Research of the structure and mechanical properties of gas-thermal coatings after induction heat treatment

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Abstract. In this article the process of induction-thermal modification of titanium coatings formed by electroplasma spraying was considered. The influence of the inductor current on the temperature of processed samples was experimentally established. The research results showed that thermal treatment of the samples with titanium coatings at a temperature of 750–1200 °C and a duration of 300 s led to an increase in porosity from 56±2 to 61±1 % and in microhardness from 1035–1532 to 1825–1883 HV0.98, the sprayed layer thickness decreased from 320±30 to 114±15 µm as well. A change of nanoscale structural elements shape was also observed.

1. Introduction

Designs of restorative medicine are often made from titanium based alloys [1]. The element-phase composition and structural-mechanical properties of the surface influence the osseointegration of implantable structure. Hence, a biocompatible coating similar in structure to bone tissue is formed on the implant.

In biomedicine, aircraft construction, mechanical engineering, coatings formed by gas-thermal spraying (GTS), electrochemical deposition and vacuum technologies (CVD and PVD) are widely used [2–4]. Gas-thermal spraying is distinguished by high productivity, technological simplicity, so it has become widespread in production. One of the GTS methods is electroplasma spraying (EPS) that allows the formation of titanium coatings by applying powder material. According to the review results the porosity of these layers is 10–50 %, whereas the adhesion strength between the coating and the base may reach 20 MPa [5–7].

The main disadvantages of the EPS method are the unevenness of the elemental-phase composition, the presence of structural defects in the spayed material, which leads to the coating destruction. To improve the functional properties of plasma coatings clad powders are used and the base is subjected to preliminary induction heating. Besides, modification methods such as microarc oxidation, laser and high-temperature processing are also used. During induction heat treatment (IHT) simultaneous heating of the coating and base occurs. This treatment contributes to a change of porosity, hardness and adhesive-cohesive strength. It is known that IHT enables the formation of nanostructured metal oxide layers on titanium, which increase osteoconductive properties of the implant surface [8].

At the moment the works devoted to IHT of sprayed coatings are not enough for the further development of surface modification technology. The purpose of this work was to study the effect of IHT parameters on the structure and hardness of titanium coatings formed by electroplasma spraying.
2. Methodology
The research samples were titanium disks with a diameter of 15 mm and a thickness of 0.5 mm. Prior to the coating deposition the sample surfaces were subjected to abrasive blasting with an electrocorundum powder having a dispersion of 250–300 µm and at a pressure of 4±0.5 MPa on the “Chaika-20” setup. Technological contaminants were removed from the samples in a surfactant solution using an “UZUMI-2” ultrasonic bath. The coating was formed by EPS of PTS grade titanium powder with a dispersion of 100–150 µm (TC 14-22-57-92 Titanium powder) using the “UPN-28” installation. The following spraying modes were set: plasmatron arc current – 350 A (±1 %); spraying distance – 150±10 mm; transporting gas consumption. IHT of samples was carried out on a “VCh-15” installation at a constant exposure of 300 s, an inductor current of 3.2–8.0 kA which provided heating of samples to the temperatures from 650 to 1150 °C (figure 1).

![Figure 1](image)

**Figure 1.** Accordance of the inductor current and the treatment temperature.

The analysis of the coating structure was carried out by scanning electron microscopy (SEM) using a “MIRA 2 LMU” microscope. Size of the structural elements, porosity and thickness of the resulting layers were determined from the images of the coating areas (size – 660 by 495 µm) using the “Metallograph” program for analyzing geometric parameters of microobjects [9]. The coating and titanium base microhardness was assessed according to the manufactured cross-sections with a step of 50 µm from the “coating – base” boundary with the use of “PMT-3” hardness tester at a load on the Vickers indenter equal to 0.98 N (ISO 6507-1:2018).

3. Results
In the course of plasma spraying the coating was formed from individual molten particles arranged in layers and forming agglomerates with a size of 100–200 µm (figure 2a). Deep pores and cracks characterized by a summarized surface porosity of 56±2 % were observed.

Thermal modification in the low-temperature range (T = 750–800 °C) allowed the reduction in the number of cracks. Porosity increased to 61±1 % (figure 2b). The number of spherical particles less than 5 µm has decreased, while the number of larger particles with the size of 10–50 µm was practically unchanged. A growth of modification temperature to 900–950 °C led to a noticeable decrease in the number of pores and cracks, while the porosity was 55±2% (figure 2c). Particles of 30–40 µm in size prevailed in the structure of surface modified coating. A further increase in the inductor current and, accordingly, the treatment temperature did not result in a significant change of porosity as the latter remained equal to 56±1% (figure 2d).

