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Microstructure and strength of a 3D-printed Ti–6Al–4V alloy subjected to high-pressure torsion

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Abstract: Currently, one of the effective 3D printing methods is wire-feed electron-beam additive manufacturing (EBAM), which allows producing large-sized commercial billets from Ti–6Al–4V titanium alloy. However, Ti–6Al–4V alloy produced by this method demonstrates reduced strength properties. It is known that it is possible to increase the strength properties of metallic materials by refining their grain structure by high-pressure torsion (HPT). This work is aimed at studying the influence of high-pressure torsion on the microstructure, and mechanical strength of a structural Ti–6Al–4V titanium alloy produced by the wire-feed electron-beam additive manufacturing method. The microstructure of a 3D-printed Ti–6Al–4V alloy in the initial state, and after high-pressure torsion, was studied using optical, scanning and transmission electron microscopy. An EBSD analysis of the material in its original state was carried out. The microhardness of the material in the initial and deformed states was measured. Using the dependence of the yield strength on microhardness, the estimated mechanical strength of the material after processing by the high-pressure torsion method was determined. The microstructural features of the 3D-printed Ti–6Al–4V alloy after high-pressure torsion, which provide increased strength of this material, are discussed. The research results demonstrate that 3D printing, using the electron-beam additive manufacturing method, allows producing a Ti–6Al–4V titanium alloy with a microstructure unusual for this material, which consists of columnar primary β-grains with a transverse size of 1–2 mm, inside of which martensitic α'-Ti needles are located. Thin β-Ti layers with a thickness of about 200 nm are observed between the α'-Ti needles. Further deformation treatment of the alloy, using the high-pressure torsion method, allowed forming an ultrafine-grained structure in its volume, presumably consisting of α-grains with an average size of (25±10) nm. High-pressure torsion of the 3D-printed alloy allowed achieving rather high microhardness values of (448±5) $HV_{0.1}$, which, according to the $HV=2.8–3\sigma_y$ ratio, corresponds to the estimated yield strength of approximately 1460 MPa.

Keywords: 3D-printed Ti–6Al–4V titanium alloy; Ti–6Al–4V titanium alloy; wire-feed electron-beam additive manufacturing; high-pressure torsion; microstructure; mechanical properties.

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INTRODUCTION

High-pressure torsion (HPT) is one of the most well-known methods of severe plastic deformation, which allows refining effectively, the microstructure in metal materials and providing an ultrafine-grained (UFG) structure, with dimensions less than 100 nm, and accordingly, an increase in their strength properties [1–3]. There are a number of works on the use of high-pressure torsion for processing the Ti–6Al–4V titanium alloy (Russian name – VT6), which is popular in industry. In this work [4], the authors applied HPT treatment on Ti–6Al–4V titanium alloy produced by hot rolling. As a result, a significant grain structure refinement to 100–200 nm, and as a consequence, a strong increase in strength up to σ_u=1740 MPa were observed. In the work [5], the authors subjected Ti–6Al–4V titanium alloy with a plate structure to high-pressure torsion treatment. The research results showed, that 10 revolutions of high-pressure torsion at a pressure of 7.5 GPa allows forming a nanostructured state in the Ti–6Al–4V alloy with an average grain size of 52.7 nm and a microhardness of 432 HV. At the same time, a significant increase in tribological properties, such as friction and wear resistance, was observed. Works [6–8] show that the formation of a UFG structure in the Ti–6Al–4V alloy ensures the manifestation of superplasticity under relatively low temperatures (550–650 °C).

In recent years, it has been demonstrated that the Ti–6Al–4V alloy, and products made from it, can be successfully manufactured by 3D additive technologies [9–11]. Moreover, works [12–14] show that the method of wire-feed electron-beam additive manufacturing (EBAM) is one of the most promising and opens broad prospects for the production of large-sized complex-shaped parts from titanium alloys. The main advantages of this technology are high productivity (up to 2500 cm^3/h), and almost 100 % efficiency of raw material consumption. Moreover, wire is much cheaper than powder raw materials, and its products are available for sale in a much wider range.

