DOI: https://doi.org/10.37434/tpwj2023.06.06

ELECTRON BEAM MELTING OF TITANIUM ALLOYS FOR MEDICAL PURPOSES

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ABSTRACT

The established regularities of the processes of alloying element evaporation and crystallization at electron beam melting were the base for determination of the melting modes and optimization of the technology of producing defect-free and chemically homogeneous ingots of a promising Ti–6Al–7Nb titanium alloy for medical purposes, having a homogeneous fine-grained structure without any traces of zonal segregation. Microstructural studies showed that Ti–6Al–7Nb alloy is a two-phase α+β titanium alloy of martensitic type, in which 1–2 mm thick precipitates of α-phase are observed on the boundaries of primary β-grains, and in the grain body formation of a platelike (widmanstaetten) morphology of α- and β-phase precipitates is found, the length of which inside the grains is equal to 10–40 μm. Such a structure ensures the best combination of the alloy mechanical characteristics, namely high values of strength (905 MPa) and ductility (13.5 %), which meet the requirements of international standards for titanium alloys for medical purposes.

KEYWORDS: titanium alloys, electron beam melting, medicine, evaporation, chemical composition, structure, mechanical properties

INTRODUCTION

Functional reliability of medical implants and structures, applied in orthopedics, traumatology, stomatology and other medical fields, primarily depends on a set of service properties, which are exhibited by the material under the real conditions of its service in a living body and the impact of this material on the surrounding tissues, biological liquids and the body as a whole [1–4]. At present, the main materials, used in implant manufacture, are metal alloys (titanium and cobalt), stainless steels, polymers and ceramics [5]. Despite intensive application of polymers and ceramic materials in implant products, metallic materials preserve their leading role (Figure 1). The best of the modern metallic biomaterials is titanium and alloys on its base.

Modern medicine uses a wide range of titanium alloys, varying by their chemical composition and mechanical parameters. Al, V, Mo, Mg, Cr, Si, Sn are the most often used of them as alloying elements.

However, vanadium poses a certain danger in titanium alloys. In the main Ti–6Al–4V medical alloy the content of toxic vanadium is just 4 % by weight, while in stainless steels the mass concentration of toxic nickel and chromium (in total) is more than 30 %.

Today traditional Ti–6Al–4V is replaced by new medical alloys, such as Ti–6Al–7Nb, Ti–13Nb–13Zr and Ti–12Mo–6Zr, not containing vanadium, which has, even though an insignificant, but toxic influence on live tissues.

Ti–6Al–7Nb alloy (international designation UNS R56700) is a special high-strength titanium alloy with excellent biocompatibility for surgical implants [6], developed for application for medical and surgical purposes.

Alloying is one of the efficient methods to improve the mechanical properties, and since niobium belongs to the same group as vanadium does, it usually acts as an α-β stabilizing element (similar to Ti–6Al–4V alloy). Although its properties are almost identical to those of Ti–6Al–4V, vanadium was replaced by niobium as a β-stabilizer, which increases the biocompatibility [7–9]. Ti–6Al–7Nb is one of the titanium alloys, which consists of hexagonal α-phase (aluminium-sta-

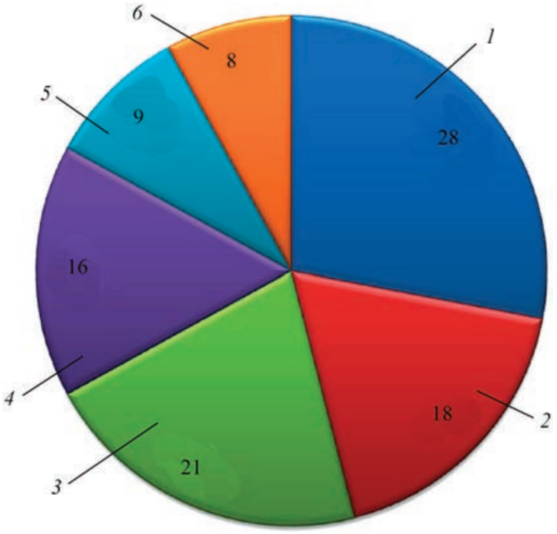

Figure 1. Main materials used for implant manufacture, %: *1* — titanium alloys; *2* — medical steels; *3* — cobalt alloys; *4* — polymers; *5* — ceramics, *6* — other materials

