Structure and properties of Ti-6Al-4V titanium alloy products obtained by direct laser deposition and subsequent heat treatment

O G Klimova-Korsmik¹, ², G A Turichin², S A Shalnova¹, M O Gushchina¹, ², V V Cheverikin³
¹Peter the Great Saint-Petersburg Polytechnic University, Saint Petersburg, Russia.
²Saint Petersburg State Marine Technical University, Saint Petersburg, Russia.
³National University of Science and Technology “MISiS”, Moscow, Russia

Abstract
A large number of studies have been carried out the effect on the structure and properties of titanium alloys produced by the DLD technology. Part of the research is devoted to the special features of the influence of various process parameters on the properties and quality of final products [1, 2]. Thus, obtaining products from Ti-6Al-4V using DLD technology has attracted increasing attention in recent years. But the technology of direct laser deposition is constantly being modernized and improved. The quality of laser radiation improves, as well as new laser heads are designed to increase the productivity of the process and improve the properties of the finished product. In this work will be studied structure and properties of Ti-6Al-4V titanium alloy products obtained by direct laser deposition and subsequent heat treatment.

1. Introduction
Today additive technologies occupy a worthy place in the most important spheres of human activity, such as aircraft building, shipbuilding and medicine. It is difficult to imagine an industry in which methods of additive manufacturing cannot be used. There are many AM methods that can be distinguished by the type of heating source (laser, arc, electron beam), the method of supplying the building material (direct or layered), material (powder, wire, liquid)[3].

Direct laser deposition is based on the principle of direct supply of powder material into the zone of action of the laser beam. Gas powder jet gets to action zone of laser beam, the molten pool is formed, then crystallization occurs and a layer with required thickness is formed. The dimensions of the products are limited only by the grip area of the manipulator, which uses a 5-axis anthropomorphic robot, and a special rotary table provides the sixth axis of displacement. Thus, it is possible to manufacture products with a profile of varying complexity [2, 4].

The main of promising materials for use in the technology of direct laser deposition is titanium alloys, which are distinguished by high corrosion resistance and low weight. The Ti-6Al-4V refers to α+β phase in a stable state contain 6% of the β phase. These alloys are characterized by a good ratio of strength and plasticity. Their wide use in engineering and the high cost of raw materials prompts the use of new technologies to produce products from titanium alloys to reduce the cost of subsequent machining, as well as material savings. A large number of studies have been carried out the effect on the structure and properties of titanium alloys produced by the DLD technology. Part of the research is devoted to the special features of the influence of various process parameters on the properties and quality of final products [2, 5]. The influence of power, speed, distribution of laser radiation, filling strategies and heat treatment on the properties and structure of titanium alloys in the process of DLD has been studied in the different studies. The products of the aviation and shipbuilding industries have big size and a complex form. The products of the aviation and shipbuilding industries have large dimensions and a complex profile. High values of power and speed are used to increase productivity, which leads to the formation of nonequilibrium structures that impair the mechanical characteristics of products. Due to the continuous improvement of the method of direct laser deposition, it is possible to increase the process speeds with the formation of a stable two-phase structure of titanium products. In this work will be investigated structure and mechanical characteristics of Ti-6Al-4V titanium alloy.
products obtained by direct laser deposition and subsequent heat treatment. In this work will be considered the as-deposited sample made with scanning by the welding head “FLW D30W Wobble + seam tracking module” made by IPG. In addition, there are three samples: as deposited without scanning, after heat treatment and after HIP. The positive effect of this method on the characteristics and phase composition of the samples is shown. A comparative analysis of the structure and phase composition of the samples obtained with and without scanning by a beam is carried out.