3D-printed Ti–6Al–4V titanium alloy has a specific initial microstructure, which significantly differs from the same alloy obtained by traditional production methods (casting, stamping, etc.). In particular, the microstructure after EBAM treatment consists of large columnar grains of the initial β-phase containing a lamellar martensitic α'-phase, which is formed due to the rapid solidification of the melt pool, and multiple phase transformations caused by repeated thermal cycles [10; 12]. However, such a structure is characterised by lower strength properties, and is noticeably inferior to those in comparison with the hot-rolled state [15; 16]. In this regard, an urgent task is to study the transformation of the microstructure obtained by additive technologies to increase the strength properties of the alloy. Of great interest is the study of the ultrafine-grained structure formation when exposed to severe plastic deformation (SPD) methods. Recently, such work was carried out on a Ti–6Al–4V alloy produced by the directed energy deposition (DED) technology, where the effect of equal channel angular pressing (ECAP) on the microstructure and mechanical properties was studied [17]. It was found that the alloy subjected to equal channel angular pressing exhibited a noticeable increase in mechanical properties in terms of strength and ductility.

High-pressure torsion treatment leads to a more significant refinement of the structure, and the possible unique properties that a 3D-printed Ti–6Al–4V alloy can acquire, after this treatment, are of scientific interest. This work is fundamental in nature. In the future, its results can become the basis for research aimed at improving the mechanical properties of 3D-printed parts of different geometries, using the SPD friction stir method, which, as is known, can be applied to treat the surface of various materials, including titanium alloys [18].

The purpose of this research is to study the effect of high-pressure torsion processing, on the microstructure and mechanical strength, of 3D-printed Ti–6Al–4V titanium alloy using wire-feed electron-beam additive manufacturing.

METHODS

As a material for research, the Ti–6Al–4V titanium alloy was used produced at the Institute of Strength Physics and Materials Science of the Siberian Branch of the Russian Academy of Sciences, Tomsk, Russia, using a unique scientific installation for wire-feed electron-beam additive manufacturing [12; 13]. As the raw material, a wire with a diameter of 1.6 mm made of Ti–6Al–4V titanium alloy was used, which was melted by an electron gun with a plasma cathode in a vacuum of 1.3×10^{-3} Pa. The wire was fed at a speed of 2 m/min at an angle of 35° to the base plate surface. The base plate moved at a speed of 2.2 mm/s relative to the electron beam along a meander path with layers deposited in an inversed manner. A rod of Ø20 mm was turned from the resulting blank. Then samples, with a diameter of 20 mm and a height of 2 mm, were cut out of it using an ARTA-120 electrical erosive machine for deformation processing and further research.

Samples of titanium alloy produced by electron beam melting of a wire, were subjected to high-pressure torsion on a SKRUDZH-200 unique scientific device at the Research Institute of Ufa University of Science and Technology, Ufa, Russia, at a specific compressive pressure of 6 GPa according to the mode: number of revolutions – 10, striker rotation speed – 0.2 rpm. After deformation, samples with a diameter of 20 mm and a height of 0.9–1.0 mm were obtained. These processing modes are described in detail in [2; 4].

The microstructure of the original and deformed samples was studied, using an Olympus GX51 optical microscope, a TESCAN MIRA LM scanning electron microscope, and a JEM-2100 transmission electron microscope (JEOL, Japan) with an accelerating voltage of 200 kV. Samples for TEM after high-pressure torsion were produced from an area 5 mm from the centre of the sample.

Microhardness was assessed using the Vickers method with a diamond pyramid at a load of 100 g for 15 s on a DuraScan 50 (EMCO-Test, Austria) device. To obtain an average value for each structural state, measurements were carried out at least 40 times.

