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Influence of Various Production and Processing Methods on the Structure and Properties of Porous Titanium Coatings

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Abstract. A comparison was made of the properties of porous coatings obtained by ion-plasma spraying methods, 3D-coating obtained by direct laser metal deposition (DLMD), as well as a GRIPTION® and POROCOAT® coating. The influence of various methods of obtaining a porous coating on the porosity and structure of the coating, as well as the effect of thermal hydrogen treatment (THT) on the adhesion strength of the titanium coating was determined.

Introduction

Hip joint endoprostheses are complex multicomponent. products, for each element of which certain functional requirements are imposed [1-4]. For example, the surface of the implant stem for cemented fixation must be of high cleanliness and durability because in the process of fixation, its micro-movement occurs in the cement mantle. And the surface of the components of the endoprosthesis for cement fixation, on the contrary, should have a high roughness. The primary fixation of the components is carried out due to the “tight” fit into the bone, and the stability of work is due to the osseointegration (germination) of the bone structures into the component [2.4]. Certain requirements are imposed on the porous coating of the cup of the acetabular component of the hip joint for the pore size and adhesive strength of the coating to the base. The bowl is made of titanium alloy VT6 (Ti-6Al-4V), and for a porous coating, alloy VT1-0 (pure titanium) is usually used, which has a higher biocompatibility [1-5]. Today, one of the main methods for producing porous coatings is plasma spraying. The disadvantage of this method is the low adhesive strength of the coating, the difficulty of obtaining a given porosity. Recently, 3D printing technologies have been used to apply porous coatings. In the modern industry, 3D technology has started to occupy a large place. They allow you to control the degree of porosity of the coatings [6.8].

Reversible doping with hydrogen is one of the promising methods of significant structural changes in titanium alloys. Hydrogen is a unique intrusive alloying element for titanium alloys. Under certain conditions, it can be quite easily both "introduced" and "removed" from the material without changing its state of aggregation. At the same time, having a strong β-stabilizing effect, its introduction makes it possible to increase the amount of β-phase at the intermediate stage of processing to almost 100% even in pseudo α-titanium alloys, in which its volume fraction under normal conditions does not exceed 10%. This makes it possible, during subsequent degassing in a wider range, to control the structure of titanium alloys and, accordingly, to obtain unique properties that cannot be achieved with traditional processing methods. The diffusion mobility of hydrogen controlled by the processing temperature is several orders of magnitude higher than the diffusion mobility of the main alloying elements in titanium alloys, therefore, by changing the modes of hydrogenation annealing, it is possible to obtain both volumetric and surface changes in the structure. [2,3,5].

Material and Methods

The objects of the study were the cups of the acetabular component of the hip joint, titanium porous coatings on which were applied by different methods: plasma spraying. For comparison, bowls were examined, on which coatings were applied using the POROCOAT® and GRIBTION® [9] technology and direct laser metal deposition (Fig. 1). The bowls with coatings obtained by plasma spraying and 3D printing were subjected to thermal and thermal hydrogen treatment. The test for the adhesion strength of the coating to the base was carried out on a TIRAtest 2300 testing machine on cylindrical specimens of titanium alloy VT6 coated with a coating of VT1-0 or VT6 alloy.

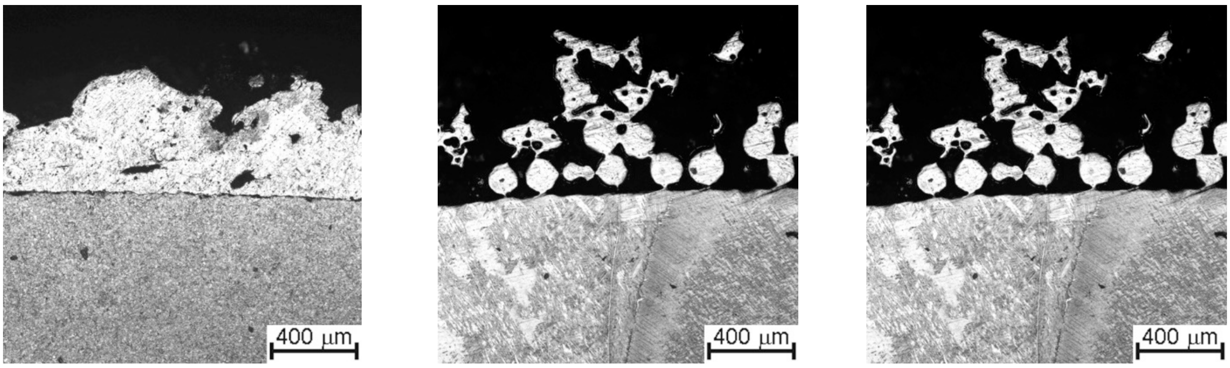
a) b) c)

