DOI: https://doi.org/10.37434/tpwj2024.03.06

ELECTROSLAG REMELTING OF TITANIUM UNDER VACUUM CONDITIONS

I.V. Protokovilov[1], **T. Beinerts**[2], **V.B. Porokhonko**[1], **D.A. Petrov**[1]

[1]E.O. Paton Electric Welding Institute of the NASU
11 Kazymyr Malevych Str., 03150, Kyiv, Ukraine
[2] Institute of Physics, University of Latvia
32 Miera Str., Salaspils LV-2169, Latvia

ABSTRACT

The results of experimental studies of the process of electroslag remelting of titanium in a chamber furnace at different values of pressure in the melting space, from vacuum to excess pressure, are given. Experiments were carried out during the melting of ingots with a diameter of 85 and 105 mm from titanium alloys VT1-0 and VT22 using fluoride-chloride flux AN-T4. The pressure of the inert gas in the furnace chamber was varied from 20 to 300 kPa. It is shown that in the entire studied range of pressures, the electroslag process proceeded stably with the formation of ingots with a high-quality side surface and a dense structure, without pores, slag inclusions and other internal defects. Experimental data on the effect of pressure in the melting space on the gas composition and structure of titanium ingots are given. The possibility of reducing the hydrogen content in titanium alloys by carrying out the electroslag process in vacuum conditions is shown. It was also established that the VT1-0 titanium ingot, melted under vacuum conditions, is characterized by a larger grain size compared to the ingot obtained under excess pressure.

KEYWORDS: electroslag remelting, chamber furnace, vacuum, titanium, gas composition, ingot, structure

INTRODUCTION

Due to the high chemical activity of titanium, in particular its interaction with atmospheric gases at temperatures above 400–450 °C, electroslag remelting (ESR) of titanium alloys should occur exclusively in chamber furnaces in a protective inert atmosphere [1–3]. Usually, in the ESR of titanium, the melting space is pre-vacuumed, and then filled with an inert gas (argon). The melting process is carried out under excess pressure of argon (120–140 kPa), which prevents air inflow in the event of leaks in the furnace units. At the same time, the melting process can be carried out both in a stagnant argon atmosphere and in a flowing one.

Such a scheme provides a reliable protection of the molten metal and consumable electrode heated to high temperatures from interaction with the air atmosphere and also prevents the evaporation of alloying components with high vapor elasticity. However, it also has certain disadvantages compared to melting under vacuum conditions.

It is known that hydrogen, which belongs to the category of harmful impurities because it causes hydrogen embrittlement of titanium alloys, has a reversible solubility in titanium. As the temperature increases and the pressure decreases, the solubility of hydrogen in titanium decreases significantly. Because of this, when remelting titanium in a vacuum, it is refined from hydrogen. This happens, for example, with vacuum-arc remelting (VAR) and electron beam melting of titanium alloys [4, 5]. When remelting titanium under excess pressure, hydrogen removal occurs to a much lesser extent or does not occur at all.

In addition, the remelting of titanium under conditions of excess pressure creates, unfavorable conditions for the removal of moisture, which can be adsorbed by the material of the consumable electrode, salt residues on the inner surface of the furnace chamber, etc. To a greater extent, this applies to the remelting of electrodes pressed from spongy titanium (or titanium shavings). This is due to the fact that in the process of storing the titanium sponge, during the production of consumable electrodes from it, their transportation and storage, physical adsorption of moisture by the developed surface of the sponge titanium (and residual chlorine salts which were not completely removed from the sponge titanium) is possible. It should be noted that atmospheric gases physically adsorbed on the surface of the sponge are not dissolved in titanium. Therefore, during further metallurgical processing, in conditions of high temperatures and vacuum, they are removed by a vacuum system. In the case of remelting of spongy titanium electrodes under conditions of excess pressure, residual moisture can cause an increased content of hydrogen and oxygen in titanium.