Table 1. Mechanical characteristics of titanium alloys

Alloy	$\sigma_{0.2}$, MPa	σ_y, MPa	σ^{-1}, MPa	E, MPa	δ, %	ψ, %
BT1-0	320	400	170	$11.1 \cdot 10^4$	25	–
Ti–6Al–4V	795	860	400	$11.5 \cdot 10^4$	10	25
Ti–6Al–7Nb	793	862	–	–	–»–	–»–
Bone	250	–	200	$2.5 \cdot 10^4$	0.5	–

bilized) and regular bulk-centered β-phase (niobium-stabilized). The alloy is characterized by additional advantages as to mechanical properties, has higher corrosion resistance and bioresistance, compared to Ti–6Al–4V alloy [10, 11].

Mechanical characteristics of Ti–6Al–7Nb alloy are given in Table 1, in comparison with those of other titanium alloys.

Ti–6Al–7Nb alloy is traditionally twice or three times remelted under vacuum to obtain a highly homogeneous composition of the ingot. The ingot is subjected to hot pressing or treatment of the round or flat rods by the traditional methods of titanium alloy treatment. Over the recent years, research work has been performed to produce medical products from Ti–6Al–7Nb titanium alloy, using different 3D printing technologies, such as SLM and EBM [12, 13].

The biocompatibility of Ti–6Al–7Nb alloy is high, and it can be applied in manufacture of implant-devices: artificial hip and knee joints, bone plates, screws for fracture fixation, heart valve prosthesis, pacemakers and artificial hearts [5, 10, 11].

As noted above, the determinant characteristic of materials for medical purposes is their biological compatibility with the human body, alongside the required physical-mechanical characteristics. This necessitates the use of technological processes, ensuring a higher degree of refining from impurities (sulphur, arsenic, phosphorus, lead, tin, copper, etc.) and gases in manufacture of such materials and products from them. Therefore, for a wide introduction of titanium alloys in medicine, it is necessary not only to develop new titanium-based materials with higher service properties, but further on to improve production of the existing titanium semi-finished products. Any imperfections of chemical and structural homogeneity in titanium alloys lead to lowering of the strength and fatigue life of the products. Producing titanium alloys involves difficulties, caused by the high sensitivity of titanium to interstitial impurities, particularly, to oxygen, nitrogen, hydrogen, and carbon, and by interaction with many chemical elements, resulting in formation of solid solutions or chemical compounds. For instance, oxygen, nitrogen and hydrogen can react with titanium to form different chemical compounds, as a result of the following reactions:

$$\mathrm{Ti} + \mathrm{O_2} \rightarrow \mathrm{TiO_2}; \tag{1}$$

$$2\mathrm{Ti} + \mathrm{N_2} \rightarrow 2\mathrm{TiN}; \tag{2}$$

$$\mathrm{Ti} + \mathrm{H_2} \rightarrow \mathrm{TiH_2}; \tag{3}$$

$$\mathrm{Ti} + 2\mathrm{H_2O} \rightarrow \mathrm{TiO_2} + 2\mathrm{H_2}. \tag{4}$$

High activity of titanium leads to running of physical-chemical processes of interaction with gases even in the solid state. Therefore, the intermetallic inclusions (hydrides, nitrides, oxides) may form both during the ingot production, and at different stages of technological processing to manufacture finished products.

More over, one of the main structural imperfections of the titanium alloys is the presence of nonmetallic inclusions [14]. Nonmetallic inclusions can penetrate into the finished product from the charge materials during melting, and can be formed at heat treatment of the finished product. Titanium actively interacts not only with gases, but also with other elements, including alloying components of the alloys, so that local enrichment of individual volumes of the ingots by alloying elements can lead to formation of intermetallic inclusions, for instance, $\mathrm{Ti_3Al}$, TiAl, etc.

At present not all the methods of manufacturing titanium ingots allow producing sound metal, and at violation of the technological process of titanium ingot production defects are found in the ingots, lowering the metal quality. Thus, solving the problem of producing sound ingots of high-strength titanium alloys from different charge materials is rather urgent.