RESULTS

Fig. 1 shows the microstructure of the Ti–6Al–4V alloy produced by the wire-feed electron-beam additive manufacturing method in the initial state. In the image obtained in an optical microscope (Fig. 1 a), columnar primary β-grains with a transverse size of 1–2 mm are observed in the volume, of which α-morphology grains were formed during the surfacing process. Such grains consist of a combination of lamellar and acicular martensitic α'-phase (dark contrast in the image). Large β-phase plates, the dimensions of which reach approximately (10±2) μm (light contrast in the image), are also observed. Detailed studies of the microstructure of the samples in SEM and TEM allowed determining the thickness of the α'-phase plates, which is approximately (1.5±0.5) μm, as well as thin interlayers, with a thickness of about 200 nm (Fig. 1 b–d), which are represented by β-phase. In the wide α'-phase plates, individual dislocations are visible.

Fig. 2 presents the results of EBSD analysis of a Ti–6Al–4V alloy sample in the initial state. In EBSD images in this state, a lamellar microstructure, united in large clusters with predominantly low-angle misorientation of grain boundaries, is observed. The length of the high-angle boundaries was 17.6 cm, and the low-angle boundaries were 1.16 cm.

Due to deformation treatment using the high-pressure torsion method, it was possible to refine significantly the structure in the Ti–6Al–4V alloy (Fig. 3). Inhomogeneous contrast is observed, because of the high level of internal