The authors of works [6, 7] also point to the possibility of grinding the microstructure of ingots melted by vacuum ESR. This effect is explained by a more uniform temperature distribution in the molten slag pool, alignment of the crystallization front, and an

increase in the local rate of solidification caused by the intensification of the hydrodynamic flows of the slag as a result of the evaporation of fluorides and the emission of CO.

In view of the above, it is of some interest to study the process of ESR of titanium under vacuum conditions. Vacuum ESR can combine the advantages of ESR and VAR processes, in particular, to ensure high quality of the ingot surface formation, dense and homogeneous metal structure, reliable gas protection and hydrogen removal.

The aim of this work was to study the technological features of the ESR process of titanium under vacuum conditions, to determine its effect on the gas composition and features of the structure formation of the ingot metal.

EXPERIMENTAL PROCEDURE

Experimental studies were carried out in the equipment for chamber ESR of highly reactive materials developed in the E.O. Paton Electric Welding Institute (Figure 1). The installation is equipped with a chamber that ensures complete sealing of the melting space, a vacuum system, an inert gas supply system and pressure monitoring devices. The installation allows the remelting process both under conditions of excess pressure up to 500 kPa, and under conditions of vacuum, when melting ingots with a diameter from 60 to 260 mm.

Experimental melting was performed by remelting pressed consumable electrodes with a diameter of 50 and 75 mm under fluoride-chloride AN-T4 flux, in copper water-cooled moulds with a diameter of 85 and 105 mm. Electrodes of two compositions were used: made of spongy titanium TG 110, for titanium VT1-0 (commercially pure titanium); made of spongy titanium TG 130 and K-5-1 master alloy (TU 48-4-306–88) for VT22 alloy (Ti–5Al–5V–5Mo–1Fe–1Cr, wt.%). Before melting, the melting space was evacuated to a pressure of 2.6 Pa ($2 \cdot 10^{-2}$ mm Hg), then filled with argon. Experiments were performed at different values of argon pressure, from a vacuum of 20 kPa to an excess pressure of 300 kPa. Acceptable pressure values were chosen taking into account the results of work [8].

According to the results of the experiments, the stability of the electroslag process was evaluated, the gas composition and features of the structure formation of the ingot metal were investigated. The gas composition was determined by the method of reductive melting of samples in a flow of inert carrier gas [9]. For this purpose, samples of a cylindrical shape with a diameter of 3 mm and a length of 3 mm (type MI-99) were produced.

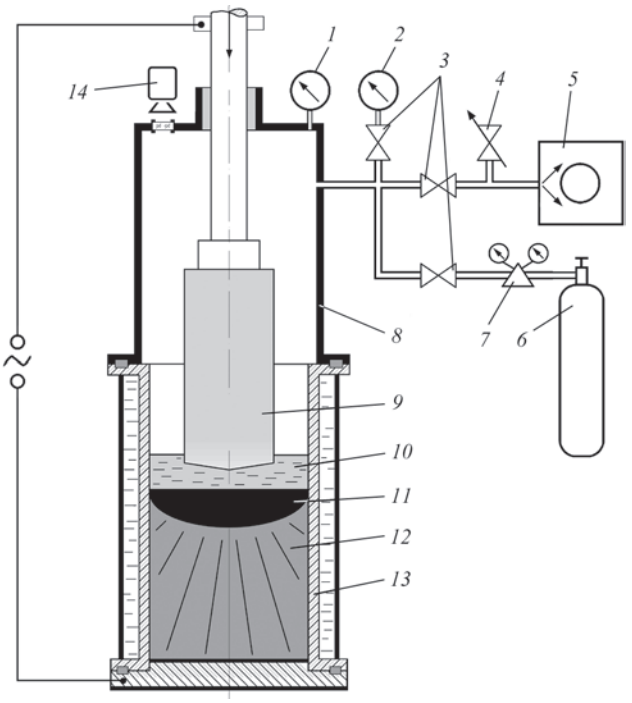

Figure 1. Schematic of the experimental installation for the ESR in a vacuum condition: *1* — vacuum/pressure gauge; *2* — ionization vacuum gauge; *3* — vacuum valves; *4* — inlet valve; *5* — vacuum pump; *6* — argon cylinder; *7* — pressure regulator with rotameter; *8* — furnace chamber; *9* — consumable electrode; *10* — slag pool; *11* — metal pool; *12* — ingot; *13* — water-cooled mould; *14* — camcorder

RESULTS AND DISCUSSION

The results of the experiments are presented in the Table 1 and in Figures 2–7. In the entire studied range of pressures, from vacuum to excess pressure, the electroslag process was stable. This confirms the results of research presented in [8], where acceptable ranges of low pressure were established when using AN-T4 flux.