Nowadays the most efficient refining of the metals and alloys is realized when conducting the processes of their vacuum melting. Electron beam melting (EBM) is the most efficient vacuum metallurgy process to produce the alloys, including the refractory and highly reactive ones with super low content of gases, low-melting impurities and nonmetallic inclusions. EBM enables regulation of the ingot melting rate in a wide range, due to an independent heating source that, in its turn, allows controlling the duration of the metal staying in the liquid overheated state. EBM is a technology, allowing practically completely ensuring removal of high-melting inclusions of high and low density [14]. Thus, EBM allows a significant improvement of the quality of titanium alloy ingots [14, 15].

A peculiarity of the work on producing materials and products for medical purposes is the fact that application of electron beam technologies allows, alongside metal refining, also obtaining the required physical-mechanical properties.

The majority of the titanium alloys, however, have a high content of alloying elements that complicates their production by EBM. Therefore, when producing titanium ingots for medical purposes there is the problem of ensuring the specified chemical composition of the ingot, as melting in relatively deep vacuum promotes selective evaporation of alloying elements with a high vapour pressure [16, 17]. This problem primarily concerns aluminium, as it has one of the highest values of vapour pressure, and this element is present in practically all the titanium alloys [14, 18]. Concentration of elements in an ingot with vapour pressure below that of titanium vapour, niobium in this case, can be even somewhat increased.

CALCULATION OF TECHNOLOGICAL MODES OF MELTING

In order to analyze the influence of EBM technological parameters and chemical composition of the initial charge on chemical composition of the produced ingots, the mathematical model of the processes of alloy component evaporation during EBM [17] was used to determine the dependencies of the alloying element content in 110 mm ingot of Ti–6Al–7Nb titanium alloy on the melting rate at different chemical composition of the initial charge [Al]$_0$, [Nb]$_0$.

Analysis of the obtained calculation results shows that losses of alloying elements for evaporation depend on their physical-chemical properties. While aluminium content during melting decreases (Figure 2), niobium content increases, on the contrary (Figure 3), compared to the content of these elements in the initial charge.

Such behaviour of alloying elements during melting is explained by the fact that aluminium vapour pressure at the specified temperature is higher than that of titanium vapour, while niobium vapour pressure is lower than that of titanium vapour. Consequently, in keeping with Longmuir law [19, 20], titanium evaporation rate is lower than that of aluminium evaporation and higher than that of niobium evaporation. Therefore, aluminium concentration in EBM ingot decreases, accordingly, compared to its concentration in the initial charge, and niobium concentration becomes higher.

Studying the behaviour of such alloying element as aluminium is critical for optimization of the process of EBM of Ti–6Al–7Nb titanium alloy. This is due to aluminium losses in EBM and considerable influence of aluminium on titanium alloy structure and mechanical properties.

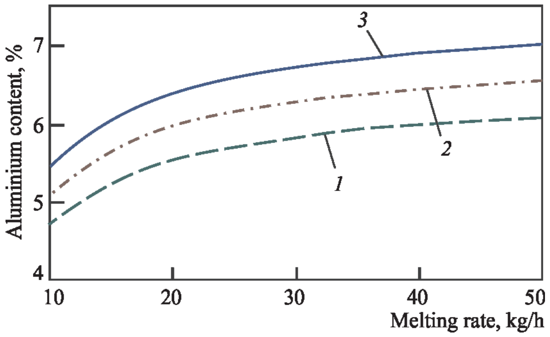

Figure 2. Dependencies of aluminium content in the ingot of Ti–6Al–7Nb alloy on melting rate at different content of aluminium [Al$_0$] in the initial charge, %: *1* — 6.5; *2* — 7.0; *3* — 7.5

Dependence of aluminium content in EBM ingot on melting rate is nonlinear. At a fixed composition of the initial charge at low melting rates (up to 30 kg/h) aluminium losses for evaporation are quite significant, and they strongly depend on the melting rate. So, for instance, at 7 % content of aluminium in the initial charge, lowering of melting rate from 30 to 10 kg/h leads to increase of aluminium losses for evaporation from 0.72 to 1.92 %, i.e. 2.7 times. At melting rates above 30 kg/h aluminium losses for evaporation are much smaller, and their dependence on the melting rate is rather weak. At increase of the melting rate from 30 to 50 kg/h aluminium losses for evaporation decrease from 0.72 to 0.45 %, i.e. 1.6 times.

It should be noted that aluminium losses for evaporation strongly depend on the melting rate, i.e. the time of titanium staying in the liquid state, when aluminium intensively evaporates from the melt (Figure 4).

