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APPLICATION OF SLM-TECHNOLOGY FOR MANUFACTURE OF DENTAL IMPLANTS FROM Ti–6Al–4V ALLOY

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ABSTRACT

Nowadays, SLM-technology has found application in various fields, including dental. The manufacture of dental implants by SLM has significant advantages. The aim of the work was the development and manufacture of equipment, practicing printing conditions by SLM technology and electrochemical polishing of dental implants with Ti–6Al–4V. The titanium Ti–6Al–4V alloy of the chemical composition, wt.%: 6.21 Al; 4.03 V; 0.04 Fe; 0.1 C; 0.7 O; 0.02 N; Ti – base, was used. The source material was examined using a scanning electron microscope REM-106 and microstructure was examined by CarlZeiss AxioVert 200M mat. The mass control was performed by the analytical scales ADV-2000. Electrochemical polishing was performed in a solution of hydrofluoric acid (HF), nitric acid (HNO_3) with glycerol ($C_3H_8O_3$). The ultra-compact 3D printer Alfa-150D with a working field size of 150×150×180 mm was designed and manufactured. The printer is equipped with a high-precision ytterbium laser with air cooling of 200 W power. The positioning accuracy of the laser beam is 0.15 μm. The thickness of the working layer is 20–100 μm. The samples of implants from Ti–6Al–4V were made according to experimental technological conditions: constant laser power — 195 W, laser beam scanning speed — 1000–1200 mm/s with a step of 50 mm/s, distance between beam passes — 0.09–0.12 mm with a step of 0.01 mm at a constant scanning speed. The set conditions: laser power — 195 W, scanning speed — 1000 mm/s and distance between tracks — 0.12 mm provide the density of metal samples of more than 99.99 %. On the implants manufactured according to the recommended conditions, the effect of current (0.5–2.5 A), voltage (12–20 V) and duration (3–6 min) on mass loss during electrochemical polishing was investigated. Rational conditions of posttreatment were established with the use of visual analysis. Rational conditions (current — 2 A, voltage — 17 V) of electrochemical polishing of dental implants for reduction of roughness and during maintenance of accuracy of geometry in the field of a thread were established. The dependence of mass loss of dental implants during electrochemical polishing depending on the duration of treatment was established.

KEY WORDS: selective laser melting, titanium alloy, dental implant, equipment development

INTRODUCTION

Selective Laser Melting (SLM) has found a wide application in recent years due to the possibility of manufacturing of high-density complex profile products based on a computer model with almost any metal powders (zinc, bronze, steel, titanium and titanium alloys, aluminium and aluminium alloys, precious metals, etc.)

Using selective laser melting, it is possible to create unique products of a complex profile without the use of a large amount of expensive fixture and almost without material wastes. Nowadays, the technology of selective laser melting has found its application in different industries, including dental.

Traditional manufacture of dental prostheses, crowns, bridges, which is performed on a cast model from teeth, becomes a thing of the past. It takes a lot of time associated with unpleasant senses and discomfort while removing cast models in a traditional way. The work of dental technicians is very labour-intensive and takes a lot of time to get a good result. A unique shape of each tooth according to the individual needs of a patient is very difficult to reproduce using manual manufacture or milling machine. In addition, in the milling technology in the course of treatment of a monolithic block of a material, up to 90 % goes to chips.

Modern clinics gradually transfer to 3D technologies, which become irreplaceable in dentistry and prosthetics. To produce the model of the implant in a digital variant, it is only necessary to carry out 3D scanning of patient's tooth cavity. And the creation of the product itself in additive manufacturing proceeds layer-by-layer by adding a portion of the material according to the set model. The used materials represent not a monolithic block as during milling, but a metallic powder. At the same time, 98 % of the powder, which was not used in building, is used for the second time during manufacture of new batches of products. Therefore, it is actually a nonwaste production. The use of modern technologies allows increasing the production volumes without increasing the number of staff.