Fig. 1. Appearance of bowls obtained by plasma spraying (a) using GRIPTION® technology (b) and DLMD (c)

Metallographic studies of the porosity of the coatings, the base-coating interface, and the structure were carried out using an AXIO Observer.Alm optical microscope at magnifications up to 1000 times. The bright field method in air was used. The images were analyzed using the NEXSYS ImageExpert Pro3.6 software package. The samples were saturated with hydrogen in a Sieverts apparatus in the field of atomic hydrogen. Vacuum annealing was carried out in a VEGA 3M furnace.

Results and Discussion

One of the main requirements for the cup of the acetabular component of the hip joint is the porosity and depth of open pores for the possibility of bone invasion. Therefore, at the initial stage of the work, the external pores were measured. For this, 5 fields were filmed at a low magnification of 25 times, obtained by plasma spraying, using the GRIPTION® technology and 3D printing (Fig. 2).



a) b) c)

Fig. 2. Structure of porous coatings obtained by plasma spraying (a), using GRIPTION® technology (b), and DLMD (c)

The coating obtained by the method of plasma spraying is "dense", has a small number of internal pores and a developed relief (Fig. 1a). The first layer of the POROCOAT® coating consists of spherical granules on top of which irregularly shaped powder particles are applied by technology GRIPTION®. This coating has a developed relief with “through porosity” (Fig. 2b). The coating obtained by 3D technology consists of through pores (Fig. 2c).

To obtain quantitative data on porosity, the distance between particles and the depth of open pores were measured. The scheme for measuring the width and depth of pores is shown in Fig. 3.

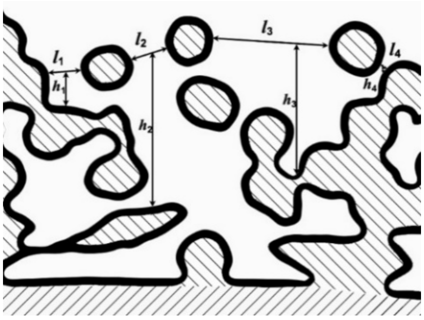
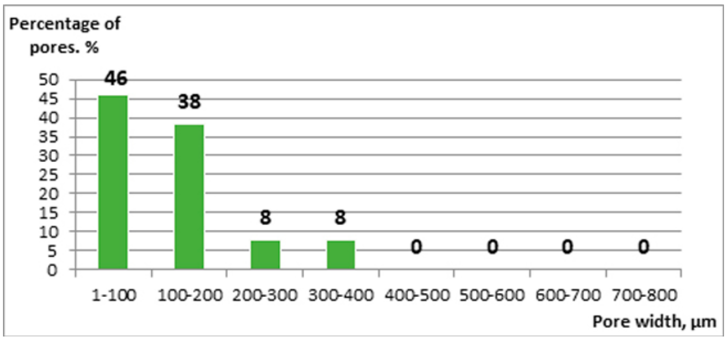
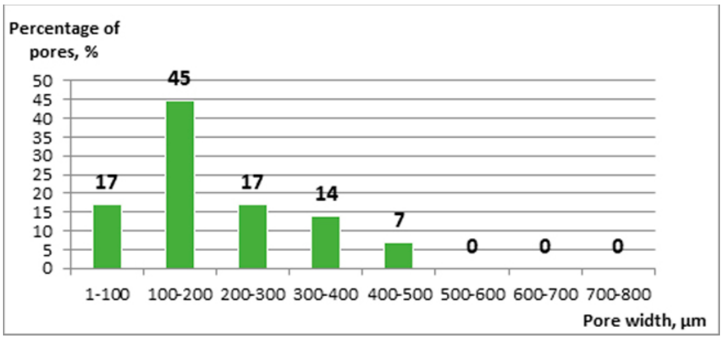


Fig. 3. Scheme for measuring pore width and depth.

