Selective laser melting of titanium alloy: investigation of mechanical properties and microstructure

A V Agapovichev, V V Kokareva, V G Smelov and A V Sotov
Department of engines manufacturing technology, Samara University, Samara, Russia
E-mail: charming_carrot@mail.ru

Abstract. This article presents the mechanical properties and microstructure of titanium alloy after selective laser melting (SLM). Titanium alloys are ideal material for selective laser melting (SLM), because they are expensive and difficult to machinery using traditional technologies. The application of SLM in the biomedical area has been slow due to the stringent performance criteria and concerns related to personification and part quality. In this article we focused on the manufacture by SLM and determination of microstructure and mechanical properties of titanium alloy (Ti Grade 2 Powder) using tensile tests and X-ray diffraction. The results reveal that the alloy exhibits a pronounced the homogeneous microstructure and high mechanical strength.

1. Introduction
Today titanium alloys with their favorable biocompatibility play an important role for implant applications. The titanium alloys offer good biomaterial properties such as a very high biocompatibility, a density as low as that of bone, high mechanical strength and fatigue resistance, low elastic modulus and good wear resistance. Commercially pure titanium is used preferentially for endosseous implant applications. There are currently four Ti grades and one titanium alloy specially made for dental implant applications. These metals are specified according to ASTM as grades 1 to 5. Grades 1 to 4 are unalloyed, while grade 5, with 6% aluminum and 4% vanadium is the strongest [1]. In Russia, we use commercially pure titanium grades VT1-0 for manufacturing implants. The selection of VT1-0 for medical implantation is determined by a combination of most favorable characteristics including immunity to corrosion, bio-compatibility, strength, low modulus and density and the capacity for joining with bone and other tissue - osseointegration. The mechanical and physical properties of titanium alloys combine to provide implants which are highly damage tolerant. The human anatomy naturally limits the shape and allowable volume of implants. Creating a biomechanical interaction of the implant with human bone tissue is current multidimensional problem, based on the development of technological methods of implants production. It should be taken into account the personalized approach in this direction, since the shape of any individual human bones is a unique.

Selective Laser Melting (SLM), an additive manufacturing technique makes it possible to produce implants which are highly damage tolerant. The human anatomy naturally limits the shape and allowable volume of implants. Creating a biomechanical interaction of the implant with human bone tissue is current multidimensional problem, based on the development of technological methods of implants production. It should be taken into account the personalized approach in this direction, since the shape of any individual human bones is a unique.

Selective laser melting method allows getting in a short time, metal products with complex geometric form, with a uniform distribution of density and homogeneous structure, taking into account the specific physiological human features. This technology provides an opportunity to produce ready-to-use parts with minimal post processing requirements. Moreover, the high cooling rates are resulting in parts with superior properties. However, SLM widespread
application is largely constrained by the lack of own production powders. In medicine, the introduction of additive technology is very promising, as the cost of implants, especially individual, very high, and the decrease in production terms is of great interest in the development and manufacture of new types of implants [5].

The goal of this work is to study the influence of mode SLM process on the structure and mechanical properties of produced samples.

2. Materials and methods
The raw materials for the SLM are powders which are subject to number of requirements [6]: the spherical form of particles, dispersion structure, phase and chemical composition as microcrystalline structure and a particle size distribution, processing characteristics (high flowability, packed density, etc.).

In this paper we used the titanium powder VT1-0 as the raw material. For the elemental composition analysis, we used electron probe microanalysis. Powder structure was investigated in a scanning electron microscope Tescan Vega.

The microstructure of samples, which were obtained by the SLM, depends essentially on the original powder characteristics and processing conditions [7, 8]. The main parameters of laser melting are the laser power (W), scan speed (mm/s), layer thickness (mm), the distance between tracks (mm), the protective atmosphere. The quality of sintered layers is characterized by maximum achievable accuracy, uniform density, the maximum and minimum thickness of the processed layer.

Laser melting was carried out on the installation SLM 280HL with the size of the working chamber 280h280h350 mm. The laser source is YLR-100-SM single-mode CW ytterbium fiber laser (1064–1100 nm). The spot size and maximum laser power are 40 μm and 400 W. The titanium powder particles floated on a titanium substrate.

The study of mechanical properties of the samples was performed on a floor system for fatigue testing model INSTRON 8802. Metallurgical studies have been conducted using a microscope Metam LP-32.