2. Equipment and materials
The samples have been carried out at laser complex for direct metal deposition based on fiber laser LS-5 (complex for direct laser deposition - CDLD). The CDLD also includes a six-axis robot manipulator, a two-axis pan-tilt positioner and a sealed chamber with a volume equal to 6 m³. The working tool is a laser welding head FLW 50 made by IPG with a co-axial nozzle with an exit diameter of 2-3 mm [6], also welding head “FLW D30W Wobble + seam tracking module” made by IPG was used to provide a linear scan of the track. This welding head allows to obtain products with a wall thickness of 3 mm in one pass. The installation is shown in Figure 1a,b:

Figure 1. (a) Robotic complex for direct laser deposition, (b) grown samples for research, (c) fill pattern

| Table 1. Parameters of the process |
|-----------------------------------|
| **Mode**          | **Power, W** | **Velocity, V/sec** | **Beam’s diameter, mm** | **Powder rate, g/min** | **dz, mm** | **dx, mm** |
|-------------------|--------------|---------------------|-------------------------|------------------------|-----------|-----------|
| As-deposited      | 2200         | 40                  | 3.5                     | 27                     | 0.8       | 2         |
| As-deposited with Wobble | 2200         | 30                  | 3.0                     | 25                     | 0.6       | -         |
| Heat treatment    | 2200         | 40                  | 3.5                     | 27                     | 0.8       | 2         |
| HIP               | 2200         | 40                  | 3.5                     | 27                     | 0.8       | 2         |

The grown plates formed samples are made using the strategy of the layer filling (Fig. 1c). The first layer is formed by successive passes of the tool, where the start and end points of the passage are on one side of the plate. As-deposited samples with Wobble are a thin wall 3 mm thick obtained in a single pass of the laser head.

The building material was a Ti-6Al-4V powder-titanium alloy. The distribution of powder particles is uniform, the fraction composition is d_{10}=45μm, d_{50}=65μm, d_{90}=90μm. The shape of the particles is spherical (Fig. 2a). There are rare individual particles of irregular shape. The distribution of powder particles is uniform (Fig. 2b), the fraction is 30-100 μm.
Figure 2. (a) The surface of the powder of Ti–6Al–4V, (b) particle size distribution of the powder

The powder studies showed the absence of internal oxide and nitride inclusions. In addition, pores, gas cavities and large non-metallic inclusions are absent in the structure.

Heat treatment mode: holding for 1 hour at 825, cooling in water, then holding at 500 for 4 hours, cooling in air. This heat treatment mode is standard for cast products from Ti-6Al-4V alloy.

Hot isostatic pressing was carried out at a temperature of 910-930°C, pressure 1000-1060 atm. for 2.5 hours.

Metallographic studies of the samples were carried out on a DMI 5000 (Leica) microscope with the Tixomet software to determine samples’ defects. There were etched in a solution of 100 ml H2O, 2 ml HNO3, 1 ml HF in order to study the microstructure.

The samples were cut from the plates by the method of electroerosion cutting in order to implement mechanical tests for static stretching in the transverse and longitudinal sections.

The tests were carried out for uniaxial tension on a Zwick unit to determine the mechanical characteristics of the samples. An explore of the phase composition and structural parameters of the samples was carried out on a Bruker Advance D8 diffractometer on CuKα-radiation. The phase composition analysis was carried out using PDF 4+ databases, and the POWDER CELL 2.4 full-profile analysis program.

3 Results

3.1 Structure analysis

Titanium alloy Ti-6Al-4V refers to two-phase $\alpha + \beta$ alloys. It is characterized by the lamellar structure of $\alpha$ phase separated by $\beta$-phase interlayers inside the $\beta$-grain. In the process of casting, Ti-6Al-4V alloy is prone to intensive grain growth and anisotropy of characteristics, which worsens the mechanical characteristics of the alloy. Titanium samples obtained by DLD in a different state were studied: as-deposited samples, as-deposited + heat treatment, as-deposited + HIP, as-deposited with “Wobble” scanning (Figure 3).
Depending on the state of the sample, it is possible to observe various sizes of grains. It is obvious that the grain size in the as-deposited state is significantly larger than the size of the prior $\beta$-grains in as-deposited with Wobble scanning samples. The smallest proportion of length and width of grains is the sample as-deposited with Wobble and after the HIP - the grain size can be controlled by subsequent processing of the samples. The description of the structure shows that the size of the prior $\beta$-grains decreases depending on the type of heat treatment, and the phase composition of the samples can be changed using heat treatment (figure 5).