But most important thing is that except of saving time and costs, modern 3D printers guarantee a high

Table 1. Mechanical properties of titanium alloy and bone [10]

Material	$\sigma_{0.2}$, MPa	σ_t, MPa	σ_{-1}, MPa	$E \cdot 10^{-4}$, MPa	δ, %	ψ, %
Ti–6Al–4V	795	860	400	11.5	10	25
Bone	250	–	200	2.5	0.5	–

accuracy and quality of finished products. Metal additive manufacturing technologies such as SLM and DMLS, are especially effective when creating unique metal parts that are not produced serially, but which are fully functional and produced in a short time. For printing in dentistry, mainly selective laser melting (SLM) is used, which provides the smallest porosity of metal products by a successive fusion of layers of metallic powder using laser. When filling the working chamber by inert gas, a nonoxidable environment is created, which allows using all types of materials, including cobalt-chromium alloys, titanium and many other.

The Company LLC “ALT Ukraine” has an experience in creating 3D-printers that allow creating parts by SLM technology used in such highly critical industries as aircraft and rocket building and precision machine building [1–3].

The production of implants from titanium alloys of medical purpose with the use of additive technologies has the following advantages: providing optimal surface porosity in the form of a spongy structure with recommended parameters for intensive osteosynthesis and osteointegration; formation of transitional surfaces with a set geometry and roughness, creating all conditions in such a way that impede the penetration of infection in the contact zone with soft tissues and skin of a patient; formation of channels of a complex shape inside the implant for inner supply of medicines as well as thin-walled elements; formation of a thread with any profile, including a variable diameter and a step; creation of joining surfaces for quick-replaceable abutments [4].

Thus, designing of equipment and its domestic production, practicing conditions of SLM-technology for manufacture of high-quality products of dental purpose is a relevant task.

STATE-OF-THE-ART OF ISSUE DEVELOPMENT

The Ti–6Al–4V alloy (the known analogues is VT6, Grade 5) is widely used in the field of additive technologies for the manufacture of implants. This alloy is inert in relation to the biological environment [5, 6]. The titanium matrix of a part enters into a comprehensive interaction with tissues, including mechanical, electrochemical, thermal and hydrodynamic [7, 8].

This alloy has low values of the elastic modulus (Table 1), which allows producing porous, spongy, mesh structures of materials close to the properties of human osseous tissue [9].

The parts manufactured by the method of additive manufacturing may have other mechanical properties as compared to a part manufactured by a traditional manufacturing method. Different behaviour can be explained by differences in microstructure [11].

One of the common methods of posttreatment of parts manufactured according to SLM technology is electrochemical polishing [12, 13]. By means of electrochemical polishing, the precision of a part is achieved, which in this case is a thread for placing implant in a jaw bone.

For the manufacture of dental products, small 3D-printers are used, which have a platform size for building of less than 150×150×150 mm. Usually small machines have a relatively small laser power of <200 W and a low speed of build-up. However, the machines of this category have a relatively small area of beam in focus (35–50 μm), and due to this fact, they have a better resolution, smaller deviations from the sizes, higher surface quality and detailing of parts. In [14, 15] a list of investigating and scientific machines and models for realization of selective laser melting technology is given.

The aim of the work was to design and manufacture equipment, practicing printing conditions using SLM technology and electrochemical polishing of dental implants with Ti–6Al–4V.

MATERIAL AND RESEARCH METHODOLOGY

For materials of medical purpose, an important issue is the ratio of biocompatibility, resistance to corrosion and strength. To manufacture dental implants in the work titanium Ti–6Al–4V alloy with the chemical composition, wt.%: 6.21 Al; 4.03 V; 0.04 Fe; 0.1 C; 0.7 O; 0.02 N; Ti — base, was used. The source material was examined by means of a scanning electron microscope REM-106 (Figure 1, *a*) to determine the shape and sizes of particles. Figure 1, *b* shows the results of the analysis.

Examination of microstructure was carried out in optical microscopes Olympus Tokyo and Carlzeiss Axio Vert 200M mat. Study of the samples density was carried out by the microstructural method using the Atlas software and its standard Threshold module with the determination of the area percent occupied by defects.