3. Results and discussion
Mass concentration of chemical elements corresponds to reference titanium powder, the powder has a sufficiently high purity, does not contain impurities (Table 1). The maximum particle size of the initial powder was 77.5 μm, particles have an equiaxial regular geometric shape and an average particle size is 50 μm. The flowability of titanium powder was 6 seconds/100g. Figure 1 shows the surface morphology of the titanium initial powder particles VT1-0.

![Surface morphology of titanium powder](image_url)

**Table 1. Chemical composition of the initial powder**

| Element         | Mass concentration of chemical elements (%) | Sample (%)     |
|-----------------|---------------------------------------------|----------------|
| Ti (base)       | 99.68                                       | 99.5 – 99.99   |
| Other impurities| 0.32                                        | 0.01-0.5       |

SLM offers several advantages compared to conventional production techniques, such as reduction of production steps, a high level of flexibility, a high material use efficiency and a near net shape production. Furthermore, hard materials or materials with a high melting point can be processed with SLM. Most important, because of the layer-wise building, SLM enables the production of parts with a high geometrical complexity. However, the unique conditions during the SLM process give rise to some problems. Because of the short interaction times and accompanying highly localized heat input, large thermal gradients exist during the process. These lead to the build-up of thermal stresses, while the rapid solidification leads to segregation phenomena and the development of non-equilibrium phases. Moreover, non-optimal scan parameters may cause melt pool instabilities during the process, which leads to an increased porosity and a higher surface roughness. To ensure optimal building
conditions, the influence of the different process parameters, such as layer thickness, scan spacing, scan strategy, laser power and scan speed on the microstructure and mechanical properties and other properties such as the density and surface quality have been investigated.

Figure 1. The surface morphology of the titanium powder VT1-0.

As a result of working out the SLM optimal processing conditions we manufactured rectangular cross section samples, the plate with a length 70 mm, width 2 mm and height 10 mm. The processing parameters under consideration included laser power (150, 165 W), scanning speed (100-500 mm/s). The amount of transmitted energy was evaluated by using the linear energy density (LET) radiation power equal to the ratio (P) to a scanning speed V (LET = P/V).

The range of the parameter LET amounted 0.30 ... 1.65 J / mm, the scanning speed was determined in increments of 50 mm / s. The main parameters of the SLM are given in Table 2.

### Table 2. Parameters of Selective Laser Melting

| No. Processing conditions | Scanning speed (mm/s) | Laser power (W) | Linear energy density (J / mm) |
|---------------------------|-----------------------|-----------------|-------------------------------|
| 1                         | 100                   | 165             | 1.65                          |
| 2                         | 150                   |                 | 1.10                          |
| 3                         | 200                   |                 | 0.83                          |
| 4                         | 400                   |                 | 0.38                          |
| 5                         | 450                   | 150             | 0.33                          |
| 6                         | 500                   |                 | 0.30                          |

The Figure 2 shows a process of selective laser melting titanium of powder on SLM 280HL machine. The growing of samples was carried out in of argon, the thickness of the layer construction of 50 µm. Time of samples manufacturing was 2 h. Figure 3 shows the samples produced. Also we made individual prototypes of small joint implants; 3D models are produced as a result of treatment CT scans of individual patients in the Samara State Medical University (Figure 3). Due to the high values of the grown samples roughness (Rz 20), surface requires subsequent machining of the contact surfaces of implants small joints.
Figure 2. SLM process of titanium powder on the SLM 280HL machine.

Figure 3. Samples were produced by SLM process.

All tensile test samples of the SLM material were tested for each treatment to determine the mechanical properties in accordance with ISO 5832-2-2014. Results of mechanical tensile tests on the samples produced by the SLM are given in Table 3. The test results show that ultimate stress limits of SLM-alloy exceed the ultimate stress limit of the titanium alloy obtained by traditional technologies, reference material (200-680 MPa, ISO 5832-2-2014). The SLM material is much stronger than the reference material. This is true for all material processed by SLM because the rapid cooling conditions always lead to a fine microstructure.

