STYDYING OF MORPHOLOGY AND ELEMENTAL COMPOSITION OF THE CALCIUM PHOSPHATE LAYER AFTER TREATMENT BY IMPULSE ELECTRON BEAM.

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One of the effective ways to improve the adhesive properties of biocompatible coatings on implants is using electron-beam melting of the surface, accompanied by a partial or complete mixing area "coating- substrate". The surface of the titanium substrate bearing calcium phosphate coating received by RF magnetron sputter is processed by a pulsed electron beam having an energy density of 0.8 - 8 J/cm2. After treatment by a pulsed electron beam under different regimes significant changes in the topography of the formed surface were observed. Treatment regimes with an energy density of 0.8 J/cm2 and 3 lead to the thermal annealing of the coating. The use of a beam having an energy density of 8 and 6.5 J/cm2 leads to partial vaporization and mixing of the coating material with a titanium matrix.

Currently, studying materials in the field of medical supplies is a very promising direction in science [1-2]. Modification of the surface by forming biocompatible coatings of different thicknesses is the most common method used in industrialized countries. Development of new materials, creation of thin solid, durable coatings (on the developed surface of the implant) is of considerable interest nowadays.

Ideally, the material should be biocompatible with the tissue, that is, to cause an adequate response, not be toxic, not cause adverse immune reactions and of the body. It should not be rejected by the body as an external body and be biologically active, that is, it should come into direct contact with biological system of the body and induce bone formation processes. At the same time, implants should maintain their functional qualities [3]. This problem was solved by using materials based on calcium phosphates biocompatible with the tissues of the body, characterized by activity of the compound towards bone tissue and the formation of new bone tissue. It represents the unique possibility of using calcium phosphate as a coating material for implants [1].

However, at this stage of development problem of destruction of the coating when it is placed in liquid biological environment, i.e. cracks that reach the interface "coating- substrate" remains unsolved in biomedicine [4]. One effective way to solve this problem is proposed by several authors [5, 6]. It is the use of electron-beam melting of the surface, accompanied by a partial or complete mixing area "coating- substrate", which will give an opportunity to get a high adhesive strength [7].

In this regard, the aim of this work is to study the morphology and elemental composition of calcium - phosphate layer after treatment by pulsed electron beam.

Deposition of silicon-based coatings of hydroxyapatite (Si-HA) was produced using an industrial installation COMDEL with magnetron source (13.56 MHz). The distance between the substrates and magnetron source is 40 mm, the RF power is 500 W, the pressure of the working gas is 0.1 Pa working gas is argon and deposition time is 8 hours. Preparation and studying of powder and spray targets is presented in [8].

Commercially pure titanium VT1-0 most often applicable in medicine is used as the material for substrate. Irradiation of surface of the samples was carried out on the vacuum impulse electron beam (IEP) installation "Solo" (Institute of High Current Electronics SB RAS, Tomsk, a team led by Koval N.N.). Irradiation was carried out by electronic pulses of duration τ = 50 ms (number of irradiation pulses N = 3-50, repetition frequency f = 0,3-5 Hz) with an energy density Es = 0,8-8 J/cm^2. Irradiation modes of the samples are given in Table 1.

Table 1

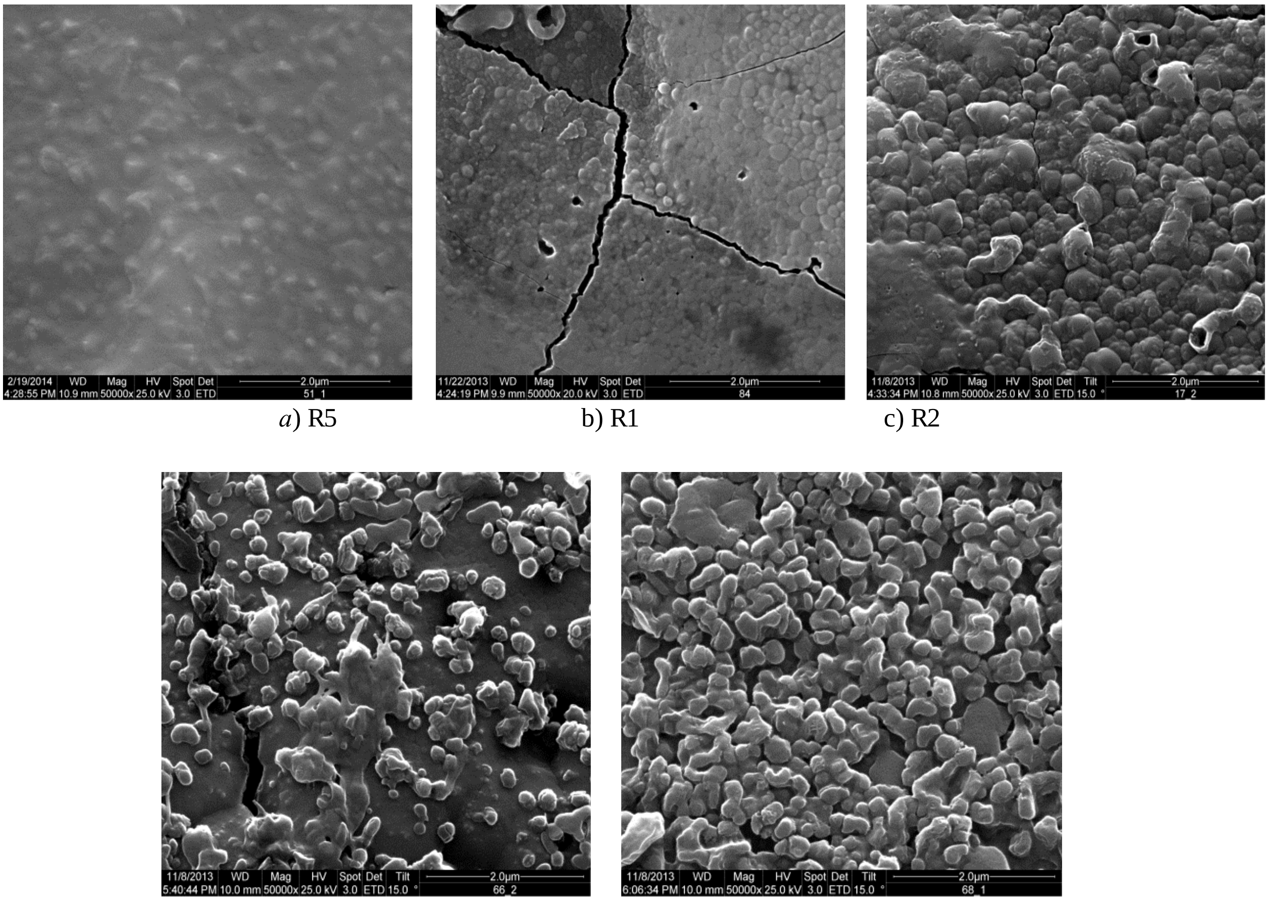
Modes of irradiation of the samples by pulsed electron beam before (R5) and after treatment (R1-R4).