The melted ingots had a good quality of side surface formation. For comparison, Figure 2 shows the appearance of VT1-0 ingots obtained at pressures of 25 and 160 kPa. When melting under vacuum conditions, the side surface turned out to be a little rougher (Figure 2, *a*) than under excess pressure (Figure 2, *b*). This can be

Table 1. The gas composition of titanium ingots obtained by ESR at different pressures in the melting space, wt.%

Alloy	Pressure, kPa	Content weight, %		
		[O]	[N]	[H]
VT1-0	25	0.074	0.0060	0.0034
	160	0.070	0.0056	0.0052
VT22	25	0.13	0.015	0.0052
	50	0.14	0.027	0.0053
	75	0.09	0.022	0.0065
	100	0.14	0.020	0.0076
	150	0.14	0.025	0.0068
	200	0.14	0.027	0.0064
	250	0.13	0.014	0.0070
	300	0.13	0.015	0.0064

Figure 2. VT1-0 titanium ingots melted at a pressure of 25 kPa (*a*) and 160 kPa (*b*)

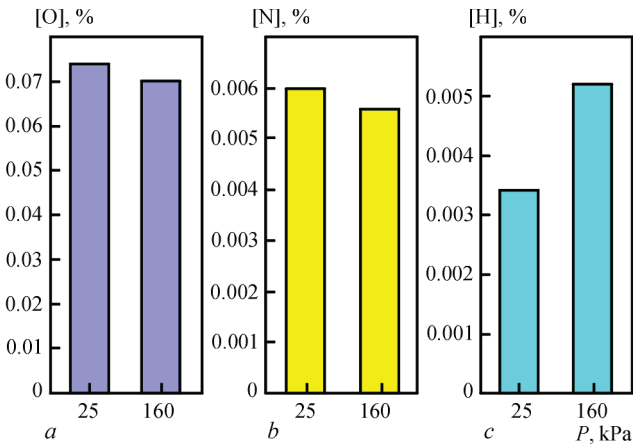

Figure 3. The content of oxygen (*a*), nitrogen (*b*) and hydrogen (*c*) in VT1-0 titanium ingots, depending on the inert gas pressure in the melting space of the ESR furnace

explained by an increase in the hydrodynamic activity of slag and metal pools during melting in a vacuum, which, in turn, leads to a periodic change in the thickness of the slag skull on the surface of the ingot. However, in both cases no surface defects were found.

The results of gas analysis of the ingots metal are given in Table 1 and in Figures 3, 4. The analysis of the obtained data indicates the absence of a clear regularity regarding the effect of pressure on the content of oxygen and nitrogen in the metal of ingots. In VT1-0 titanium ingots, a slight (by 5–7 %) increase in the content of oxygen and nitrogen was observed in the metal melted under vacuum conditions (25 kPa) compared to the metal obtained under excess pressure (160 kPa) (Figure 3 *a*, *b*). However, the difference in the content of elements is within the measurement error, which does not give grounds to assert a certain regularity.

The absence of regularity in the effect of pressure (in the studied range of 25–300 kPa) on the content of oxygen and nitrogen was also observed during the smelting of the VT22 alloy. In this case, the oxygen and nitrogen content in the experiments ranged from 0.09 to 0.14 and 0.014 to 0.027 %, respectively, without increasing or decreasing trends (Figure 4 *a*, *b*). Obviously, such results are related to various random factors that influenced the obtained data (measurement errors, impossibility to maintain absolutely identical conditions for different experimental melting, etc.). This suggests the need for further statistical supplementation of the obtained results. Nevertheless, it can be stated that in the studied range, the pressure level in the melting space has no effect on the content of oxygen and nitrogen in titanium ingots.

