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PRODUCTION OF TITANIUM INGOTS WITH REGULATED OXYGEN CONTENT BY ELECTRON BEAM MELTING

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ABSTRACT

Comprehensive research work was performed to produce ingots of Grade 2 titanium alloy of 600 mm diameter with regulated oxygen content of 0.12–0.16 % and an ingot of Grade 3 titanium alloy of 1100 mm diameter and up to 3 m length by the method of electron beam cold-hearth melting in the production facilities of SC «SPC «Titan» of the E.O. Paton Electric Welding Institute of the NAS of Ukraine» in multifunctional electron beam unit UE5810. A method of forming the charge billet and a formula for calculation of the amount of TiO_2 powder for alloying are proposed. Defectfree ingots of titanium alloys of Grade 2 and Grade 3 with regulated oxygen content were produced and the range of deviation of its distribution in the ingot metal of ± 0.02 % was provided. It is shown that the proposed modes of electron beam heating of the consumable billet, metal melting in the cold hearth and in the mould, as well as the melting rate ensure complete dissolution of titanium dioxide particles in the cold hearth, and absence of defects in the produced ingots, enriched in oxygen.

KEY WORDS: electron beam cold hearth melting; electron beam unit; titanium ingot; oxygen; regulated oxygen content; titanium dioxide; melting rate; macrostructure

Titanium, as one of the most important modern structural materials, is becoming more and more often used in medicine, construction industry and production of consumer goods. However, just about 5 % of titanium raw materials that are mined in the world today are processed into metallic titanium and its alloys, which are of greatest importance for many sectors of industry. Here, it should be noted that titanium-based alloys, the strength of which is 4–5 times higher than that of pure titanium, are now becoming ever wider applied [1, 2].

At present, alongside a stable global tendency to increase the use of titanium alloys in different industries, the issue of the high cost of titanium and its alloys remains unsolved [3]. The cost of titanium alloys is inseparably connected with the technology of producing them and ensuring the required mechanical properties. In order to increase the level of mechanical properties, titanium alloys have expensive alloying elements (aluminium, vanadium, zirconium, silicon, molybdenum) in their base [3]. However, it should be noted that sparsely-alloyed titanium alloys have been more and more widely accepted in recent years, in which the expensive alloying elements are replaced by inexpensive and available elements, namely iron, carbon, oxygen and nitrogen [4, 5]. At alloying by such elements it is taken into account that α-stabilizers are nitrogen, oxygen and carbon, which ensure the greatest increase of strength in titanium alloys, and β-stabilizer is iron.

In the small concentration range (up to 0.02 wt.%) each one hundredth fraction of oxygen increases the ultimate strength and yield limit of titanium by approximately 1.0–1.25 kgf/mm^2. Oxygen has the most noticeable effect on the mechanical properties of titanium at its concentration of up to 0.6 wt.% in the metal [6]. In this case, a considerable increase of strength is observed at relatively small drop of the ductility properties. At the same time, at oxygen concentration of more than 0.7 wt.%, titanium completely looses its capacity for plastic deformation. Thus, controlling oxygen content in the metal to a certain extent allows reaching an optimum ratio of ductility and strength characteristics of the titanium alloy. Therefore, oxygen can be regarded as a promising alloying element to produce new titanium alloys. It is particularly important for medical products, for which corrosion resistance and biocompatibility come to the fore, alongside the mechanical properties. Unlike other alloying components (for instance, vanadium) oxygen is safer [6–8].

Over the last decades, local and foreign metallurgists performed a number of studies on producing titanium, alloyed by oxygen [6, 7, 9], both from the gas phase during chamber electroslag remelting [7, 9], and with application of titanium dioxide powder as an alloying element at its addition to the charge billet [10, 11].

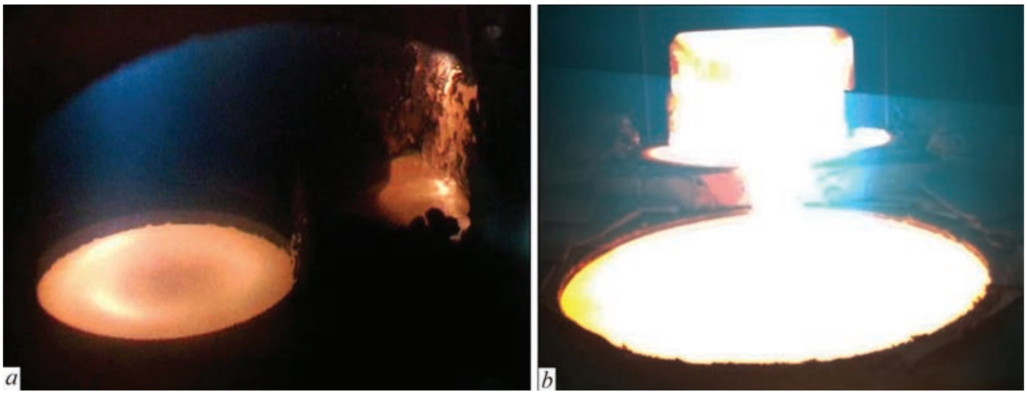

Figure 1. The process of producing titanium alloy ingots of Grade 2 (*a*) and Grade 3 (*b*)