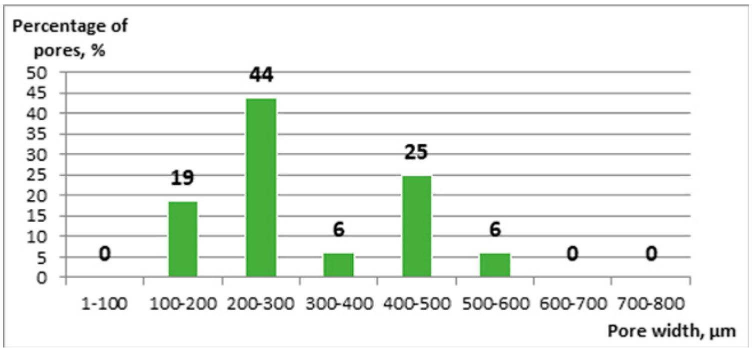
The calculation was carried out for 5 fields of view, while the total distance of the measured coating was 2555 µm. The measurement results are shown in Figure 4. A porous coating obtained by plasma spraying is characterized by a minimum number of open pores - 5 pores / mm. The main number of pores has a size from 100 to 200 µm with an average depth of 80 µm (Fig. 4a). The porous coating obtained using the POROCOAT® technology is characterized by a significantly larger number of open pores - 11 pores / mm. Almost 50% of the pores have a size from 100 to 200 µm (Fig. 4b) with an average depth of 200 µm. The 3D printed coating has 7 open pores in one millimeter. The maximum proportion of pores falls on a size of 200-300 microns (Fig. 4c) and an average depth of 200 microns.



a



b



c

Fig. 4. Pore width distribution in coatings obtained by plasma spraying (a), using the POROCOAT® technology (b), and DLMN (c)

An important indicator for porous coatings, in addition to the size and number of open pores, is the adhesive strength. The coating must have strong adhesion to the base of the bowl to avoid peeling during operation. Therefore, at the next stage in the work, a metallographic study of the interface between the base and the porous coating was carried out.

For coatings obtained by plasma spraying and 3D printing, the interface is clearly visible when passing from the base metal to the coating (Fig. 5 a, b) after annealing at a temperature of 850 ° C. At the same time, the POROCOAT® and GRIBTION® coating is characterized by the presence of common grains at the interface, which indicates the formation of physicochemical contact at the base-coating interface.