The scheme of grain size measurement is shown in Figure 4(a). The formation of grains in the structure of the sample without scanning is uneven: the equiaxed grains are at the boundaries of the wall, but in the middle of the sample the grains are stretched toward the superposition of the layers. The average grain width is from 40 to 230 $\mu$m (Figure 4 (b)).

The formation of grains in the structure with scanning is more uniform (Figure 4 (c)): equiaxial grains are located over the entire surface of the sample, more elongated grains towards the heat removal can be seen in the middle of the sample, the average grain width is from 100 to 175 microns. Figure 5 shows the microstructure images of the samples.
Figure 5. Microstructure of the samples: (a) as-deposited (b) as-deposited with Wobble (c) after heat treatment (d) after HIP

The structure of samples obtained by the DLD process in as-deposited state is characterized by thin acicular fine α-phases located perpendicular to each other (martensitic structure), also X-ray phase analysis shows the presence of α + α'-phase, which is formed during rapid cooling from the β-phase, there is no β phase. Samples as-deposited state are also characterized by the occurrence of processes with the formation of metastable phases, such as α', ω in as-deposited state. These phases largely embrittle the material [9 Using heat treatment and HIP, it is possible to achieve an equilibrium phase composition corresponding to the α + β Ti-6Al-4V alloy, which is confirmed by X-ray phase analysis. (Figure 6). Fine basket weave α lath structures are commonly observed in the samples after heat treatment and HIP made by direct laser deposition. Also, the samples obtained with Wobble scan contain α + β phase, the content of β-phase reaches 5%. The structure of the sample as-deposited with Wobble is characterized by a martensitic structure with a thicker α lath than the sample in as-deposited state.
Fig. 6b shows the XRD pattern for the as-deposited material. Since there is no JCPDS standard for this material, and the known results from the literature are not sufficiently numerous for a detailed comparison, we will fit our results to those reported for the hexagonal α-Ti (JCPDS file #44-1294) and for the cubic β-Ti (JCPDS file #44-1288). The β phase is clearly characterized by the (110) and (200) reflections, with $d_1=0.2284$ nm and $d_2=0.1614$ nm, respectively (Fig. 6a). [7].

The absence of the β phase in the as deposited is confirmed by the X-ray diffraction pattern in Fig. 3b. The analysis showed the presence of only the α-phase peaks, the lattice parameters of the α phase and the α'-phase coincide, and the location of the intensities as in Fig. 6c makes it possible to assume a high content of the α'-phase in the as deposited state [8]. In the as-deposited samples and after HIP the β phase content is increasing.

3.2 Mechanical tests results
The mechanical characteristics are shown in Table 2. Also in the table are the values of the microhardness of the samples, it is noted that all samples have a hardness higher than the standard.

|                | Strength, [MPa] | Relative elongation, [%] | Microhardness, HV |
|----------------|-----------------|--------------------------|-------------------|
| As-deposited    | 1309            | 1.6                      | 435               |
| As-deposited +  | 1135            | 10.0                     | 380               |
| Wobble          | 1227            | 4.6                      | 424               |
| after heat treatment | 1084     | 15.0                     | 386               |
| after HIP       | 885             | 8.0                      | 293-361           |

The mechanical characteristics of tensile tested as-deposited specimens have the highest tensile strengths as compared to the rest of the samples, but the value of the relative elongation is lower than the value represented in GOST. All samples have the values of strength higher than stated in the GOST. The best ratio between the strength and the relative elongation is the as-deposited + Wobble and after HIP samples. Microhardness measurements were made in the layer and between layers for as deposited and as deposited with Wobble samples. Figure 7 shows the graphs of the hardness distribution over the width of the sample.
Figure 7. Microhardness of the samples: (a) as-deposited in the layer (b) as-deposited between layers (c) as-deposited with Wobble in the layer (d) as-deposited with Wobble between layers

The hardness is distributed non-uniformly along the width of the sample in the layer and in the overlap of the layers in samples without scanning. The samples obtained with the scan of the wobblers have a high hardness uniformly distributed over the entire surface of the sample.