It should be noted that today titanium and its alloys are produced by the following special electrometallurgy methods: vacuum-arc and plasma-arc remelting; vacuum-induction, electroslag and electron beam melting of titanium [12, 13]. The technology of vacuum-arc remelting of the consumable electrode became the most widely accepted. However, electron beam cold hearth melting (EBCHM) is the most promising from the viewpoint of metal refining, removal of nonmetallic inclusions of high and low density.

At titanium alloying by titanium dioxide powder it should be taken into account that its melting temperature is equal to 1870 °C that is higher than titanium melting temperature (1670 °C), so that titanium dioxide will not melt, but dissolve in the melt. In its turn, EBCHM is a technology, which, owing to an independent heat source, enables regulating the charge billet melting rate is a wide range, which allows regulation of the duration of the metal staying in the liquid overheated state. Thus, EBCHM technology can be regarded as the most efficient one for obtaining titanium ingots, alloyed by oxygen.

So, taking into account the experience of studies performed by the authors in work [11], it was proposed to conduct at PWI investigations on producing ingots of oxygen-doped titanium alloys. During investigations a batch of ingots of Grade 2 titanium alloy of 600 mm diameter with regulated oxygen content within 0.12–0.16 % an of and ingot of Grade 3 titanium alloy of 1100 mm diameter with regulated oxygen content within 0.28–0.32 % were melted in the production facilities of SC «SPC «Titan» of the E.O. Paton Electric Welding Institute of the NAS of Ukraine» in a multifunctional electron beam unit UE5810 (Figure 1).

Materials and investigation procedures. In order to obtain a uniform regulated oxygen content in ingots of titanium alloys of Grade 2 and Grade 3, a method to form the charge billet was proposed, which is based on that a water-dispersed emulsion of TiO_2 powder (Figure 2) is uniformly applied along its length as an alloying element, with further drying of the charge billet.

To obtain the specified level of oxygen in the metal, the required quantity of TiO_2 powder is calculated by the following formula:

$$M[TiO_2] = K(M_{in}[O]_{sp}\,\% - M_{bil}[O]_{bil}\,\%),$$

where $M[TiO_2]$ is the weight of TiO_2 powder for preparation of its water-dispersed emulsion; $K = 0.025$ is the coefficient of proportionality, which allows for oxygen percentage in TiO_2 powder; M_{in} is the ingot weight; $[O]_{sp}$ % is the specified oxygen percentage in the ingot; M_{bil} is the charge billet weight; $[O]_{bil}$ % is the oxygen percentage in the charge billet.

Considering that the melting temperature of TiO_2 powder is almost 200 °C higher than that of pure titanium, oxygen-enriched zones appear at its dissolution in the melt. In its turn, in keeping with titanium-oxygen constitutional diagram, titanium with a higher oxygen content has a higher melting temperature than that of pure titanium. The authors of work [14] established that at melt overheating above the titanium melting temperature by more than 150 °C, increase or decrease of the inclusion diameter two times, extends or shortens the dissolution time two times, accordingly, and at melt overheating by less than 150 °C increase or decrease of inclusion diameter two times extends or shortens the dissolution time three times, respectively. If the melt does not have enough time to

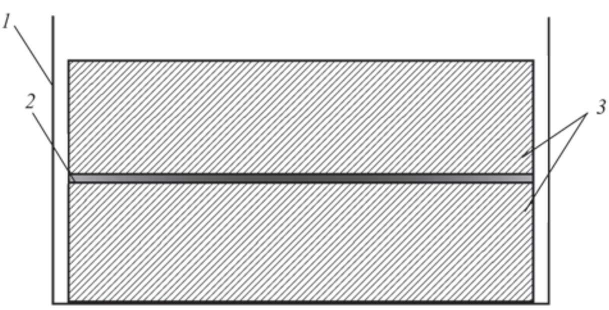

Figure 2. Scheme of the cross-section of the charge billet: *1* — nonconsumable box; *2* — layer of TiO_2 powder; *3* — charge billet

Figure 3. Ingot of Grade 3 titanium alloy of 600 mm diameter after EBCHM

homogenize before pouring into the mould, the oxygen-enriched metal can solidify ahead of the crystallization front, as its solidification temperature is higher, and it can form a zone of higher hardness in the ingot [14]. Here, micropores can form. Thus, it is necessary to reach a higher temperature of the melt, overheating the metal and soaking it in such a state the longer, the thicker is the TiO_2 layer in the charge billet.