Despite the relatively weak dependence of aluminium losses for evaporation on aluminium concentration in the initial charge (Figure 4), a detailed analysis of the dependence of these losses on aluminium content in the initial charge showed (Figure 5) that the higher the aluminium content in the initial charge, the greater are the aluminium losses for evaporation.

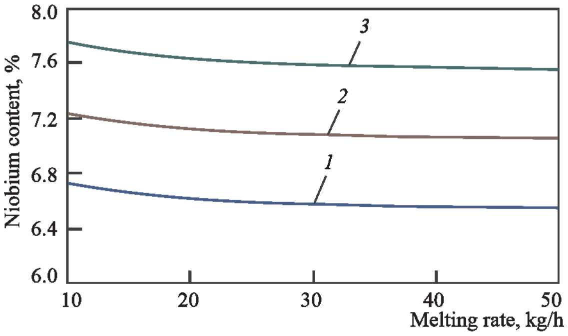

Figure 3. Dependencies of niobium content in an ingot of Ti–6Al–7Nb alloy on melting rate at different content of niobium [Nb]$_0$ in the initial charge, %: *1* — 6.5; *2* — 7.0; *3* — 7.5

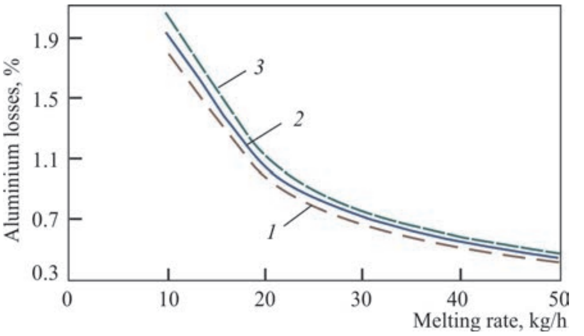

Figure 4. Aluminium losses for evaporation, depending on melting rate at different concentration of aluminium $[Al]_0$ in the initial charge, %: *1* — 6.5; *2* — 7.0; *3* — 7.5

Analysis of aluminium losses for evaporation at each EBM stage (Figure 6) showed that, depending on the melting rate, at the end of the consumed billet, the relative losses of aluminium for evaporation are equal to approximately 25 % % of the total losses of aluminium during EBM, the relative losses of aluminium for evaporation in the cold hearth are equal to 51 % of the total aluminium losses, and in the crucible they are close to 24 %. Such a distribution of aluminium losses between the melting stages weakly depends on the melting rate, and is due, primarily, to the area of the melt free surface at each melting stage, from which aluminium evaporates. This fact should be taken into account when designing the technological fixtures for producing ingots by EBM.

Conducted analysis of the process of aluminium evaporation during EBM shows that the main factors influencing aluminium concentration in EBM ingot are its content in the initial charge and the melting rate.

Results of calculations conducted by the mathematical model revealed that niobium concentration in Ti–6Al–7Nb alloy practically does not change in EBM ingot at the melting rate higher than 40 kg/h (see Figure 3). It should be noted that at melting rates of up to 30 kg/h niobium content in the ingot rises by 0.09–0.24 %, compared to its concentration in the initial charge that is associated with relatively high losses of aluminium for evaporation at these melting rates (see Figure 4).

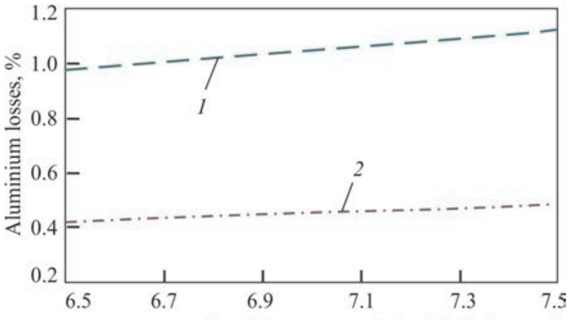

Figure 5. Aluminium losses for evaporation, depending on aluminium content in the initial charge at the following melting rate: *1* — 20; *2* — 50

