Structure and properties of near-α titanium products obtained by direct laser deposition and heat treatment

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Abstract. Advanced techniques of obtaining products require careful selection of materials for various industries. Titanium alloys are widely used in the aerospace, shipbuilding and mechanical engineering industries. The development of near-α titanium alloys should be considered a significant achievement in the field of metallurgy and heat treatment (HT) of titanium alloys. This article presents a study carried out with the aim of optimizing heat treatment modes for high-temperature titanium alloys obtained by direct laser deposition (DLD). Heat treatment was carried out in the temperature range (700-1000°C), covering three typical temperature ranges, i.e. the temperature range for the partial decomposition of martensite, the temperature range for the complete decomposition of martensite, and the phase transformation temperature were subsequently selected as the heat treatment temperatures. Based on metallographic analysis, the influence of heat treatment modes on the structure, as well as the tensile properties at room temperature, of TA15 titanium DLD-samples.

1. Introduction
Additive manufacturing technologies provide new opportunities for product development and allow the manufacture of products with complex shapes. Today, one of the promising areas of manufacturing large-sized parts is additive technology, namely the method of direct laser deposition (DLD). The advantage of direct laser deposition is that this method allows increasing the productivity and, accordingly, reducing the manufacturing time of parts [1-4]. Especially advantageous is the use of this technology in the case of complex products, when multi-stage design and engineering, creation of demonstration samples in a short time [5-8].

Titanium alloys are one of the main structural materials used in various industries. Widespread use is due to the properties inherent in titanium alloys - high specific strength, corrosion resistance, non-magnetic, low density, and good heat resistance at operating temperatures from 250 to 600°C. Currently, the aircrafts and shipbuilding industry is considered the largest consumer of titanium alloys [9]. In recent years, the use of titanium and its alloys in chemical, transport and food industry, equipment for oil and gas industry, medicine, construction and other fields has significantly increased [10-12]. The development of near-α (near-α) titanium alloys should be considered a significant achievement in the field of metallurgy and heat treatment of titanium alloys. Near-α titanium alloys belong to the class of structural titanium alloys of medium strength and have a higher technological ductility than α-titanium alloys [13]. Increased ductility of near-α alloys is achieved by the formation of a small amount of plasticizing β-phase in α-matrix [14-15].

In this paper the samples from TA15 (Ti-6Al-2Zr-1Mo-1V) titanium alloy obtained by direct laser deposition are considered. Given the complex combination of alloying elements in the TA15 alloy, it can be assumed that the reserve for increasing its strength and plastic properties can be realized by varying the mode of heat treatment. The purpose of this work is to study the possibility of obtaining
satisfactory mechanical properties while preserving high strength characteristics of the DLD TA15 alloy by selecting the mode of heat treatment.

2. Equipment and materials

2.1. DLD process

The custom robotic complex for direct laser deposition was used for sample production (SMTU, Russia). During the deposition process, the building chamber was filled with protective gas, argon. Technological mode of DLD: Laser power 2200W, scanning speed 20 mm/sec, layer thickness 3 mm, width offset 1.5 mm, height offset 0.8 mm, powder consumption 20 g/min.

2.2. Materials

For the DLD, plasma atomized TA15 (Ti-6Al-2Zr-1Mo-1V) titanium powder was chosen (fig. 1a), the fractional size distribution (fig. 1b) is 40-140μm. Scanning electron microscopy (SEM) Tescan Mira3 (TESCAN, Czech Republic) equipped with an Energy Dispersive X-Ray (EDX) detector and the console Oxford AZtec (Oxford Instruments NanoAnalysis, United Kingdom) was used to determine chemical composition of titanium powder TA15 (Table 1).