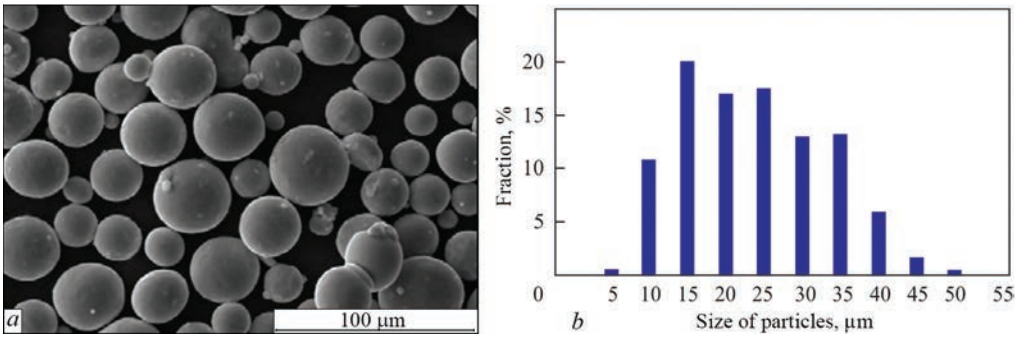


Figure 1. Particles of the source material Ti–6Al–4V (×500) (*a*) and results of granulometric analysis (*b*)

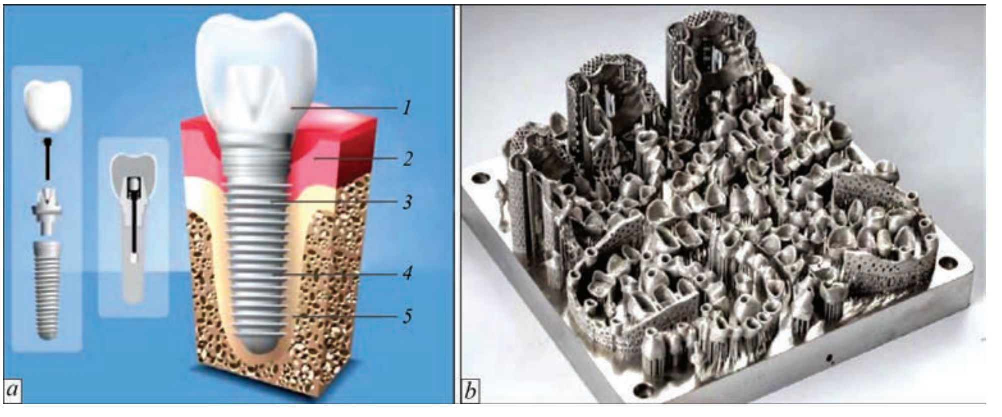


Figure 2. Schematic image of dental implant (*a*): *1* — crown; *2* — gum; *3* — dental implant; *4* — tooth root; *5* — jaw bone [16], implants simultaneously manufactured on the platform (*b*)

Application of modern technologies of additive manufacturing allows not only manufacturing implants as close as possible to the individual parameters of a patient (Figure 2, *a*), simultaneously producing a large number of implants with a different geometry (Figure 2, *b*), achieving high values of mechanical properties due to a unique structure, which is formed at high cooling rates in small melt pools, but also improving osteointegration due to the developed surface between the apexes of a thread.

Printing of samples was carried out in the 3D-printer Alfa-150D of the LLC "Alt Ukraine" production. Electrochemical polishing was carried out in the solution of hydrofluoric acid (HF), nitric acid (HNO_3) with glycerol ($C_3H_8O_3$). Mass control was carried out using analytical scales ADV-2000.

RESULTS OF INVESTIGATIONS

Ultracompact 3D-printer (overall dimensions: 730×700×1818 mm; mass: 450 kg) of the updated Alfa-150D model with a size of the camera of the working field 150×150×180 mm was designed and manufactured (Figure 3). This allows placing it even in a small-sized room. The printer is equipped with a single optical system, a high-precision fiber (ytterbium) laser with continuous radiation and air cooling with a rated optical capacity of 200 W and the diameter of the focal spot of 45 μm was used. The thickness of the layer ranges from 20 to 100 μm, the accuracy of positioning of the laser beam is 0.15 μm, the inert gas consumption (argon) during printing is up to 3 l/min.