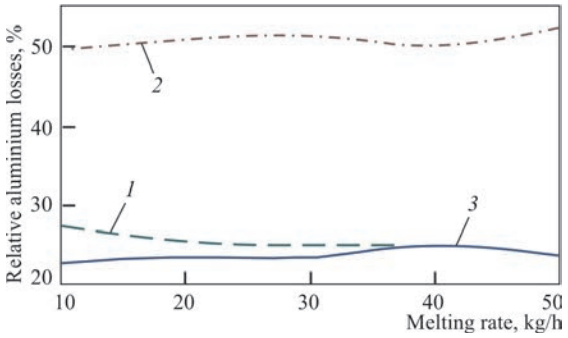

Figure 6. Relative losses of aluminium for evaporation at the consumable electrode end face (*1*), in the cold-hearth (*2*) and in the mould (*3*), depending on melting rate

Thus, results of the conducted investigations lead to the conclusion that to ensure the specified chemical composition of 110 mm ingot of Ti–6Al–7Nb titanium alloy for medical purposes the melting should be performed at the rate of 40–50 kg/h, and aluminium should be added to the initial charge in the quantity 0.5 % higher than the targeted aluminium concentration in the ingot.

In order to improve the technology of manufacturing ingots from titanium alloys for medical purposes, PWI performed work on producing ingots of an alloy of Ti–Al–Nb system. Ingots of 110 mm diameter were produced by the technology of cold-hearth EBM with portioned feed of liquid metal into a water-cooled mould.

EXPERIMENTAL PART OF THE INVESTIGATIONS

Investigations of the processes of alloy component evaporation from the melt in vacuum were the base for calculations of the predicted chemical composition of the produced ingots, the results of which were used to adjust the charge billet components. Alloying components with high vapour pressure (Al) were blended taking into account the compensation of evaporation losses.

The charge billet was a slab preproduced from titanium sponge TG-100 with alloying components in the form of pure niobium and primary aluminium (Figure 7).

UE-208M electron beam unit was used to conduct experimental melts to produce 110 mm ingots of Ti–6Al–7Nb titanium alloy.

During melting the following technological parameters were monitored: accelerating voltage of electron beam guns, beam current, speed of feeding the initial charge into the melting zone, speed of ingot pulling out of the mould, and cooling water temperature.

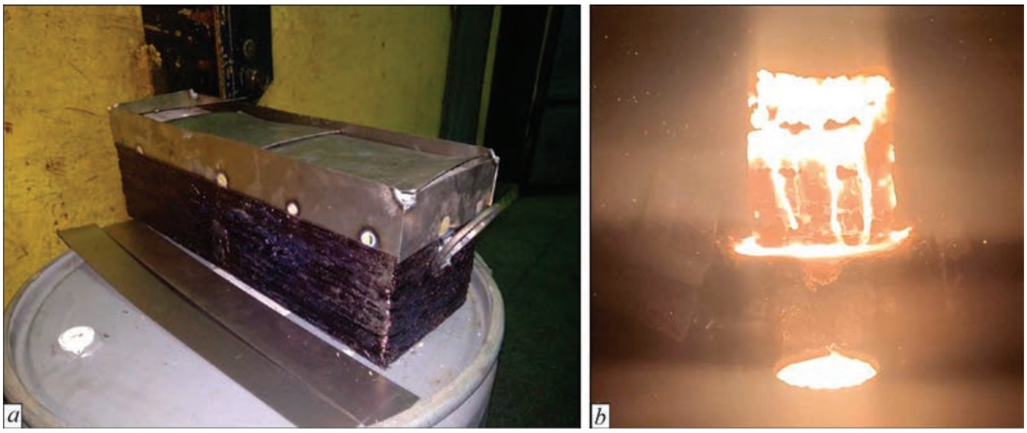

Figure 7. Charge billet (*a*) and electron beam melting (*b*) of 110 mm ingot of Ti–6Al–7Nb titanium alloy

Table 2. Distribution of alloying elements and impurities along EBM ingot of Ti–6Al–7Nb titanium alloy, wt.%

Ingot part	Al	Nb	Ta	Fe	C	H	O	N
Upper	6.2	6.8	0.01	0.09	0.01	0.005	0.11	0.012
Middle	6.3	6.7	–»–	0.10	–»–	–	–	–
Lower	6.2	–»–	–»–	0.08	–»–	–	–	–
UNS R56700	5.6–6.6	6.5–7.5	<0.5	<0.25	<0.03	<0.05	<0.20	<0.05

Numerical values of the technological parameters of melting used to produce 110 mm ingots of Ti–6Al–7Nb titanium alloy are as follows:

Melting rate, kg/h	40–50
Height of portions simultaneously poured into the mould, mm	10
Power in the mould, kW	16
Power in the cold hearth, kW	80

At the end of melting, shrinkage cavity removal was performed by gradual lowering of the power of heating the ingot upper end face in the mould.