It was revealed that IHT affected the sizes and shapes of the coating structural elements. Nanoparticles with a size of 100–200 nm were observed during EPS (figure 3a). After the coating was
modified at a minimal temperature of 750–800 °C prismatic crystals appeared in its structure, whereas the particle size increased to 500–600 nm (figure 3b).

Figure 2. SEM of the sample images: a – plasma sprayed coating (no IHT); b – T = 750–800 °C; c – T = 900–950 °C; d – T = 1150–1200 °C.

The thickness of the initial layer obtained by the EPS method was 320±30 μm. Microindentation showed that the resulting porous structure was characterized by a hardness of 1035–1530 HV$_{0.98}$ at a depth of 200 μm from the "coating – base" interface (figure 4). Microhardness of the titanium base of samples did not exceed 145 HV$_{0.98}$. Oxidation processes were likely to occur during low-temperature induction treatment, which led to an increase of the coating thickness to 360±35 μm. The modified layer microhardness at a depth of 100–150 μm reached 1825 HV$_{0.98}$, while at the value of 25 μm (close to Ti base) it was 600 HV$_{0.98}$. Thermal modification in the medium and high temperature ranges (900–950 and 1150–1200 °C) resulted in a decrease of the coating thickness to 115±15 μm and 190±25 μm and stabilization of microhardness at the level of 1600 HV$_{0.98}$ and 1885 HV$_{0.98}$, respectively. After heat treatment at 900–950 °C, the hardness of the titanium base grew to 270 HV$_{0.98}$. At the highest IHT temperature, the hardness of the sample bases reached 440 HV$_{0.98}$. Thus, the change in hardness was associated with oxidation and the formation of strengthened diffusion layers.

Figure 3. SEM showing the images of samples with nanoparticles: a – coating without IHT; b – T = 750–800 °C.
4. Conclusions

Thus, the plasma sprayed coatings had a high summarized porosity of about 56±2 % at a thickness of 320±30 μm. In this case, nanoparticles with a size of 100–200 nm were observed. The resulting structure had a high microhardness of 1035–1530 HV0.98. The titanium base hardness during this treatment did not exceed 145 HV0.98. IHT at an inductor current of 3.5 kA, which corresponded to a temperature of 750–800 °C, contributed to an increase of open porosity up to 61±1 % and a coating thickness to 360±35 μm. Particle size grew to 500–600 nm. The microhardness of this coating was 1100–1825 HV0.98 and it gradually fell to 600 HV0.98 at the interface of the titanium base. An increase of the inductor current to 4.8 kA and the process temperature to 900–950 °C led to a porosity reduction of 55±2 %. At the maximum value of the inductor current equal to 8.0 kA, and, accordingly, the exposure temperature of about 1150–1200 °C, no significant changes in porosity were revealed (56±1%). Individual structural elements of the coating have acquired a prismatic shape. Treatment at an inductor current of 4.8–8.0 kA led to a decrease in the coating thickness to 114±15 μm and 186±24 μm, the microhardness over the cross-section of the sprayed material did not exceed 1600–1880 HV0.98. Thus, it can be concluded that the resulting coatings can be used in the manufacture of titanium designs in the field of restorative medicine.

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References

[1] Pohler O E M 2000 Injury 31 7
[2] Vilardell A M, Cinca N, Garcia-Giralt N, Dosta S, Cano I G, Noguès X and Guilemany J M 2018 Journal of Materials Science: Materials in Medicine 29(2) 1
[3] Jeyachandran Y L, Karunagaran B, Narayandass S K, Mangalaraj D, Jenkins T E and Martin P J 2006 Materials Science and Engineering: A 431(1-2) 277
[4] Cinca N, Barbosa M, Dosta S and Guilemany J M 2010 Surface and Coatings Technology 205(4) 1096
[5] Qian M, Froes F H 2015 Butterworth-Heinemann 627
[6] Vogel D, Dempwolf H, Baumann A and Bader R 2018 Journal of the Mechanical Behavior of Biomedical Materials 77 600
[7] Kalita V I, Komlev D I and Radyuk A A 2016 Inorganic Materials: Applied Research 7(4) 536
[8] Fomin A A, Dorozhkin S, Fomina M, Koshuro V, Rodionov I, Zakharevich A, Petrova N and Skaptsov A. 2016 Ceramics International 42(9) 10838
[9] Fomina M, Koshuro V, Papshev V, Rodionov I and Fomin A 2018 Data in Brief 20 1409