Thus, only the homogenized melt, kept for the required time in the cold hearth, should be poured into the mould. Therefore, it is necessary to take into account the cold hearth geometry and the melting rate [14]. Based on the investigations performed by the authors in work [14], and taking into account the geometry of the cold hearth in electron beam unit UE5810, modes of electron beam heating of the consumable billet, metal melting in the cold hearth and in the mould, as well as the melting rates for ingots of 600 and 1100 mm diameter, were proposed. Thus, the total specific heating power was equal up to 0.14 kW/cm^2 for ingots of 600 mm diameter, and up to 0.11 kW/cm^2 for ingots of 1100 mm diameter. Here, the melting rate was 270 kg/h for 600 mm ingots, and 275 kg/h for 1100 mm ingots.

The process of producing the titanium ingots, alloyed by oxygen, was conducted as follows. Preparation of the initial charge billet was performed, along the length of which water-dispersed emulsion of TiO_2 powder was uniformly applied as an alloying element, with further drying of the charge billet. Then, electron beam cold hearth remelting of this initial charge billet was performed. The ingot was produced by periodically pouring the melt portions from the cold hearth into the mould, where its heating and periodical drawing were performed. The process was carried on up to deposition of the ingot of the required length. After that the ready ingot was cooled in a chamber to the required temperature under vacuum.

As a result of the conducted melting operations, titanium alloy ingots of Grade 2 of 600 mm diameter (Figure 3) and of Grade 3 of 1100 mm diameter and up to 3 m length were produced.

Examination of the quality of the produced ingots showed that their surface after cooling in vacuum is clean, oxidized or alpha layers are absent. The depth of surface defects in the form of corrugations which are characteristic for electron beam melting, is not more than 1–3 mm. Defects in the form of tears, cracks or lacks-of-fusion are absent.

Produced ingots of Grade 2 titanium alloy of 600 mm diameter were used to cut out transverse templates at 150 mm distance from the head and bottom part and from the ingot middle to study oxygen distribution along its length and cross-section. In Grade 3 titanium ingot of 1100 mm diameter the transverse templates were cut out from its head and bottom part.

Investigation results and their discussion. Investigations of the chemical composition of the ingots produced by the proposed method showed (see Table 1) that the impurity element content meets the requirements of the standards for titanium alloys of Grade 2 and Grade 3. Analysis of the results of studying the chemical composition of ingots with regulated oxygen content (see Table 1) showed that the proposed process of alloying by titanium dioxide powder and method to calculate its required quantity allows rather precisely reaching the required level of oxygen in the ingot metal under the condition of exact following of the melting modes.

Hydrogen concentration in the metal of the studied ingots did not exceed 0.002 %. No increased content of nitrogen was revealed, either in the bottom, or in the head part of the ingots, its maximum concentration being 0.02 %. Iron concentration in the studied ingot metal was in the range from 0.08 up to 0.13 %.

The quality of titanium ingot metal is due to absence of nonmetallic inclusions in it, particularly in the form of nitrogen-containing alpha particles or titanium nitrides, leading to formation of defects, which negatively affect the titanium alloy mechanical properties.

Therefore, after machining of the surface layer of the produced ingots (Figure 4), ultrasonic flaw detection method was used to detect inner defects in the form of nonmetallic inclusions, pores and discontinuities. Investigations of the ingot metal were conducted by sequential manual scanning of the side surface by the radius along the entire longitudinal axis of the ingot. To guarantee covering of the entire ingot volume, all of its side surface was scanned. Ultrasonic testing of the ingot metal was performed using ultrasonic

Table 1. Chemical composition of metal of titanium alloy ingots of Grade 3 of 1100 mm diameter and of Grade 2 of 600 mm diameter produced by EBCHM, wt.%

Alloy	Ingot part	Sampling location	C	Fe	O	N	H	Other elements (max), total
Grade 3	Top	Surface	0.01	0.08	0.29	0.01	0.002	0.13
		1/2 of radius	»	»	0.27	»	»	0.11
		Center	»	0.09	»	»	»	»
	Middle	Surface	»	0.10	0.29	»	»	0.12
	Bottom	Surface	0.02	»	0.30	0.02	»	»
		1/2 of radius	0.01	0.11	»	0.01	»	0.10
		Center	»	»	»	»	»	»
	ASTM B977-13		≤0.08	≤0.30	≤0.35	≤0.05	≤0.003	≤0.40
Grade 2	Top	Surface	0.02	0.11	0.13	0.01	0.002	0.13
		1/2 of radius	0.01	0.10	»	»	»	0.11
		Center	»	»	0.12	»	»	0.14
	Middle	Surface	»	»	0.15	0.02	»	0.11
		1/2 of radius	0.02	0.09	0.14	»	»	0.13
		Center	0.01	0.10	»	0.01	»	0.14
	Bottom	Surface	0.02	0.13	0.16	»	»	0.16
		1/2 of radius	0.01	0.11	»	»	»	0.14
		Center	»	»	0.15	»	»	»
	ASTM B977-13		≤0.08	≤0.30	≤0.25	≤0.03	≤0.003	≤0.40