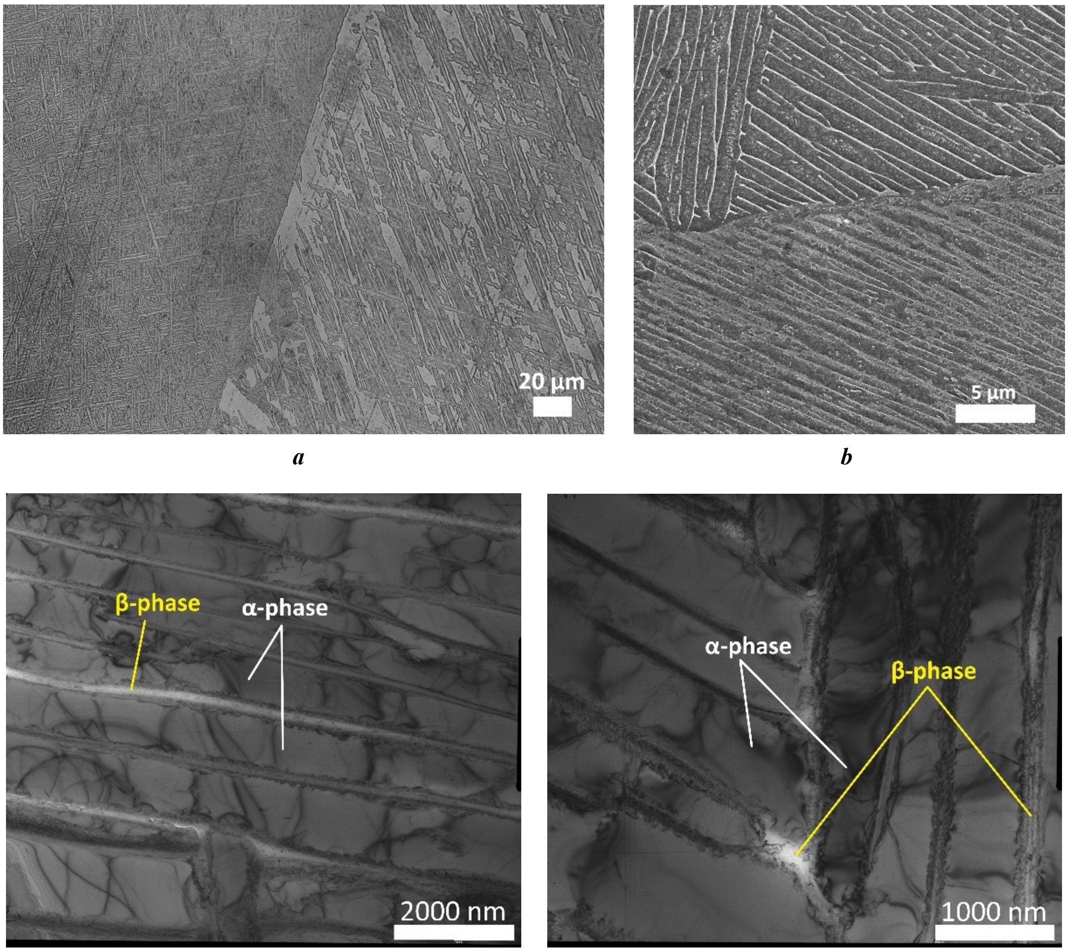


Fig. 1. *Microstructure of a 3D-printed Ti–6Al–4V alloy:*
a *– in an optical microscope (OM);* ***b*** *– in a scanning electron microscope (SEM);*
c, d *– in a transmission electron microscope (TEM)*
Рис. 1. *Микроструктура 3D-напечатанного сплава Ti–6Al–4V:*
a *– в оптическом микроскопе (ОМ);* ***b*** *– в растровом электронном микроскопе (РЭМ);*
c, d *– в просвечивающем электронном микроскопе (ПЭМ)*

stresses caused by the increased density of crystal lattice defects. According to dark-field images, the structure consists of equiaxial grains with an average size of (25±10) nm. Electron diffraction patterns show numerous reflections located around a circle, which indicates the presence of grains with predominantly high-angle boundaries. The blurring of diffraction reflections also indicates high internal stresses and elastic distortions of the crystal lattice.

Unfortunately, due to the small grain size and high internal stresses, it was not possible to obtain EBSD microstructure maps of Ti–6Al–4V alloy produced using EBAM and subjected to HPT.

In the initial state, the average microhardness of the EBAM-produced Ti–6Al–4V sample is (308±4) $HV_{0.1}$ (Fig. 4 and Table 1). Subsequent HPT processing of the 3D-printed Ti–6Al–4V alloy made it possible to increase significantly the microhardness of the material. In this case, a slight heterogeneity along the diameter of the sample is observed, which is typical for torsional deformation. The best development in the HPT process is observed in the region of the middle of the radius, where the average microhardness is approximately (448±5) $HV_{0.1}$.

DISCUSSION

In current work, the authors studied Ti–6Al–4V titanium alloy produced by wire-feed electron-beam additive manufacturing, and subjected to high-pressure torsion. The microstructure of the initial state consists of columnar primary β-grains, inside of which martensitic α'-Ti plates with transverse dimensions of about (1.5±0.5) µm are located. Between the martensitic plates, thin β-phase interlayers about 200 nm thick are also visible. A similar structure is often observed in Ti–6Al–4V titanium alloy, produced by additive technologies [12–14]. This structure is caused by the fact that in the process of wire-feed electron-beam additive manufacturing, the action of an electron beam leads to the formation of a melt pool in the near-surface volume of the substrate. Subsequently, as a result of solidification of the pools and upper layers of the grown metal, a columnar structure of β-grains is