Table 3. Mechanical properties of samples.

| No. Processing conditions | Ultimate stress limit, $\sigma$ (MPa) | Relative extension, $\varepsilon$ (%) |
|---------------------------|--------------------------------------|-------------------------------------|
| 1                         | 717                                  | 21.7                                |
| 2                         | 732                                  | 19.0                                |
| 3                         | 750                                  | 16.7                                |
| 4                         | 740                                  | 19.0                                |
| 5                         | 708                                  | 17.3                                |
| 6                         | 702                                  | 12.5                                |

The dependence of ultimate stress limit from the technological selective laser melting parameters is shown in Figure 4.
The graph shows that the best value of the ultimate stress limit of material reaches at a laser scanning speed of 200 mm/s. In this paper we show the material microstructure obtained by titanium powder melting at six different processing conditions (Table 2). Metallographic studies showed homogeneity of the structure in the presence of small pores. The pore size for the adopted processing conditions of melting observed in the range of 10-50 μm (Figure 5).

4. Summary
The SLM technology is a successful method of melting titanium directly by consolidating powder mixtures of commercially pure metals. In this research the change in surface morphology of SLM titanium with process parameters, such as laser power, scan speed was investigated to control the pore structure. Following results were obtained:

1) The powder is characterized by high purity, does not contain harmful impurities, which is an important requirement of the medical industry. The presence of fraction 35-50 μm does not prevent normal formation layer and does not reduce the flowability of the powder.

2) Titanium samples and prototypes of small joint implants. The quality of sintered layers is characterized by uniform density, the strength of 750 MPa, a spherical particle shape. SLM technology provides the high mechanical properties of samples, and has good prospects for further receiving implants of titanium alloys.
The results presented in the paper are the basis of research and development aimed at improving the SLS process and enhances the performance properties of the implants produced. [9] Next important step is the selection of the optimal parameters of selective laser melting and subsequent processing as by experimentation and mathematical modeling techniques that will improve and accelerate the process of creating a product with the specified requirements, including the desired roughness of samples [10]. To improve the quality of titanium surfaces samples we choose the suitable post-processing by methods of electrochemical machining [11].

Then we plan to carry out complex research aimed at the formation of biocompatible coatings on the samples in order to improve the functional characteristics of the implant, and experiments on the biocompatibility of implants manufactured prototypes.

Acknowledgments
The work described in this article is made at the equipment of Collective Use Center of CAM-technologies (a unique cipher RFMEFI59314X0003 agreement).

References
[1] Abdel-Hady Gepreel M and Niinomi M 2013 Biocompatibility of Ti-alloys for long-term implantation Journal of the Mechanical Behavior of Biomedical Materials 20 407-15
[2] Attar H, Löber L, Funk A, Calin M, Zhang L, Prashanth K, Scudino S, Zhang Y and Eckert J 2015 Mechanical behavior of porous commercially pure Ti and Ti-TiB composite materials manufactured by selective laser melting Materials Science and Engineering 625 350-56
[3] Popovich A, Sufiiarov V, Borisov E and Polozov I 2015 Microstructure and mechanical properties of Ti-6AL-4V manufactured by SLM Key Engineering Materials 651-653 677-82
[4] Sufiiarov V, Popovich A, Borisov E and Polozov I 2015 Selective laser melting of titanium alloy and manufacturing of gas-turbine engine part blanks Tsvetnye Metally 2015, 8 76-80
[5] Frazier W E 2014 Metal additive manufacturing: A review Journal of Materials Engineering and Performance 23 1917-28
[6] Sallica-Leva E, Jardini A and Fogagnolo J 2013 Microstructure and mechanical behavior of porous Ti-6Al-4V parts obtained by selective laser melting Journal of the Mechanical Behavior of Biomedical Materials 26 98-108
[7] Smelov V, Sotov A and Murzin S 2016 Particularly selective sintering of metal powders by pulsed laser radiation Key Engineering Materials 685 403-07
[8] Khaimovich A and Balaykin A 2014 Analysis of titanium alloys plastic properties under severe deformation conditions in machining ARPN Journal of Engineering and Applied Sciences 9 10 1828-33
[9] Agapovich A, Balaykin A and Smelov V 2015 Production technology of the internal combustion engine crankcase using additive technologies Modern Applied Science 9 4 335-43
[10] Bose S, Vahabzadeh S and Bandyopadhyay A 2013 Bone tissue engineering using 3D printing Materials Today 16, 12 496-504
[11] Nekhoroshev M, Pronichev N and Smirnov G 2014 Computer simulation of high-speed anodic dissolution processes of geometrically-complex surfaces of GTE details Open Mechanical Engineering Journal 8 436-40