As for the hydrogen content, a certain regularity was established in this case, both for ingots of commercially pure VT1-0 titanium and for ingots of titanium VT22 alloy. It manifests itself in a decrease in the presence of hydrogen when conducting the ESR process under vacuum conditions (Figures 3, *c*, 4, *c*). When smelting VT1-0 ingots, a decrease in the pressure in the melting space from 160 to 25 kPa led to a decrease in the hydrogen content in the metal from 0.0052 to 0.0034 wt.%., i.e. by 35 %. Similar results were obtained for VT22 alloy ingots. When the pressure decreased from 100–300 kPa (overpressure range) to 25 kPa, there was a regular decrease in the hydrogen content in the ingot metal from 0.0064–0.0076 to 0.0052 wt.% (by 25 % on average).

Thus, the data obtained give grounds to assert that it is possible to reduce the hydrogen content in titanium alloys by 20–35 % by carrying out the ESR process under vacuum conditions (20–25 kPa).

Figure 5 shows the cross-sectional macrostructures of VT1-0 titanium ingots obtained by ESR at an inert gas pressure in the melting space of 25 and

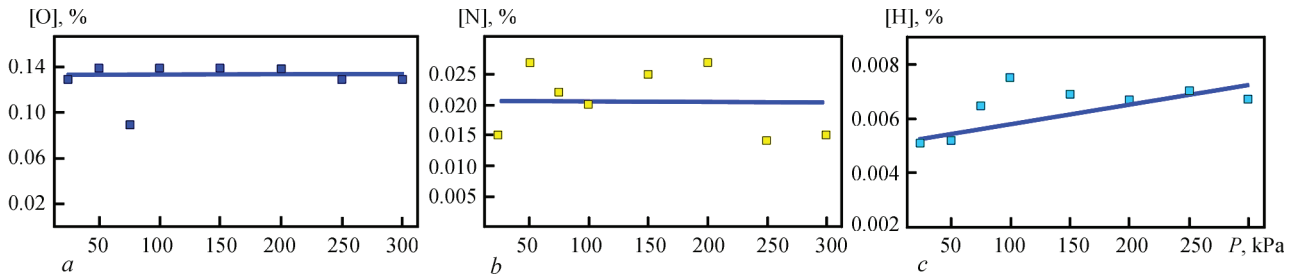

Figure 4. The content of oxygen (*a*), nitrogen (*b*) and hydrogen (*c*) in ingots of the VT22 alloy, depending on the inert gas pressure in the melting space of the ESR furnace

160 kPa. In both cases, the metal structure is dense, without pores, non-metallic inclusions and other internal defects.

Figures 6–7 present the results of metal macrostructure analysis using the MIPAR software. The average grain size (equivalent diameter) in the ingot melted at a pressure of 25 kPa was 2.07 mm, and at 160 kPa – 1.26 mm. That is, the ingot smelted under excess pressure had a finer-grained structure than the ingot smelted in a vacuum. On the one hand, this coincides with the data given in [10], where it is noted that the ingots of ESR under pressure have a more dispersed structure, compared to the ingots of traditional ESR. On the other hand, it contradicts the data of the work [6], where the grain grinding of ESR ingots melted under vacuum conditions was found.

The effect of excessive pressure on the nature of structure formation can be explained by known factors: an increase in the degree of supercooling and the crystallization rate, a decrease in the size and more uniform formation of crystal nuclei, a decrease in the energy of interphase interaction at the melt-crystal interface, etc. [11].

In our case, the influence of pressure on the formation of the structure of ingots can also be explained by its influence on the modes of ESR. Thus, it was shown in [8] that a decrease in the pressure in the melting space below the atmospheric pressure leads to a decrease in the melting current. In turn, this causes a decrease in heat generation in the slag pool (without a forced increase in the melting current due to the electrode feed rate), which affects the nature of metal structure formation, the local rate of hardening and the size of the grains of the cast metal.