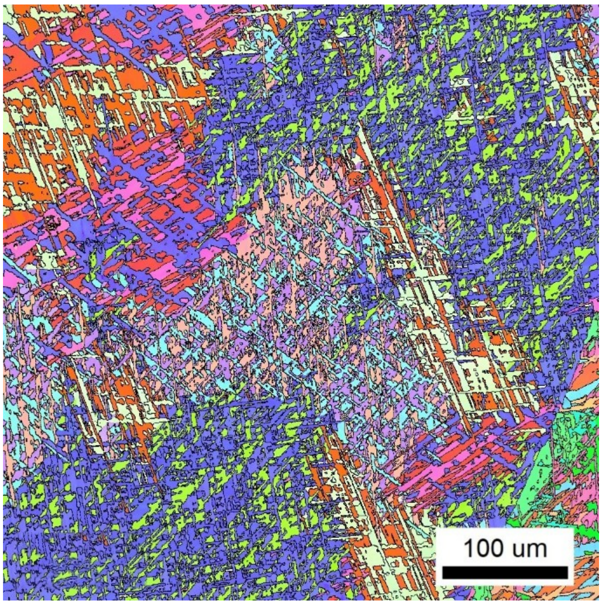
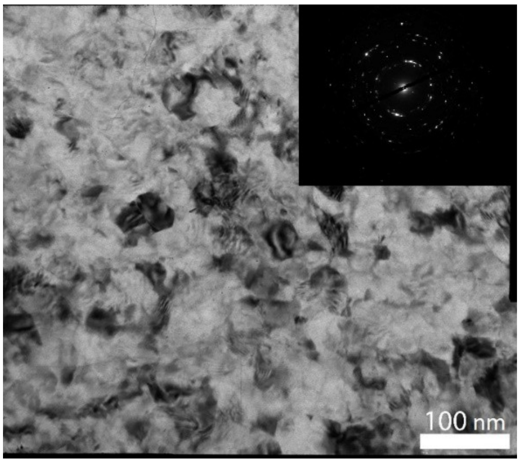
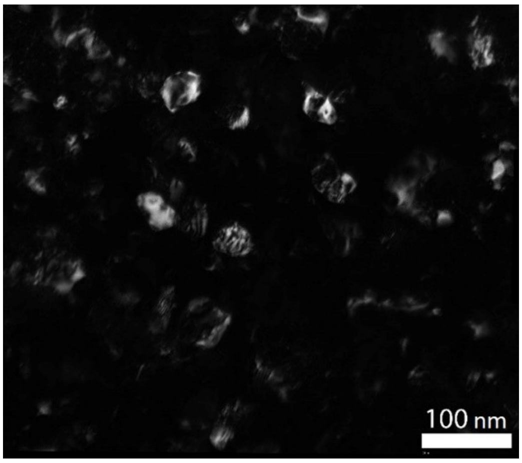


***Fig. 2.** EBSD image of a 3D-printed Ti–6Al–4V alloy sample*
***Рис. 2.** EBSD-картина 3D-напечатанного образца сплава Ti–6Al–4V*





a ***b***

***Fig. 3.** TEM images of the structure of a 3D-printed Ti–6Al–4V alloy after high-pressure torsion (HPT):*
***a** – bright-field image; **b** – dark-field image*
***Рис. 3.** ПЭМ-изображения структуры 3D-напечатанного сплава Ti–6Al–4V после КВД:*
***a** – светлопольное изображение; **b** – темнопольное изображение*

formed. During crystallisation of the deposited layer, epitaxial growth of columnar primary β-grains takes place, the sizes of which are determined by the cooling rate of the melt pool. After passing through the electron beam, the material solidifies into β-grains, and then undergoes rapid cooling, transforming into the martensitic α'-phase, which occupies almost the entire volume of the β-grain. The high cooling rate of the melt pool during 3D printing leads to a low content of the β stabilising element (vanadium) in the β-phase, and its presence in the α-phase [19].

Subsequently, the sample with the initial structure was subjected to high-pressure torsion in a mode of 10 revolutions, the rotation speed of the striker was 0.2 rpm, at a specific compressive pressure of 6 GPa, which allowed refining significantly the grain structure to (25±10) nm and increasing considerably the level of internal stresses. The resulting structure presumably consists entirely of the α-phase, since it is known that during high-pressure torsion of the Ti–6Al–4V alloy the β-phase dissolves [5–7]. The resulting microstructure differs from that observed after high-pressure torsion in the Ti–6Al–4V titanium alloy, with an (α+β) structure characteristic of the hot-rolled state. The difference in structures manifests itself primarily in the sizes of deformed grains. Thus, in the hot-rolled Ti–6Al–4V alloy after high-pressure torsion under various modes, the average grain size ranges from 40 to 100 nm [4–6],