![Figure 1](image)

**Figure 1.** (a) Surface of TA15 powder particles; (b) Fractional size distribution

| Table 1. Chemical composition of titanium powder TA15 |
|----------------|---------|---------|-------|------|------|
|                | Ti, wt.% | Al, wt.% | Zr, wt.% | Mo, wt.% | V, wt.% |
| EDX            | 87.0     | 6.9      | 2.3      | 1.8     | 2.0    |

2.3. Heat treatment

In this work, heat treatment was carried out using a retort furnace SNOL type with a chamber made of thermal fiber plates (SNOL, Russia). The heat treatment modes were the holding time was 1, 2, 5 and 8 hours at different temperatures: 700°C, 900°C, 1000°C. The heat treatment was carried out in air with cooling in a furnace to 300°C and further cooling in air.

2.4. Microstructure analysis

To analyze the microstructure, polished sections were prepared. The samples surfaces were greeted using SiC grit papers up to 2500-grit and further polished with Aluminum suspension of 1 μm and final polishing with colloidal silica of 100 nm. Additionally, the sample surfaces were etched using the solution of 1 ml HF + 2 ml HNO3 + 97 ml H2O. Scanning electron microscopy The SEM images were obtained on polished samples using the backscattered electron mode to study microstructure and secondary electron mode for investigation of fracture surface.

2.5. Microhardness and mechanical test
The microhardness of the samples was measured according to Vickers (HV) on polished sections using a Future Tech FM-310 microhardness tester (Future Tech, Japan) in accordance with ISO 6507-1-2007. For each sample, 20 measurements were made at a load of 500 g. For tensile testing, specimens were cut along the deposited layers. The tensile tests were carried out on the universal tensile testing machine Zwick Roell Z100 (Zwick/Roell, Germany) according to ASTM E8. The speed of tension was $1.0 \times 10^{-3}$ s$^{-1}$. The geometry of the samples had a cylindrical gage section with a diameter of 5 mm and a gage length of 55 mm.

3. Results

3.1. Microstructure analysis

Figures 2-5 show the microstructure formation of the samples in the as-deposited sample and after heat treatment (HT). Thus, 13 samples were examined. The influence of the holding time on the formation of the structure at three different temperatures is considered: the 700°C temperature range for the partial decomposition of martensite, the 900°C temperature range for the complete decomposition of martensite, and the 1000°C phase transition temperature.

![as-deposited](image)

Figure 2. As-deposited sample, magnification x7500, SEM- BSE

The acicular $\alpha' + \alpha$ martensite phase formed in the DLD TA15 alloy (Fig. 2). It was transformed into the Widmannstatten structure after solution in the single $\beta$ phase region followed by rapid cooling, the dimensions of the lamellas are 1.13 µm in length and 0.23 µm in width. This structure is characterized by high strength and low elongation.
Figure 3. The effect of holding time on microstructure at temperature 700°C, magnification x5000, SEM- BSE

The partial decomposition temperature 700°C of the martensite structure, the content of α' + α martensite phase practically does not change: the size of the α-phase plates’ length slightly increases from 2.91 μm to 3.21 and 0.30 μm to 0.39 μm in width for 1 and 8 hour, respectively (Fig. 3a, d).
Figure 4. The effect of holding time on microstructure at temperature 900°C, magnification x5000, SEM- BSE

Heat treatment at 900°C (Fig. 4) (the complete decomposition temperature of martensite) leads to decrease in the content of α’+ α of the martensite structure, and plates of the α-phase grow. Length from 3.98 μm to 7.06 μm for 1 and 8 hours, respectively, width does not change 1.05 μm and 1.06 μm for 1 and 8 hours, respectively. After holding for 8 hours in the space between the plates of the α-phase, a secondary α-phase is formed.

![Figure 4](image)

Figure 5. The effect of holding time on microstructure at temperature 1000°C, magnification x5000, SEM- BSE

At temperatures above the polymorphic transformation, secondary α-phase is formed even with holding time of 1 hour (Fig. 5a). However, in the interval between the α-plates, the secondary α-phase begins to grow in the spaces between α-phase. With 1h of HT, barely noticeable needles of the martensitic secondary α-phase are formed (Fig. 5 b-d). With an increase in HT time, the secondary α-phase increases up to a size of 1.97 μm in length and 0.65 in width for a heat treatment time of 8 hours.