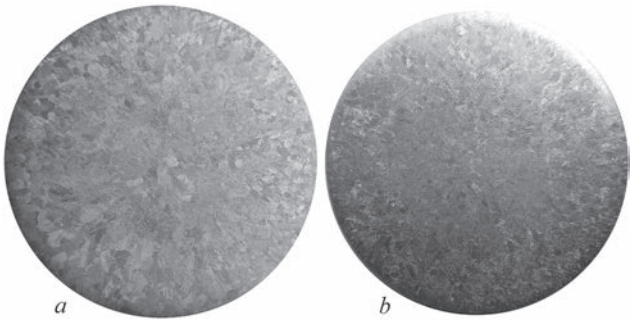

Figure 5. Macrostructure of the cross-section of titanium ingots with a diameter of 80 mm, obtained by ESR at an inert gas pressure of 25 (*a*) and 160 kPa (*b*)

Figure 7 shows the microstructure of VT1-0 titanium ingots obtained at different pressure values. Defects in the form of microporosity, cracks, non-metallic inclusions were not detected. The metal is char-

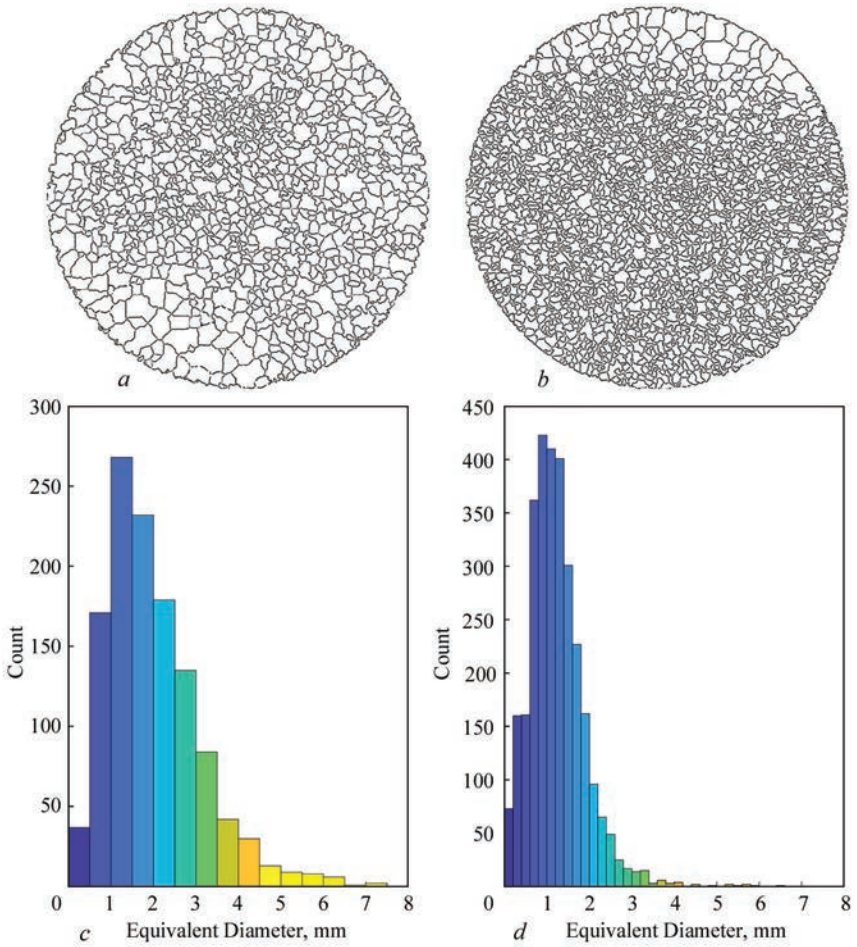

Figure 6. Grain structure (*a*, *b*) and grain size distribution (*c*, *d*) in VT1-0 ingots obtained at pressure: *a*, *c* — 25 kPa; *b*, *d* — 160 kPa