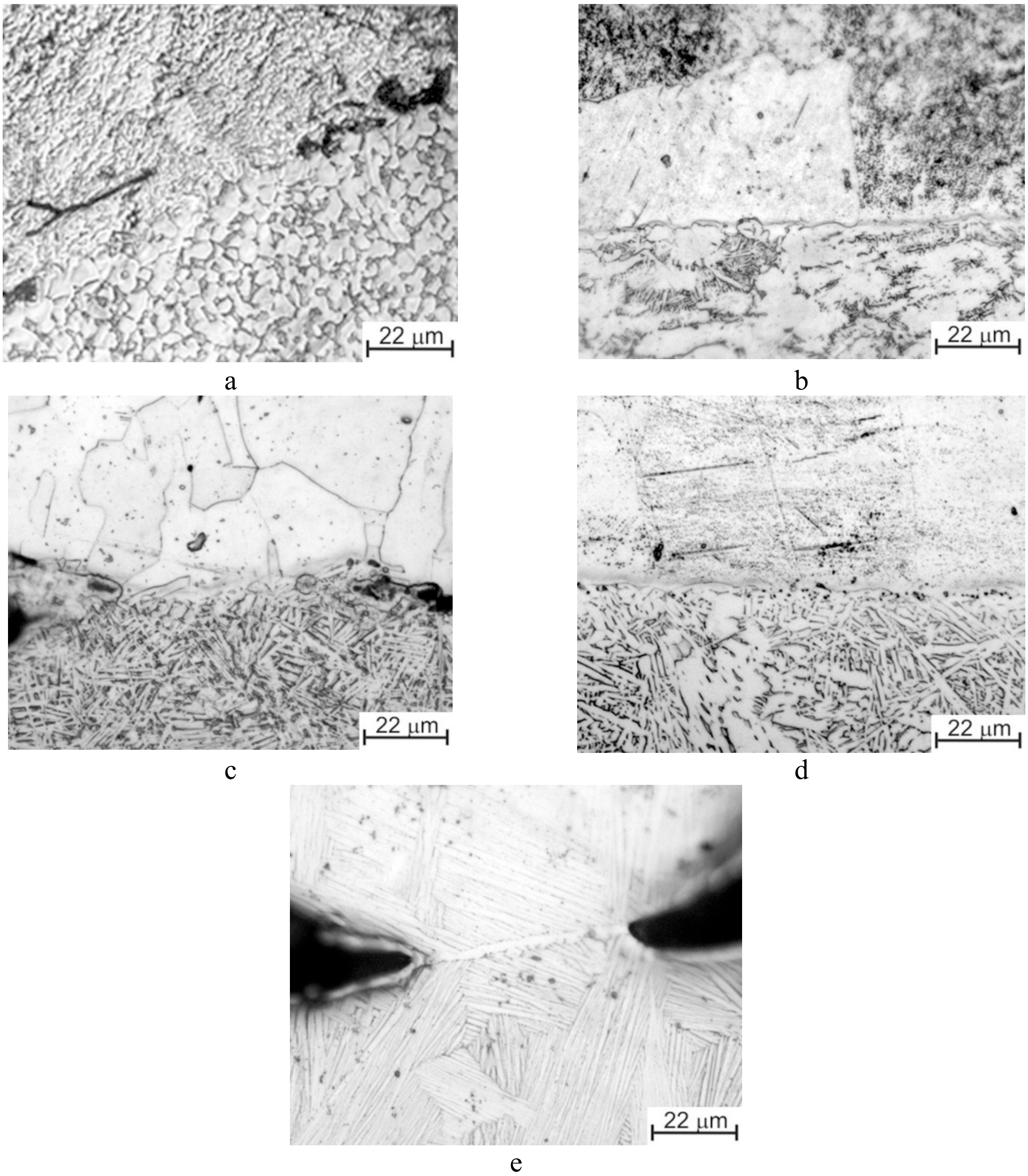


Fig. 5. Microstructure of the base-coating interface obtained by plasma spraying (a, c), 3D-printing (b, d), using POROCOAT® and GRIBTION® technologies (e). After annealing (a, b, e) and thermal hydrogen treatment (c, d)

One method for determining the adhesion strength of a coating is a shear test of the coating. The results of testing the shear stress for porous coatings obtained by the method of plasma spraying in the initial state were obtained and was 18 MPa, and by the method of 3D printing after vacuum annealing - 17 MPa. Additionally, thermal hydrogen treatment of samples with porous coatings obtained by plasma spraying and 3D printing increased the shear stress to 210 MPa and 175 MPa, respectively.

Conclusion

An important property for the cups of the acetabular component of the hip joint is their osseointegration property. Based on the results obtained, it can be concluded that the most developed coating (width and depth of open pores) is for a bowl with a coating applied using POROCOAT® and GRIPTION® technologies. The worst indicators of the development of the coating have a bowl with a coating obtained by plasma spraying.

The second important property of the coating is adhesive strength. Indirectly, this can be judged by the physical and chemical contact of the coating and the base of the bowl. The best physicochemical contact has a coating applied using POROCOAT® and GRIPTION® technologies. For bowls with plasma spraying and 3D coating, even after TVO, the boundary between the base and the coating is clearly traced, which also indicates a low physical and chemical contact.

Shear stress tests have shown that thermal hydrogen treatment is preferred for 3D coated bowls to increase adhesion strength.

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