The side surface of the produced ingots after cooling in vacuum to the temperature below 300 °C is clean, and there is no higher concentration of impurity elements in the form of oxidized or alpha layer (Figure 8). The depth of surface defects of corrugation type is 1–3 mm, defects in the form of tears, cracks or lacks of fusion are absent.

To assess the quality of metal of the produced ingots, chemical composition of the samples cut out along the sample from the upper, middle and lower parts was investigated. Results of analysis of chemical composition of the produced ingot metal showed that alloying element distribution along the ingot length is homogeneous, and corresponds to the grade composition (Table 2).

Considering the small dimensions of 110 mm ingot of Ti–6Al–7Nb titanium alloy, it was subjected to surface machining in a lathe. The surface layer was removed to the depth of not more than 3 mm to the side, and its end faces were machined (Figure 9).

Ingot macrostructure was studied on transverse templates, cut out from the ingot middle. The structure was revealed by etching the templates in 15 % solution of fluoric acid with addition of 3 % nitric acid at room temperature.

Ingot metal structure is dense, homogeneous, without any zones which etch differently over the ingot cross-section (Figure 10). No significant difference in the structure of the central and peripheral zones of the ingot was observed. No defects in the form of pores,

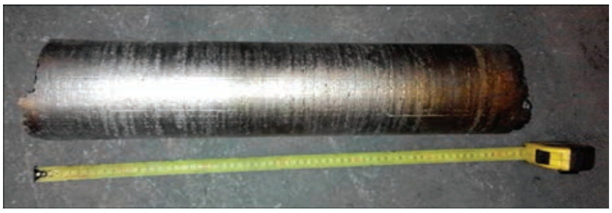

Figure 8. Ti–6Al–7Nb titanium alloy ingot of 110 mm diameter

Figure 9. Machined ingot of Ti–6Al–7Nb titanium alloy

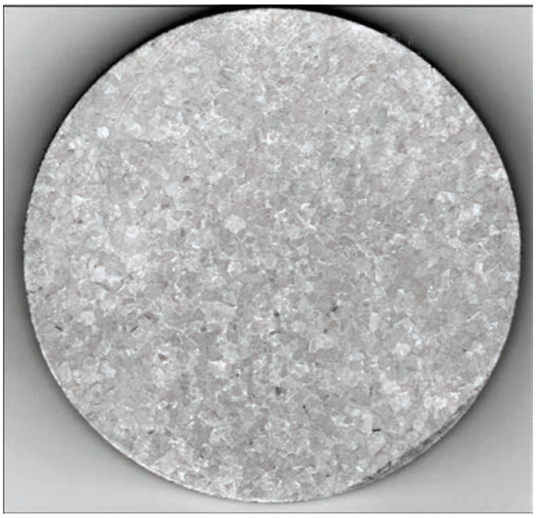

Figure 10. Macrostructure of cast metal of an ingot of Ti–6Al–7Nb titanium alloy

cavities, cracks, nonmetallic inclusions or alloying element segregation characteristic for VAR ingots was found.

The crystalline structure of the metal was the same along the entire ingot length, and it is characterized by crystals, the shape of which is close to the equiaxed one. Areas of columnar structure are absent.

The most important criterion of the quality of titanium alloy ingots is absence of nonmetallic inclusions in the metal, particularly in the form of nitrogen-containing alpha particles or titanium nitrides. At titanium remelting by cold-hearth EBM technology thermal and physical-chemical conditions are in place, which ensure removal of the above inclusions.

The method of ultrasonic flaw detection was used to investigate the presence or absence of internal defects in the form of nonmetallic inclusions, as well as pores or discontinuities in the titanium ingots. Investigations were conducted with application of ultrasonic flaw detector UD4-76 by echo-pulse method at contact variant of testing. Working frequency of analysis was equal to 1.25 MHz, which ensured the maximal signal-to-noise ratio. Multiple reflections of a small amplitude were observed when studying the ingots. This is typical for cast metal and is the result of signal reflection from the grain boundaries. Conducted analysis did not reveal any reflections, which could be interpreted as large nonmetallic inclusions, pores, or shrinkage cavities. Backwall pulse reflection was clearly visible on the scan (Figure 11). Noises are present in the area of introducing the probing signals, which are due to loose contact between the sensor and the ingot surface, which cannot be compensated by application of sealing lubricant.