Also, among the advantages of the 3D-printer Alfa-150D the following can be noted:

- build-up chamber with the ability to prepare printing process and unpacking of finished products without the use of additional tools for individual protection;
- system of filters regeneration, which allows carrying out the procedure of self-cleaning of filtering

Figure 3. Appearance of 3D-printer Alfa-150 D

Table 2. Experimental conditions of SLM-technology during printing from the powder of titanium Ti–6Al–4V alloy and density of sample metal

Number of sample	Scanning speed, mm/s	Interval between beam passes, mm	Metal density, %
1	1000	0.09	99.85
2		0.10	99.92
3		0.11	99.95
4		0.12	99.99
5	1050	0.09	99.88
6		0.10	99.93
7		0.11	99.98
8		0.12	99.95
9	1100	0.09	99.91
10		0.10	99.95
11		0.11	99.99
12		0.12	99.93
13	1150	0.09	99.92
14		0.10	99.98
15		0.11	99.95
16		0.12	99.88
17	1200	0.09	99.94
18		0.10	99.99
19		0.11	99.3
20		0.12	99.82

elements, significantly reduces the costs and the need to replace gas filters;

● support of inert medium and minimum oxygen content in the build-up chamber, insulation of powder in the feeding hopper from the outer environment to prevent oxidation is particularly important for manufacture of medical devices;

● built-in industrial video camera for continuous monitoring of the process of building and archiving data with a high resolution of shots.

Samples of implants were manufactured by the SLM-technology with the use of titanium Ti–6Al–4V alloy powder on the following experimental technological conditions: constant laser power — 195 W, scanning speed of the laser beam changed from 1000 to 1200 mm/s with a step of 50 mm/s, distance between the beam passes changed from 0.09 to 0.12 mm with a step of 0.01 mm at a constant scanning speed (Table 2), shielding gas — argon was used.

The examination of microsections of main body of samples showed that their average density amounts to 99.9 % (Tables 2, 3).

The rational conditions were selected so, that allow manufacturing parts of Ti–6A1–4V alloy by SLM

Table 3. Microstructure (×100) of Ti–6Al–4V alloy during manufacture by SLM-technology with the use of the following experimental conditions

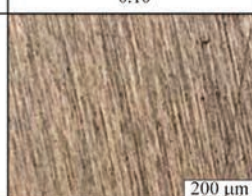
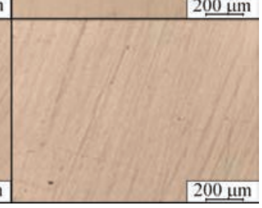
Scanning speed, mm	Interval between laser beam passes, mm			
	0.09	0.10	0.11	0.12
1000				
1050				
1100				
1150				
1200				

Table 4. Conditions of electrochemical polishing of samples

Marking of samples	Current, A	Voltage, V
4	2.5	19
4–2	1.0	15
3–1	1.0	12
6–1	2.0	17
2–2	0.5	12
5–2	1.5	17
3–2	2.0	19
4–1	2.5	20

technology with a high metal density, based on which implants were manufactured with a developed surface between the apexes of a thread according to the model provided by a customer.

An important stage in the manufacture of dental implants is the posttreatment of their surface. Usually, sandblast treatment and electrochemical etching or polishing are used. In the study of implants of world manufacturers [17, 18], it was established that the use of sandblasting leads to the presence of silicon oxide residues on the implant surface, which can negatively affect osteointegration and the formation of osseous tissue.

In the work practicing of conditions of electrochemical polishing of products from Ti–6Al–4V alloy was carried out. Experimental conditions are presented on the ideal volt-ampere curve (Figure 4) and in Table 4.

Based on the results of the analysis of the volt-ampere curve, it was found that zones with optimal polishing conditions were on the samples 6–1, 4–1 and 3–2. According to these conditions, a stable zone of optimal conditions of electrochemical polishing was obtained, this zone was formed in the voltage range from 25 to 30 V, with a current range from 2.0 to 2.5 A. Samples that were subjected to electrochemical polishing with current parameters from 0.5 to 1.0 A have a small zone of stability with a rapid transition to the zone of bubble boiling, which resulted in a point corrosion of the material, followed by a small destruction of the implant thread.