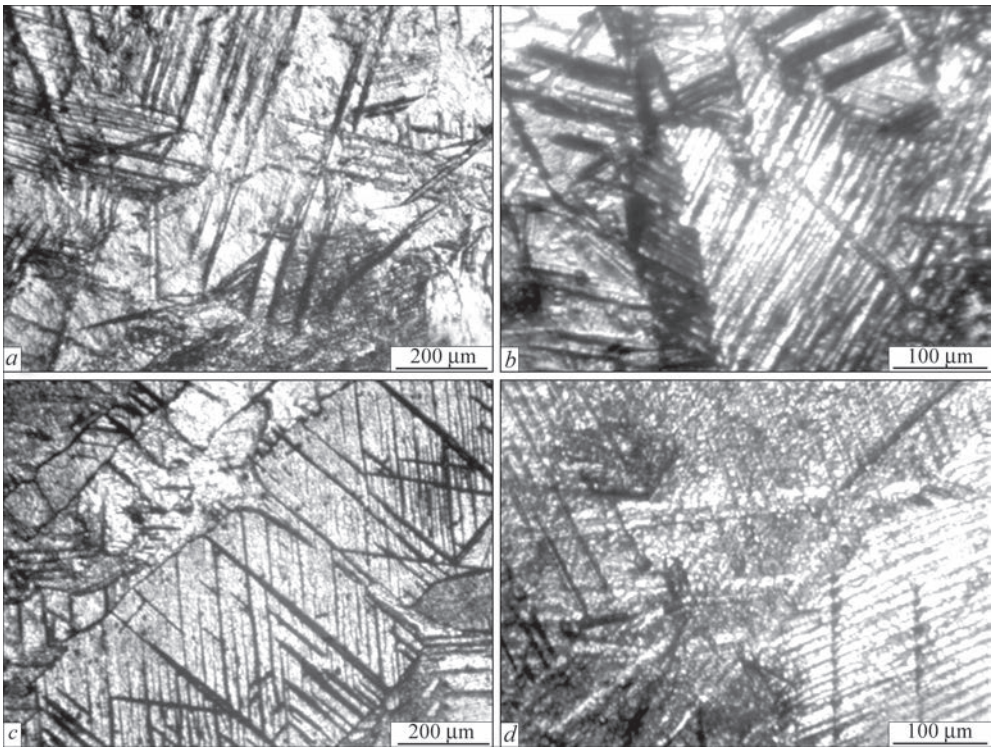

Figure 7. Microstructure of the titanium ingots, obtained by ESR at an inert gas pressure of 25 (*a*, *b*) and 160 kPa (*c*, *d*)

acterized by a coarse lamellar (acicular) structure with a disoriented intra-grain structure. The thickness of α-plates is on average 8–15 μm. This structure is typical for titanium in the cast state and is formed due to high overheating of the melt and low crystallization rates. Fundamental differences in the microstructure of ingots obtained under vacuum conditions (25 kPa) and under excess pressure (160 kPa) were not found.

CONCLUSIONS

1. Ingots of titanium alloys VT1-0 and VT22 were obtained by the ESR method in a chamber-type furnace at different values of pressure in the melting space, from vacuum to excess pressure. It is shown that in the entire investigated pressure range of 20–300 kPa, the electroslag process was stable with the formation of ingots with a high-quality side surface and a dense structure, without pores, slag inclusions and other internal defects.

2. Experimental data on the influence of the pressure in the melting space of the ESR furnace in the range of 20–300 kPa on the gas composition of titanium ingots were obtained. The possibility of reducing the hydrogen content in titanium alloys by 20–35 % by carrying out the ESR process under vacuum conditions (20–25 kPa) has been established. At the same time, a regular influence of the pressure in the melting space on the content of oxygen and nitrogen in titanium ingots in the investigated range was not found.