Conclusions
In this study, the effect of mechanical characteristics and phase composition of the samples formed using by welding head FLW D30W Wobble + seam tracking module produced by IPG on the compared to the properties of the samples in the as-deposited state, after HT and HIP will be considered. Four types of samples were considered: as-deposited, as-deposited with Wobble, after heat treatment and HIP. It is noted that grain formation occurs in the direction of heat removal in as-deposited samples, as-deposited with Wobble, this orientation also persists in the samples after heat treatment and HIP. The study of the structure showed that the size of the prior β-grains decreases depending on the type of heat treatment, and the phase composition of the samples can be varied using the heat treatment. The microstructure of the samples is considered: in the as-deposited state, the acicular α-phase is characterized, and the X-ray phase analysis shows the presence of the α + α'-phase, which is formed during rapid cooling from the β-phase zone. As-deposited with Wobble sample, α + β phases are formed, the structure is also martensite. By applying heat treatment and HIP, it is possible to achieve the phase composition corresponding to the two-phase α + β Ti-6Al-4V alloy.

The investigation of the mechanical characteristics of the samples showed low values of the relative elongation for as-deposited and after heat treatment samples. HIP is most suitable heat treatment for improving of mechanical characteristics. As-deposited + Wobble and after HIP samples have the best ratio between the strength and the relative elongation. Also, the microhardness values are close to the standard values in these samples. The use of "Wobble" scanning allows achieving high capacity, a defect-free structure, as well as a sample thickness of up to 3.5 mm in one process pass. Uniform distribution of hardness, as well as high value of the mechanical characteristics of the sample allow the use of products with scanning of Wobble without additional heat treatment.
References

[1] Jayme S. Keist, Todd A. Palmer Role of geometry on properties of additively manufactured Ti-6Al-4V structures fabricated using laser based directed energy deposition Materials and Design 106 (2016) 482–494

[2] Sklyar M. O., Klimova-Korsmik O. G., Cheverikin V. V., Formation Structure and Properties of Parts from Titanium Alloys Produced by Direct Laser Deposition // Solid State Phenomena, 2017, Vol. 265, Pp. 535-541.

[3] Nima Shamsaeia, Aref Yadollahi, Linkan Bianc, Scott M. Thompsona, An overview of Direct Laser Deposition for additive manufacturing; Part II: Mechanical behavior, process parameter optimization and control Additive Manufacturing 8 (2015) 12–35

[4] G.A. Turichin, O.G. Klimova, E.V. Zemlyakov, K.D. Babkin, D.Yu. Kolodyazhnyy, F.A. Shamray, A.Ya. Travyanov, P.V. Petrovskiy, Technological aspects of high speed direct laser deposition based on heterophase powder metallurgy, Physics Procedia 78 (2015) 397–406.

[5] Jingjing Yang, Fangzhi Li, Zemin Wang, Xiaoyan Zeng Cracking behavior and control of Rene 104 superalloy produced by direct laser fabrication Journal of Materials Processing Technology 225 (2015) 229–239

[6] Turichin, G.A. High-Speed Direct Laser Deposition: Technology, Equipment and Materials/ Somonov, V.V., Babkin, K.D., Zemlyakov, E.V., Klimova, O.G. // – IOP Conference Series: Materials Science and Engineering. – №125, 2016. — P. 245–254

[7] X-ray diffraction measurements of plasma-nitrided Ti–6Al–4V S.L.R. da Silva, L.O. Kerber, L. Amaral, C.A. dos Santos * Instituto de Física —UFRGS, Av. Bento Gonçalves, 9500, C.P. 15051— Campus do Vale, 91501-970 Porto Alegre RS, Brazil

[8] G.A. Turichin Features Of Structure Formation In α+β Titanium Alloys. O.G. Klimova-Korsmik, R. S. Korsmik, M.O. Guschkina,b, S.A. Shalnova, Cheverikin, A.S. Tataru—10th Conference on Photonic Technologies – LANE 2018 — September 03 - 06, 2018 Fürth, Germany

[9] M. Neikter, P. Åkerfeldt, R. Pederson, M.-L. Antti Microstructural characterization and comparison of Ti-6Al-4V manufactured with different additive manufacturing processes Materials Characterization 2018