The morphology of the surface of the Si-HA coatings was investigated by scanning electron microscopy (SEM, ESEM Quanta 400 FEG) with integrated energy-dispersive X-ray spectrometer (EDS analysis system Genesis 4.000, S-UTW-Si (Li) detector). A glow discharge optical emission spectrometer (RF GD-OES) GD Profiler 2 was used for measuring of the elemental composition.

Modes	E_s, J/cm^2	τ, μs	f, Hz	N
R1	3	50	0,3	3
R2	0,8	50	5	50
R3	6,5	50	0,3	3
R4	8	50	0,3	3
R5	–			

Fig .1 shows the surface morphology of the system "coating-substrate" before and after the IEB. Treatment of the calcium phosphate film and the titanium in this way results in changes to the topography of the formed surface. Prior to treatment, the surface of the coating is an alternation of evenly spaced ridges passing into the cavities. Modes of R1 and R2 are given a short term thermal annealing surface which causes formation of a homogeneous surface morphology. However, cracks are present. The scaly structure of the surface is preserved but the shape and size of the grains undergo changes. In the case where the energy density is 6.5 and 8 J/cm^2, the surface of the composite consists of a plurality of small, fused, shapeless and unevenly distributed particles (Fig. 1, d – e), with areas having low vividness.

In the case of modes R3 and R4 the elements of the substrate are the dominant elements. Thus, the ratio of Ca/P increased significantly for these regimes (Table 2), this indicates the preferential evaporation from the surface during processing of the phosphorus elements. However, at these energies of the beam coating could be mixed with material in the process of formation of new phases.



a) R5 b) R1 c) R2

d) R3 e) R4

Fig.1. The morphology of the composite surface layer "coating-substrate" before (a) and after treatment with the pulsed electron beam (b - e).

a) b)

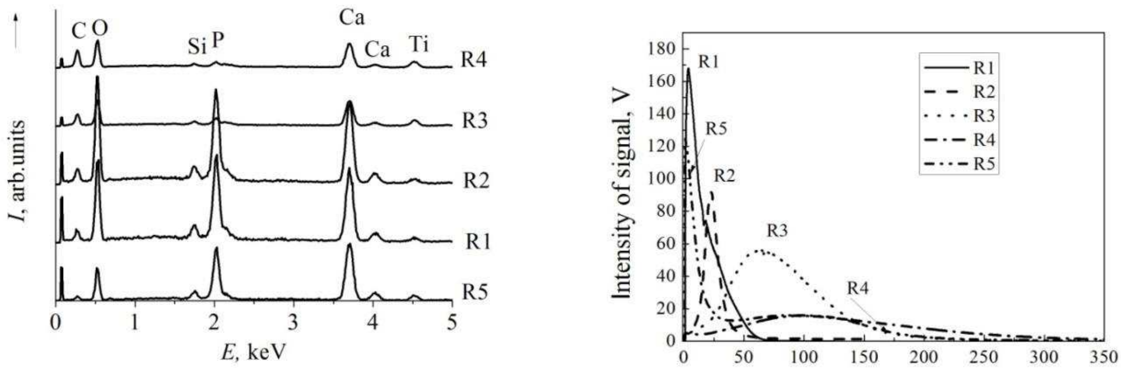


Fig. 2. EDS-spectra (a) and the calcium distribution in the depth (b) for the composite surface layer "coating-substrate" before (R5) and after treatment with the pulsed electron beam (R1-R4)

Table 2

Values of the ratios of Ca / P and Ca / (P + Si)

Regime	Ca/P	Ca/(P+Si)
R5 (before treatment)	1.94	1,66
R1	1.80	1.56
R2	1.87	1.66
R3	8.27	5.82
R4	11.96	8.29

Assumption about mixing is confirmed by the analysis of calcium content in the composite surface over depth, which is shown in Figure 2 b.

The abscissa indicates the time when the coating is sputtered at the moment of investigation, reflecting the composition of the sample changes depending on depth. Thus, treatment regimens by pulse electron beam having an energy density of 0.8 and 3 J/cm2 lead to a thermal annealing of the coating formed on the titanium substrate.

Using the beam with energy density of 8 and 6.5 J/cm^2 leads to partial vaporization and mixing of the coating material with titanium matrix. It is necessary to use x-ray diffraction to identify formed phases in the surface layer.

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