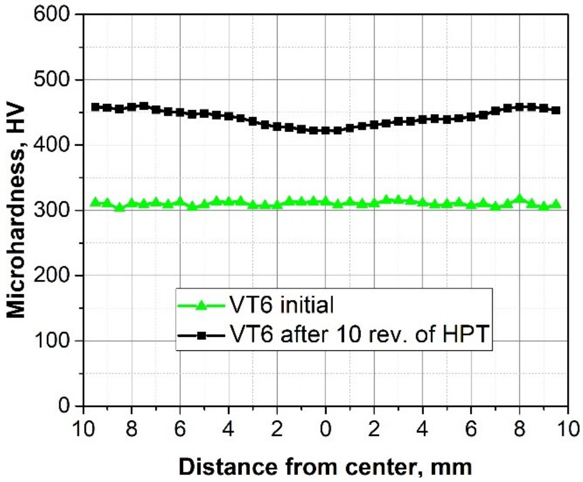


Fig. 4. *Microhardness distribution along the diameter of a 3D-printed Ti–6Al–4V alloy in the initial state and after high-pressure torsion (HPT)*
Рис. 4. *Распределение микротвердости по диаметру 3D-напечатанного сплава Ti–6Al–4V в исходном состоянии и после кручения под высоким давлением (КВД)*

Table 1. *Average values of grain size and microstructure of a 3D-printed sample of the Ti–6Al–4V titanium alloy*
Таблица 1. *Средние значения размера зерен и микротвердости 3D-напечатанного образца титанового сплава Ti–6Al–4V*

State	Average grain size	Microhardness, $HV_{0.1}$
Initial state	(1.5±0.5) μm	308±4
High-pressure torsion	(25±10) nm	448±5

while in the Ti–6Al–4V titanium alloy produced by electron-beam additive manufacturing, grain refinement to (25±10) nm is observed. This difference in the structures of the Ti–6Al–4V titanium alloy after high-pressure torsion is determined by different initial states. In 3D-printed Ti–6Al–4V, rapid cooling of the material during wire-feed electron-beam additive manufacturing leads to the formation of a predominantly martensitic α'-phase. Research [6; 7] shows that such an initial Ti–6Al–4V microstructure influences significantly the structure and properties of the alloy after high-pressure torsion. The martensitic structure contains a high level of residual stresses, dislocations and stacking faults, as well as twins due to shear transformation [6; 7]. Moreover, the initial microstructure has a high volume fraction of martensitic α'-phase boundaries, which likely act as nucleation sites for rapid grain fragmentation, and subgrain formation during the initial stages of HPT treatment.

The average value of microhardness of the 3D-printed Ti–6Al–4V alloy in the initial state is (308±4) $HV_{0.1}$, which is typical for the coarse-grained hot-rolled Ti–6Al–4V titanium alloy [5–7]. High-pressure torsion treatment allowed increasing significantly the values of the 3D-printed Ti–6Al–4V alloy microhardness to a level of the order of (448±5) $HV_{0.1}$. The obtained microhardness values are quite high for the Ti–6Al–4V alloy subjected to high-pressure torsion [5–7]. Using the known ratio of microhardness and yield strength ($HV=2.8–3\sigma_y$), it is possible to determine the expected mechanical strength of the Ti–6Al–4V alloy [20; 21]. Thus, one can assume that after high-pressure torsion, the yield strength of the Ti–6Al–4V titanium alloy 3D-printed, using the electron-beam additive manufacturing method, reaches 1460 MPa, which is a rather high value for this material.