3.2. Microhardness
The effect of heat treatment holding time and temperature on the microhardness of the DLD TA15 alloy is shown in Fig.6.
As-deposited sample microhardness is 420HV.

At 700°C (fig. 6a), the microhardness decreases depending on the holding time, but upon reaching 8 hours it again increases to 400HV, which may be associated with the formation of a secondary α-phase.

At 900°C (fig. 6b), with increasing time, the microhardness decreases and remains approximately the same for 1, 2 and 5 hours - 370 HV, with an increase in the holding time up to 8 hours, the hardness significantly increases, to the value of the as-deposited sample microhardness (410 HV).

At 1000°C (fig. 6c), the microhardness decreases significantly for 1 hour of exposure, and then gradually increases and remains unchanged for 2.5 and 8 hours of exposure at 395 HV.

3.3. Mechanical tests results
Based on the results of the analysis of the microstructure and microhardness, the HT sample 900 °C 1h was selected for tensile testing. Because, in the sample (900°C 1hour) an equilibrium phase is formed and the microhardness is reduced during the heat treatment. The results of mechanical tests are presented in Table 2.

|                  | Yield stress, MPa | Ultimate Stress, MPa | Elongation, % |
|------------------|-------------------|----------------------|---------------|
| HT sample 900°C 1h | 900               | 1041                 | 6.0           |
| As-deposited sample | 986               | 1040                 | 3.0           |

As a result of heat treatment, it was possible to increase the value of the relative elongation, without losing the strength of the material. Fig. 7 shows the fractures surface.
The fracture surface of the as-deposited sample is characterized by brittle fracture; fracture HT sample 900 °C 1h is a ductile-brittle fracture mechanism.

4. Conclusion

In the course of this work, the study of the effect of heat treatment on the structure and properties of products from metal powders of the TA15 near-α titanium alloy obtained by direct laser deposition was carried out. Three main temperatures and different heat treatment furnace holding time were considered.

Heat treatment at 700°C does not change the nature of the microstructure, however, during the HT process, the hardness decreases in comparison with the as-deposited sample after holding time for 5 hours, which may be associated with the removal of internal stresses.

Heat treatment at 900°C leads to a decrease in hardness with an increase in α-plates, when the holding time of 1 hour is reached, the formation of a secondary α-phase occurs with a simultaneous increase in the hardness of the material.

In the process of heat treatment at 1000°C, the formation of the secondary α-phase between the plates of the α-phase occurs from the first hour of exposure, to the end of heat treatment (with holding time of 8 hours), while the hardness of the material for all options of holding time is about 395 HV. Mechanical properties were determined on an as-deposited sample and HT sample 900 °C 1h.

As a result of the heat treatment, it was possible to increase the value of the relative elongation, without losing the strength of the material. During the HT, the material became more ductile, which is determined by the fracture surface.

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References

[1] Korsmik, R., Tsybulskiy, I., Rodionov, A., Klimova-Korsmik, O., Gogolukhina, M., Ivanov, S., Zadykyan, G., Mendagaliev, R. The approaches to design and manufacturing of large-sized marine machinery parts by direct laser deposition (2020) Procedia CIRP, 94, pp. 298-303. DOI: 10.1016/j.procir.2020.09.056

[2] Drokonov, D.A., Zadykyan, G.G., Korsmik, R.S. Investigation of the influence of nickel-based alloy powder EP648, obtained by plasma rotating electrode process, on powder utilization rate, structure and chemical composition, applied to direct laser deposition (2018) Journal of Physics: Conference Series. DOI: 10.1088/1742-6596/1109/1/012058
[3] Babkin, K., Zemlyakov, E., Ivanov, S., Vildanov, A., Topalov, I., Turichin, G. Distortion prediction and compensation in direct laser deposition of large axisymmetric Ti-6Al-4V part (2020) Procedia CIRP, 94, pp. 357-361. DOI: 10.1016/j.procir.2020.09.145

[4] Golovin, P.A., Vildanov, A.M., Babkin, K.D. Optimization of high-speed direct laser deposition regime parameters in titanium blades production (2019) Lecture Notes in Mechanical Engineering, 0 (9783319956299), pp. 2475-2483. DOI: 10.1007/978-3-319-95630-5_268