converter P121-1.25-40-M-003 of 1.25 MHz frequency, which provides a smaller attenuation factor and better signal/noise ratio. During ingot examination, multiple small amplitude echoes typical for cast metal were observed which resulted from signal reflection from the grain boundaries. Performed testing did not reveal any pulses, which could be interpreted as non-metallic inclusions, pores or shrinkage cavities.

Macrostructural studies of the produced ingots were conducted on transverse templates, which were cut out to study the uniformity of oxygen distribution over the ingot cross-section. The structure was revealed by template etching in 15 % solution of fluoric acid with addition of 3 % nitric acid at room temperature. As a result, it was found that the ingot metal is dense, homogeneous, with absence of differently etched zones in the ingot cross-section. The studied metal is characterized by crystals close to the equiaxed ones of 25 to 50 mm size for ingots of 1100 mm

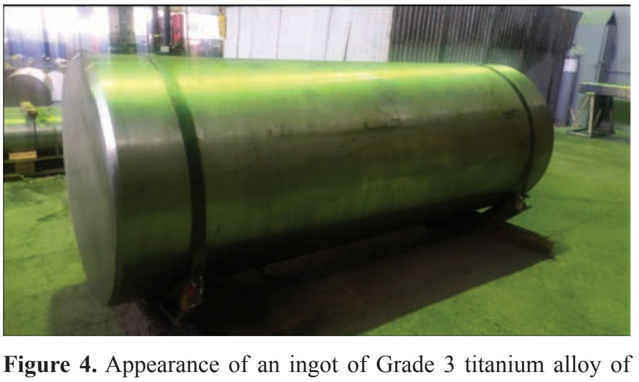

Figure 4. Appearance of an ingot of Grade 3 titanium alloy of 1100 mm diameter after machining

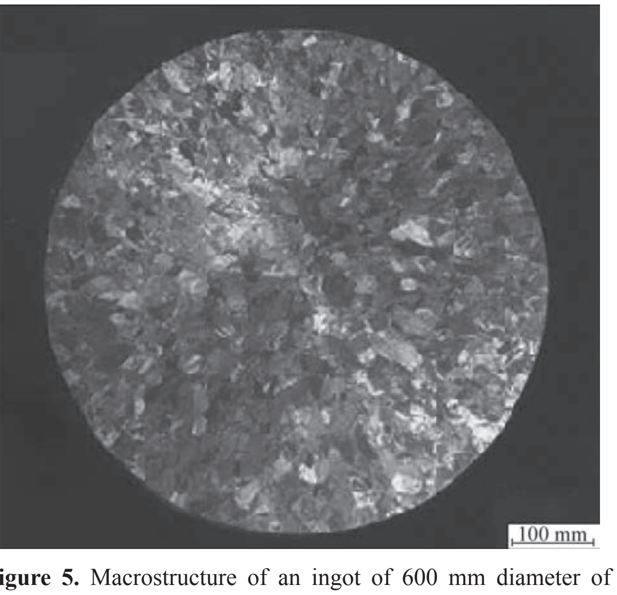

Figure 5. Macrostructure of an ingot of 600 mm diameter of Grade 2 alloy

diameter and of 10–30 mm size for ingots of 600 mm diameter. No difference is found in the structure of the ingot central and peripheral zones (Figure 5).

The microstructure of the grain, produced as a result of cast metal melting, is characterized by packets of α-phase plates oriented in one direction towards the grain boundary.

Thus, EBCHM technology allows producing defectfree titanium ingots with regulated oxygen content that meet the standard requirements, and the proposed formula for calculation of the quantity of TiO_2 powder for alloying allows achieving not more than ± 0.02 % range of oxygen distribution deviation in the ingot metal.

CONCLUSIONS

1. Proceeding from the research results, a method to form the charge billet and a formula for calculation of the quantity of TiO_2 powder for alloying were proposed that allowed producing defectfree titanium ingots with regulated oxygen content, while ensuring a not more than ± 0.02 range of its distribution deviation in the ingot metal.

2. It is shown that the proposed modes of electron beam heating of the consumable billet, metal melting in the cold hearth and the mould, as well as the melting rate ensure complete dissolution of titanium dioxide particles in the cold hearth and absence of oxygen-enriched defects in the produced ingot.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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