Thus, conducted investigations showed that 110 mm EBM ingots of Ti–6Al–7Nb titanium alloy do not have any discontinuities, nonmetallic inclusions of more than 1 mm size, or dense clusters of finer inclusions.

The produced metal microstructure was studied in a light microscope. General view of the microstructure of Ti–6Al–7Nb titanium alloy produced by the developed EBM technology is given in Figure 12.

Metal of Ti–6Al–7Nb titanium alloy consists of equiaxed grains of 100–500 μm size (Figure 12). A positive moment is formation of a homogeneous structure, without any manifestations of zonal segregation traces (Figure 12, *a*). This is indicative of the optimal mode of producing the ingot in electron beam melting. The grain boundaries have an α-fringe, sometimes continuous, sometimes intermittent. The thickness of α-fringe is 0.8–3.0 μm (Figure 12, *d*). The intragranular structure of deep metal layers is lamellar, and it consists of α-colonies of different size. The thickness of α-plates is equal to 0.8–3.0 μm. In-

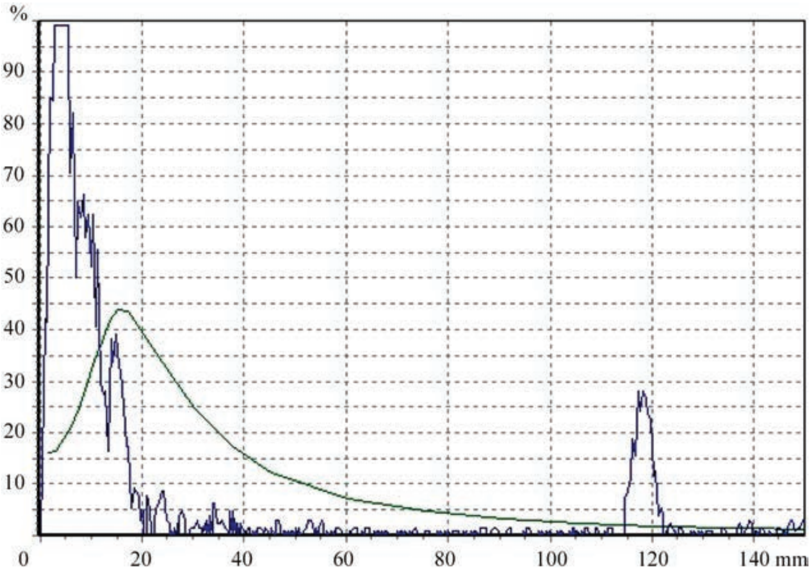

Figure 11. Scan of the central part of 110 mm ingot

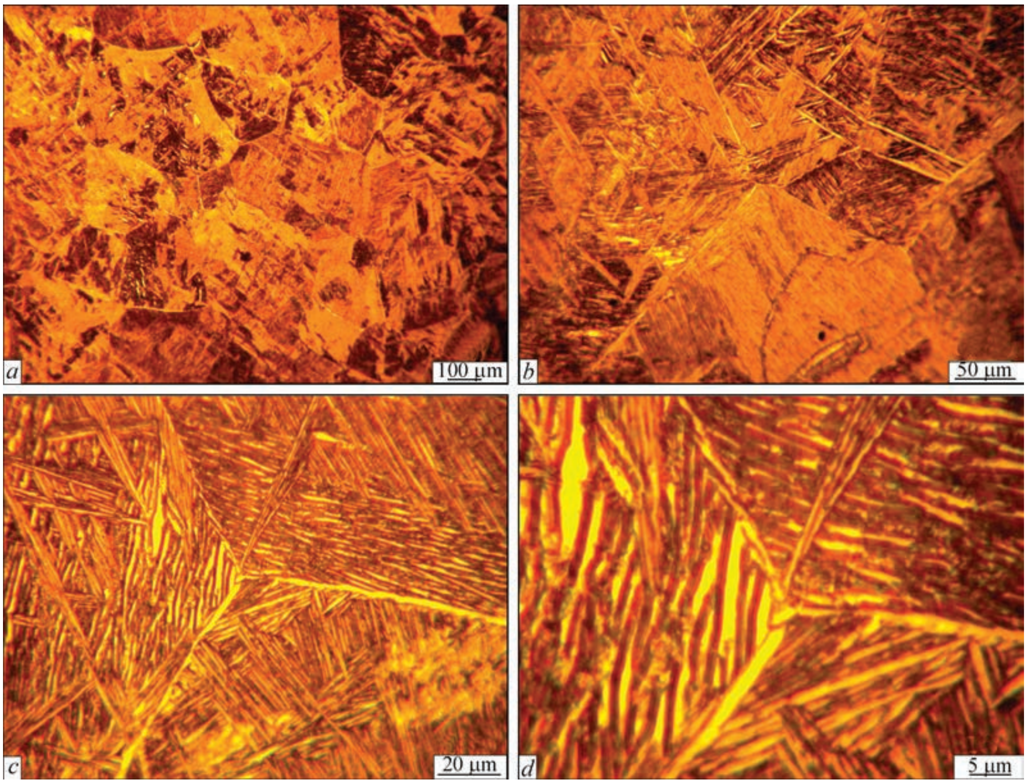

Figure 12. Microstructure of Ti–6Al–7Nb titanium alloy

Table 3. Mechanical characteristics of Ti–6Al–7Nb titanium alloy produced by EBM

Ti–6Al–7Nb	$\sigma_{0.2}$, MPa	σ_t, MPa	δ, %	ψ, %	*KCV*, J/cm^2
EBM	840.8	905.1	13.5	35.8	48.8
UNS R56700	793.0	862.0	>10	>25	–

terlayers of β-phase are located between the plates. The interlayer width is 0.3–1.0 mm.

Formation of lamellar (widmanstaetten) morphology of α- and β-phase precipitates is observed in the grain body. The length of the phase packets inside the grains is equal to 10–40 μm (Figure 12, *d*).

Thus, microstructural studies give grounds to consider that the alloy of the abovementioned composition is a two-phase titanium α+β-alloy of martensitic type.

Standard samples for studying the mechanical characteristics were cut out of 110 mm ingot of Ti–6Al–7Nb titanium alloy produced by EBM (Table 3).

Obtained results were indicative of high ductility values of Ti–6Al–7Nb titanium alloy at preservation of the strength values. Such data can be explained by formation of an equiaxed fine-grained structure in EBM ingots. As one can see from the comparison table, values of mechanical characteristics of the produced samples fully meet the requirements of international standard UNS R56700, which is indicative of the high quality of the metal produced by the developed technology.