The feature of SLM-technology is the presence of a powder that contacts tracks of a product contour. As a result, powder particles are fused-in into the limiting volumes of metal near the outer surface. In this work,

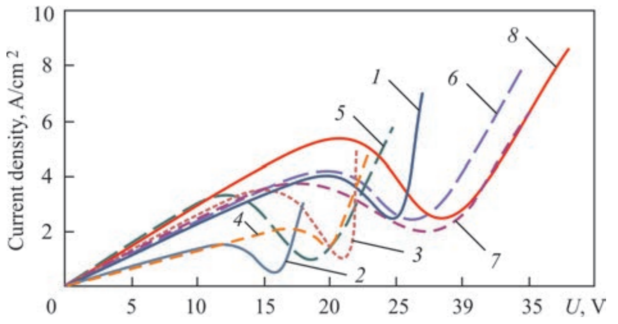


Figure 4. Volt-ampere curves of studied samples: *1* — 4; *2* — 2–2; *4* — 5–2; *6* — 3–2; *7* — 6–1; *8* — 4—1

the control of the mass loss was carried out in order to determine the rational conditions of electrochemical polishing to remove particles without destroying the main part (Figures 5–7).

Based on the received data of mass loss from voltage at a variable current, the diagrams were constructed, from which the highest mass loss intensity occurs after 3 min of electrochemical polishing at a current of 2.5 A and a voltage of 20 V, and during 6 min it occurs in the range of current of 2.0–2.5 A and voltage of 19–21 V (Figure 5).

It was established that an increase in the duration of polishing from 3 to 6 min leads to some increase in the mass loss (Figure 6). At a small voltage of 12 V with an increase in the duration of the process, a slight change in the mass of an implant occurs, indicating that the polishing process occurs according to nonrational conditions, the oxide film on the surface becomes an obstacle to the course of the polishing process. With an increase in voltage, the difference in mass loss after 3 and after 6 min increases. The applied current corresponds to the conditions given in Table 4.

A visual analysis of implant data was performed (Figure 7). It can be seen that at the conditions: current — 1 A and voltage — 12 V (sample 3–1) after 3 min of polishing, on the surface a significant amount of fused-in particles remained, a metallic luster is absent, indicating a high roughness. After polishing during 6 min, the roughness decreased, but it still remained high enough. At the same time, the oxide film of blue colour was formed.

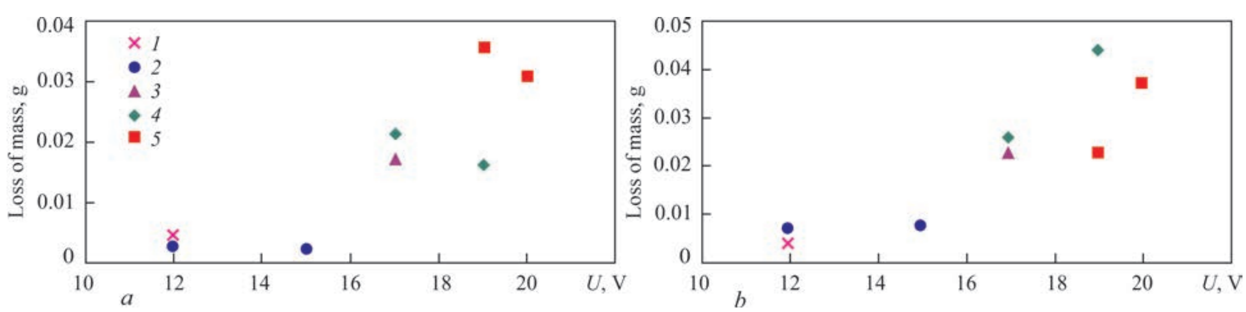


Figure 5. Loss of mass of dental implants under the following experimental conditions of electrochemical polishing: *a* — after 3; *b* — after 6 min: *1* — current — 0.5; *2* — 1.0; *3* — 1.5; *4* — 2.0; *5* — 2.5 A

