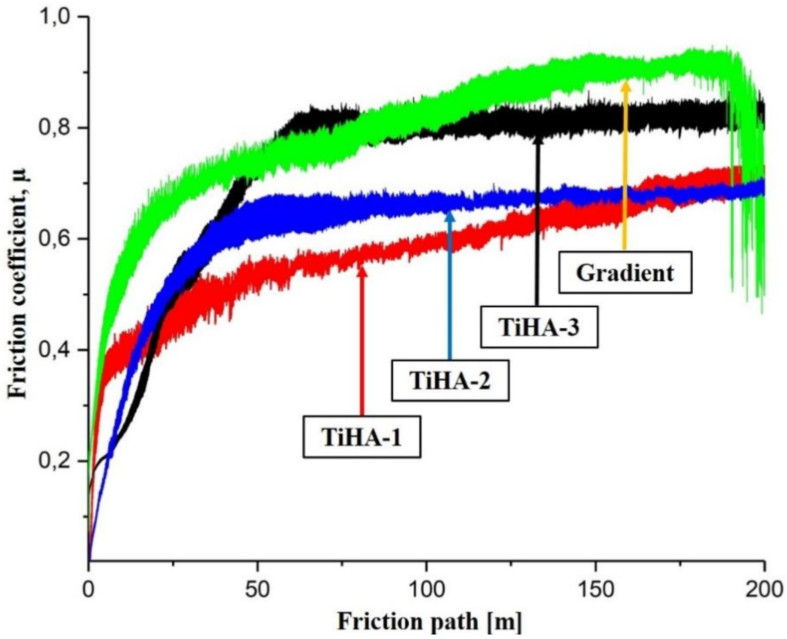

Figure 6. Coefficient of friction of composite and gradient coatings

Conclusions

The functional-gradient TiHA coating was successfully applied to the surface of Ti6Al4V alloy by detonation spraying. To obtain functional-gradient coatings, the following detonation spraying process parameters were varied: detonation barrel filling volume with acetylene-propane gas mixture from 60% to 30% and exposure time between shots from 1 to 0.25 s. A study of the phase composition of the coatings depending on the technological parameters showed that with increasing volume of barrel filling the content of HA phases in the composition of the coatings decreases, and the intensity of the diffraction peaks of Ti and TiO_2 increases. The study of the coatings surface morphology showed that the roughness of the coatings by Ra parameter varies from 5.44 to 8.61 μm with a change in the detonation barrel filling volume from 30% to 60%. The higher surface roughness and porosity of the outer layer of the functional gradient coating is a favourable microstructure for bone growth. We can conclude that the functional-gradient coatings obtained by the detonation spraying method have great potential for use as bioimplantation materials and need further research to reveal its hidden potential.

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Ж.Б. Сағдолдина, Д.Р. Байжан, Е.Е. Қамбаров, Қ. Төребек

Детонациялық бүрку әдісімен функционалды-градиентті жабындарды алу

Функционалды градиентті титан / гидроксиапатит (TiHA) жабыны құрылымы мен механикалық қасиеттерін жақсарту үшін детонациялық бүрку технологиясын қолдану арқылы алынды. Функционалды-градиентті жабындарды алу үшін импульсті энергия көздері ең қолайлы, атап айтқанда, газ қоспаларының жарылыс энергиясы импульстік әрекет көзі ретінде пайдаланылатын детонациялық бүрку. Детонациялық бүрку режимдерін басқару арқылы жабынның тұндыру температурасы мен жылдамдығын өзгертуге болады, сәйкесінше жабындардың белгілі құрылымдық-фазалық құрылымын алуға болады. TiHA детонациялық жабындарының құрылымдық-фазалық күйлері мен трибилогиялық қасиеттері заманауи материалтану әдістерімен анықталды: рентгенофазалық талдау (XRD), сканерлеуші электронды микроскопия (SEM), энергодисперсиялық спектроскопия (EDX-карталау), профильометрия және «шар-диск» схемасы бойынша тозуға төзімділігін сынау. Нәтижелер жабындардың элементтік интерфейсінсіз жабындардың көлденең қимасы бойынша үздіксіз градиентті элементтік құрамы бар екенін көрсетті. Ti мөлшері біртіндеп азайды, ал гидроксиапатит мөлшері субстраттан жабын бетіне қарай біртіндеп өсті, бұл сүйек имплантталары үшін TiHA жабындарын қолдану мүмкіндігін кеңейтуге мүмкіндік береді. Беткі қабат HA–дан тұратындықтан, алынған функционалды градиент жабыны керемет биожетімділік пен жаңа сүйек тінін жасау қабілетін жақсартады. Функционалды-градиент жабындарының жоғары механикалық беріктігі Ti фазасымен қамтамасыз етіледі.

Кілт сөздер: детонациялық бүрку, функционалды градиентті жабындар, микроқұрылым, фазалық құрамы, механикалық қасиеттері, гидроксиапатит, титан, тозуға төзімділігі, кедір-бұдырлығы.

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Получение функционально-градиентных TiHA покрытий методом детонационного напыления

Функционально-градиентные покрытия титан/гидроксиапатит (TiHA) были получены с использованием технологии детонационного напыления с целью улучшения структуры и механических свойств. Для получения функционально-градиентных покрытий лучше всего подходят импульсные источники энергии, а именно детонационное напыление, в котором в качестве источника импульсного действия используют энергию взрыва газовых смесей. Управляя режимами детонационного напыления, можно варьировать температуру и скорость нанесения покрытий, соответственно, можно получить определенное структурно-фазовое строение покрытий. Структурно-фазовое состояние и трибилогические свойства детонационных покрытий TiHA исследованы методами современного материаловедения: рентгенофазного анализа (XRD), сканирующей электронной микроскопии (SEM), энергодисперсионной спектроскопией (EDX-картирование), профилометрии и испытанием на износотойкость по схеме «шар–диск». Результаты показали, что покрытия имели непрерывный градиентный элементный состав по поперечному сечению покрытий без границы раздела элементных слов покрытий. Количество *Ti* постепенно уменьшалось, а количество гидроксиапатита постепенно увеличивалось по направлению от подложки к поверхности покрытий, что позволяет расширить возможности применения *TiHA* покрытий для костных имплантатов. Поскольку поверхностный слой состоит из *HA*, полученное функционально-градиентное покрытие демонстрирует отличную биосовместимость и способность для создания новой костной ткани. Превосходная механическая прочность функционально-градиентных покрытий обеспечивается фазой *Ti*.

Ключевые слова: детонационное напыление, функциональные градиентные покрытия, микроструктура, фазовый состав, механические свойства, гидроксиапатит, титан, износостойкость, шероховатость.