3. It was established that the VT1-0 titanium ingot, melted under vacuum conditions of 25 kPa, is characterized by a larger grain size compared to the ingot obtained at an excess pressure of 160 kPa. At the same time, the average equivalent diameter of the grains was 2.07 mm and 1.26 mm, respectively.

REFERENCES

1. Protokovilov, I.V., Petrov, D.A., Porokhonko, V.B., Nazarchuk, A.T. (2018) Technological and metallurgical peculiarities of melting of titanium alloy ingots in chamber-type electroslag furnaces. *Suchasna Elektrometal.*, **2**, 45–51 [in Russian]. DOI: http://dx.doi.org/10.15407/sem2018.02.06.
2. Kompan, Y.Y., Protokovilov, I.V. (2002) Some technological aspects of magnetically-impelled electroslag melting (MEM) of titanium alloys. In: *Proc. of Int. Conf. on Special Metallurgy: Yesterday, Today, Tomorrow (8–9 October 2002, Kyiv)*, 256–262 [in Russian].
3. Protokovilov, I.V., Porokhonko, V.B. (2014) Methods to control metal solidification in ESR. *Sovrem. Elektrometall.*, **3**, 7–15 [in Russian].
4. Anoshkin, N.F., Glazunov, S.G., Morozov, E.I., Tetyukhin, V.V. (1978) *Melting and casting of titanium alloys*. Ed. by V.I. Dobatkin. Moscow, Metallurgiya [in Russian].
5. Paton, B.E., Trigub, N.P., Kozlitin, D.A. et al. (1997) *Electron beam melting*. Kyiv, Naukova Dumka [in Russian].
6. Yu, Liu, Xijie, Wang, Guangqiang, Li et al. (2020) Cleanliness improvement and microstructure refinement of ingot processed by vacuum electroslag remelting. *J. of Materials Research and Technology*, 9(**2**), 1619–1630. DOI: https://doi.org/10.1016/j.jmrt.2019.11.087
7. Yu, Liu, Xijie, Wang, Guangqiang, Li et al. (2018) Role of vacuum on cleanliness improvement of steel during electroslag remelting. *Vacuum*, **154**, 351–358. DOI: https://doi.org/10.1016/j.vacuum.2018.05.032
8. Protokovilov, I.V., Petrov, D.A., Porokhonko, V.B. (2020) Investigation of the technological features and admissible pressures of the process of vacuum ESR. *Suchasna Elektro-*

metal. **2**, 3–9 [in Russian]. DOI: https://doi.org/10.37434/sem2020.02.01
9. Kalyniuk, N. (2014) Arrangement of the process for titanium alloys analysis on content of oxygen, nitrogen, hydrogen and carbon impurities. *Metrologiya ta Prylady*, **2**, 50–57 [in Russian].
10. Rashev, Ts. (1995) *High-nitrogen steels. Metallurgy under pressure*. Sofia. Publ. House of the Bulgarian Academy of Sci. [in Russian].
11. Batyshev, A.I. (1990) *Crystallization of metals and alloys under pressure*. 2nd ed. Moscow, Metallurgiya [in Russian].

ORCID
I.V. Protokovilov: 0000-0002-5926-4049
T. Beinerts: 0000-0001-7616-0025
V.B. Porokhonko: 0000-0002-6490-7221
D.A. Petrov: 0000-0003-2937-9299

CONFLICT OF INTEREST
The Authors declare no conflict of interest

CORRESPONDING AUTHOR
I.V. Protokovilov
E.O. Paton Electric Welding Institute of the NASU
11 Kazymyr Malevych Str., 03150, Kyiv, Ukraine.
E-mail: lab38@paton.kiev.ua

SUGGESTED CITATION
I.V. Protokovilov, T. Beinerts, V.B. Porokhonko, D.A. Petrov (2024) Electroslag remelting of titanium under vacuum conditions. *The Paton Welding J*., **3**, 40–45.

JOURNAL HOME PAGE
https://patonpublishinghouse.com/eng/journals/tpwj
Received: 09.01.2024
Received in revised form: 14.02.2024
Accepted: 03.04.2024