[5] Kovchik, A.Y., Babkin, K.D., Rakhmatulina, Z.A. Distortion compensation in LMD process (2019) Materials Today: Proceedings, 30, pp. 568-571. DOI: 10.1016/j.matpr.2020.01.136

[6] Ivanov, S., Zemlyakov, E., Babkin, K., Turichin, G., Karpov, I., Em, V., Rylov, S. Stress distribution in laser metal deposited multi-layer thick-walled parts of Ti-6Al-4V (2019) Procedia Manufacturing, 36, pp. 240-248 DOI: 10.1016/j.promfg.2019.08.031

[7] Golovin, P.A., Vildanov, A.M., Babkin, K.D., Ivanov, S.Y., Topalov, I.K. Distortion prevention of axisymmetric parts during laser metal deposition (2018) Journal of Physics: Conference Series, 1109 (1), article No 012065. DOI: 10.1088/1742-6596/1109/1/012065

[8] Zadykyan, G.G., Korsmik, R.S., Guschchina, M.O., Zhukov, A.S., Promakhov, V.V. Research and development of technology for obtaining small-scale GTE parts from ceramic composite materials by 3D printing (2018) Journal of Physics: Conference Series, 1109 (1). DOI: 10.1088/1742-6596/1109/1/012057

[9] Magerramova, L., Klimova-Korsmik, O., Turichin, G., Volkov, M., Guschina, M. Studies of hybrid titanium compressor blisks for gas turbine engines manufactured using additive technologies (2019) Proceedings of the ASME Turbo Expo, 7A-2019. DOI: 10.1115/GT2019-90073

[10] Chao Cai, Xiangyun Gao, Qing Teng, Raj Kiran, Jie Liu, Qingsong Wei, Yusheng Shi, Hot isostatic pressing of a near α-Ti alloy: Temperature optimization, microstructural evolution and mechanical performance evaluation, Materials Science and Engineering: A, Vol. 802, 2021. https://doi.org/10.1016/j.msea.2020.140426

[11] Guschchina, M.O., Shalnova, S.A., Gerasimov, N.I., Lebedeva, N.V., Klimov, G.G. Comparison of titanium powders and products manufactured by the direct laser deposition method (2019) Key Engineering Materials, 822, pp. 473-480. DOI: 10.4028/www.scientific.net/KEM.822.473

[12] Guschchina, M., Turichin, G., Klimova-Korsmik, O., Babkin, K., Magerramova, L. Features of heat treatment the ti-6al-4v gtd blades manufactured by dld additive technology (2021) Materials, 14 (15) DOI: 10.3390/ma14154159

[13] Chao Cai, Xu Wu, Wan Liu, Wei Zhu, Hui Chen, Jasper Chua Dong Qiu, Chen-Nan Sun, Jie Liu, Qingsong Wei, Yusheng Shi, Selective laser melting of near-α titanium alloy Ti-6Al-2Zr-1Mo-1V: Parameter optimization, heat treatment and mechanical performance, Journal of Materials Science & Technology, Volume 57, 2020, Pages 51-64. https://doi.org/10.1016/j.jmst.2020.05.004.

[14] Junjie Jiang, Zhihao Ren, Zhibo Ma, Tao Zhang, Peng Zhang, David Z. Zhang, Zhongfa Mao, Mechanical properties and microstructural evolution of TA15 Ti alloy processed by selective laser melting before and after annealing, Materials Science and Engineering: A, Volume 772, 2020. https://doi.org/10.1016/j.msea.2019.138742.

[15] Fang Wang, Li-Ming Lei, Xin Fu, Zhu-Man Song, Lei Shi, Bin Zhang, Guang-Ping Zhang, Effect of heat treatment on microstructures and tensile properties of TA19 alloy fabricated by laser metal deposition, Materials Science and Engineering: A, Vol. 782, 2020. https://doi.org/10.1016/j.msea.2020.139284.