As is known, the high strength of metal materials with a UFG structure, obtained by the methods of severe plastic deformation can be caused by a number of factors [3; 22; 23] – grain structure refinement, the presence of a high density of dislocations, impurity atoms, dispersed particles of second phases, twins, etc. Moreover, an important factor is the structure and state of grain boundaries, which usually have a nonequilibrium structure and contain a significant amount of grain boundary segregations and insertions [22]. It is obvious that the nature of the high strength of the 3D-printed Ti–6Al–4V titanium alloy, after high-pressure torsion obtained in this work, is determined by a number of structural features, including a highly refined grain structure and a high density of crystal lattice defects.

CONCLUSIONS

1. High-pressure torsion processing of a 3D-printed Ti–6Al–4V alloy allows refining significantly the grain structure to dimensions of (25±10) nm. Such strong refinement is not observed in the initial hot-rolled state of the alloy and is associated with the initial martensitic structure of Ti–6Al–4V.

2. High-pressure torsion of the 3D-printed Ti–6Al–4V alloy allowed increasing significantly the microhardness of the material to a level of approximately (448±5) $HV_{0.1}$, which, according to the HV=2.8–3σ_y ratio, corresponds to σ_y≈1460 MPa. Such a high strength is associated with the strong refinement of the structure and the significant density of crystal lattice defects.

3. The results of the study demonstrate, that the initial state of the Ti–6Al–4V alloy significantly affects the grain refinement, and phase transformations caused by high-pressure torsion, which consequently affects the strength characteristics achieved during this treatment.

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Микроструктура и прочность 3D-напечатанного сплава Ti–6Al–4V, подвергнутого кручению под высоким давлением

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Аннотация: В настоящее время одним из эффективных методов 3D-печати является проволочная электронно-лучевая аддитивная технология (ЭЛАТ), которая позволяет изготавливать крупногабаритные промышленные заготовки из титанового сплава Ti–6Al–4V. Однако Ti–6Al–4V, полученный данным методом, демонстрирует пониженные прочностные свойства. Известно, что повысить прочностные свойства металлических материалов можно посредством измельчения их зеренной структуры кручением под высоким давлением (КВД). Настоящая работа направлена на исследование влияния КВД на микроструктуру и механическую прочность конструкционного титанового сплава Ti–6Al–4V, полученного методом ЭЛАТ. Посредством оптической, растровой и просвечивающей электронной микроскопии изучена микроструктура 3D-напечатанного сплава Ti–6Al–4V в исходном состоянии и после КВД. Проведен EBSD-анализ материала в исходном состоянии. Измерена микротвердость материала в исходном и деформированном состояниях. С использованием зависимости предела текучести от микротвердости определена предположительная механическая прочность материала после обработки методом КВД. Обсуждаются микроструктурные особенности 3D-напечатанного сплава Ti–6Al–4V после КВД, за счет которых обеспечивается повышенная прочность данного материала. Результаты исследований демонстрируют, что 3D-печать методом ЭЛАТ позволяет получить титановый сплав Ti–6Al–4V с необычной для данного материала микроструктурой, которая состоит из столбчатых первичных β-зерен с поперечным размером 1–2 мм, внутри которых располагаются мартенситные иглы α'-Ti. Между иглами α'-Ti наблюдаются тонкие прослойки β-Ti толщиной около 200 нм. Дальнейшая деформационная обработка сплава методом КВД позволила сформировать в его объеме ультрамелкозернистую структуру, состоящую предположительно из α-зерен со средним размером (25±10) нм. КВД-обработка 3D-напечатанного сплава позволила достичь довольно высоких значений микротвердости (448±5) $HV_{0,1}$, что по соотношению HV=2,8–3$\sigma_т$ соответствует предположительному пределу текучести, равному примерно 1460 МПа.

Ключевые слова: 3D-напечатанный титановый сплав Ti–6Al–4V; титановый сплав Ti–6Al–4V; электронно-лучевая проволочная аддитивная технология; 3D-печать; кручение под высоким давлением; микроструктура; механические свойства.

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