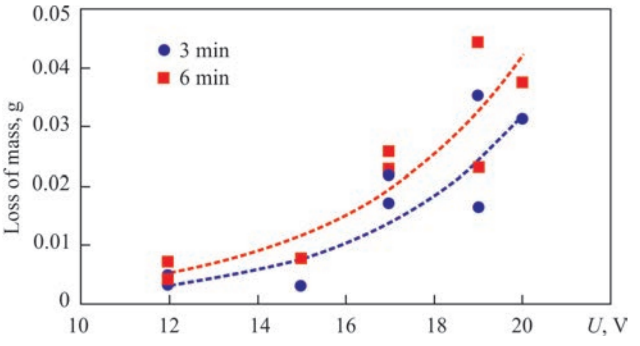


Figure 6. Loss of mass of dental implants in electrochemical polishing depending on duration of treatment

During polishing on the conditions of 2 A and 17 V (for example, sample 6–1) after 3 min of treatment, the implant has a moderate metallic luster, the surface of the thread does not contain a great amount of fused-in particles, the product geometry has a high accuracy. With an increase in the duration of polishing, an excessive etching of the thread near the apex of the implant occurs. As far as this area has a smaller surface area, then all the processes occur most intensively near it.

Application of conditions 2.5 A and 20 V (for example, sample 4–1) already after 3 min of polishing leads to the loss of the thread geometry, and after 6 min, this effect is intensified.

Based on the results of the analysis of the obtained data, rational conditions of electrochemical polishing of dental implants were established, in which the process occurs stably and effectively: current — 2 A, voltage — 17 V, duration — 3 min.

CONCLUSIONS

1. Modern progressive equipment was designed and manufactured by the domestic manufacturer to realize the technology of selective laser melting for using in the dentistry: 3D-printer Alfa-150D manufactured by LLC “ALT Ukraine”.

2. Technological conditions of manufacturing dental implants from Ti–6A1–4V alloy by the technology of selective laser melting were experimentally determined, allowing achieving a density of 99.99 %: laser power — 195 W, speed of scanning – 1000 mm/s, distance between tracks — 0.12 mm.

3. Based on the constructed actual volt-ampere curves and experimental studies, rational conditions (current — 2 A, voltage — 17 V) of electrochemical polishing of dental implants were established to reduce roughness when providing the accuracy of geometry in the region of the thread.

4. The dependence of the mass loss of dental implants was established during electrochemical polishing depending on treatment conditions. It was shown that the pattern of mass loss with an increase in voltage has a nonlinear character. For different exposure

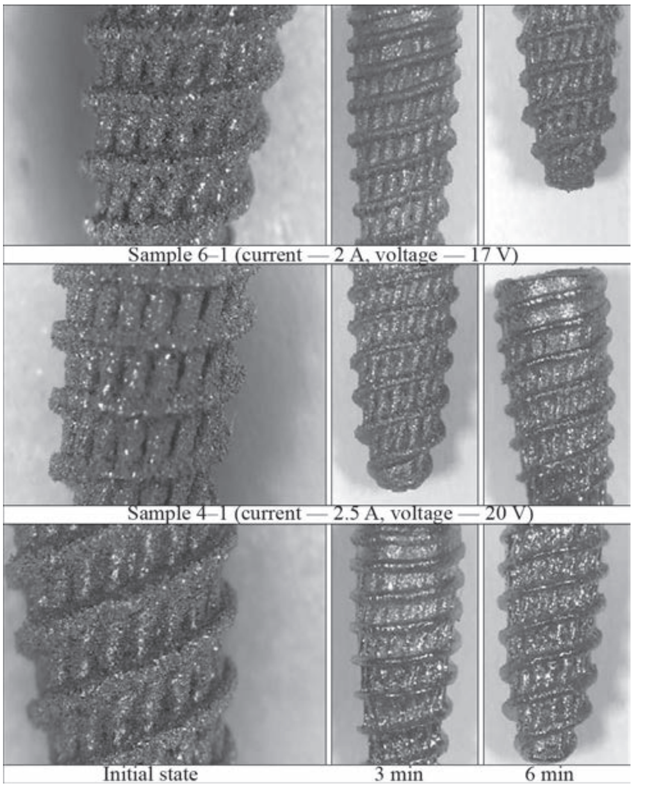


Figure 7. Change in the appearance of samples after electrochemical polishing on the experimental conditions

time in the electrolyte of experimental samples, this pattern has a close character. Moreover, an increased duration leads to a greater mass loss.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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