Thus, results of the performed work were used to develop an EBM technology of producing a Ti–6Al–7Nb titanium alloy for medical purposes, the quality of which fully complies with the standard requirements as to its chemical composition, structure and mechanical properties.

CONCLUSIONS

1. A technology of producing 110 mm ingots of Ti–6Al–7Nb titanium alloy for medical purposes by electron beam melting method was developed with process productivity of 40–50 kg/h and 16 kW power of electron beam heating in the mould.

2. The methods of mathematical modeling were used to determine the dependencies of alloying element content in 110 mm ingot of Ti–6Al–7Nb titanium alloy on the melting rate at different chemical composition of the initial charge, and it was established that the dependence of aluminium content in EBM ingot on the melting rate is nonlinear: at melting rates up to 20 kg/h aluminium losses for evaporation are higher than 1 % and they increase considerably at lowering of the melting rate, while at higher melting rates aluminium losses are equal to 0.5–0.7 %.

3. It was determined that the relative losses of aluminium for evaporation in the cold hearth are equal to

approximately 50 % of the total losses of aluminium during EBM, while at the end face of the consumable billet and in the mould they are 25 % each, which is due, primarily to the area of the melt free surface at each melting stage.

4. It is shown that the selected modes of electron beam melting ensure formation in Ti–6Al–7Nb titanium alloy of a homogeneous fine-grained structure without any manifestations of traces of zonal segregation.

5. It is also shown that the developed EBM technology is an efficient method to produce ingots of Ti–6Al–7Nb titanium alloy for medical purposes with a high-quality surface, homogeneous chemical composition and high mechanical properties.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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SUGGESTED CITATION

V.O. Berezos, D.S. Akhonin (2023) Electron beam melting of titanium alloys for medical purposes. *The Paton Welding J*., **6**, 41–48.

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https://patonpublishinghouse.com/eng/journals/tpwj

Received: 11.04.2023
Accepted: 17.07.2023