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CENTRIFUGAL PLASMA ATOMIZATION OF TITANIUM AND HIGH-TEMPERATURE ALLOY POWDERS USING ELECTRON BEAM MELTED CONSUMABLE BLANKS

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ABSTRACT

Under current conditions, Ukrainian industry faces challenges related to metal powders for additive manufacturing: high cost, difficulties in ensuring stable supplies, and reliance solely on foreign alloy grades are the main factors hindering the adoption of 3D printing in high-tech manufacturing sectors. Therefore, research aimed at developing domestic technologies for the production of spherical powders from titanium and high-temperature nickel alloys is a pressing priority. This paper investigates the process of producing spherical powders using rotary plasma atomization technology and demonstrates the effectiveness of using electron beam melted ingots as feedstock. The study also determines the influence of atomization process parameters on the size and quality of powders made from high-temperature and titanium alloys.

KEYWORDS: ingot, high-temperature alloys, electron beam melting, rotary plasma atomization, spherical powder, additive manufacturing

INTRODUCTION

The development of metal powder manufacturing for 3D printing in Ukraine helps address the issue of consumables' supply and promotes the more active implementation of additive technologies in high-tech industrial sectors, making it a priority area of scientific research. In the current circumstances, additive manufacturing has become particularly relevant for the rapid and precise manufacturing of parts for new-generation gas turbine engines with limited service life.

A general requirement for metal powders used in additive manufacturing is that the particles should be spherical, with a typical size of 15–200 μm. In the Electron Beam Melting (EBM) process, powders with a particle size of 45–160 μm are effectively used; for Selective Laser Melting (SLM) and Selective Laser Sintering (SLS) processes, cold spray (CS) processes require powders with particle sizes ranging from 15 to 53 μm, while direct plasma metal deposition (PMD) and laser-directed energy deposition (LDED) processes use powders with particle sizes ranging from 53 to 150 μm. These particle sizes determine the minimum thickness of the bulk layer and the low surface roughness of the future part. The requirement for a spherical shape is also related to the fact that such particles fit more compactly within a specified volume and exhibit the required flowability in the powder feed system with minimal resistance [1]. At the same time, the powders must have high chemical homogeneity and a low content of harmful impurities; their structure must be free of lacks of fusion, pores, cavities, and inclusions.

The main powder production technologies capable of meeting these requirements are melt dispersion methods. The main technological variants of melt dispersion include gas atomization, where VIGA (Vacuum Induction Melting and Inert Gas Atomization) of an ingot or EIGA (Electrode Induction Melting and Inert Gas Atomization) of a rod takes place in a vacuum chamber, followed by atomization with a jet of cold inert gas, as well as centrifugal (rotational) atomization. Plasma atomization (PA) of wire is another technology within this group [2, 3]. Among the main drawbacks of VIGA, EIGA and PA technologies are the presence of irregularly shaped particles in the atomized powder batches, as well as satellite particles with internal porosity. Strict requirements for additive powders do not allow the aforementioned defects, as they can cause instability in the 3D printing process. In the domestic industry, the implementation of additive technologies has encountered a number of problems related to the source powders. The high cost of raw materials — specifically, foreign-produced spherical powders — along with difficulties in securing a stable delivery and the limited availability of foreign alloy grades, are the main limiting factors. Nickel and titanium alloys for additive manufacturing have the greatest potential, since the raw material is very expensive, and the cost of purchasing rods made of the required alloy accounts for about half the price of the ready part. Therefore, an important task is to replace imported powder materials with domestically produced powders. First of all, this requires the development of technologies and improvements to equipment for producing powder materials from various alloys. One way to improve the process chain in this regard could be the use of ingots melted by leading Ukrainian companies (as a substitute for purchased rods).

The PREP (Plasma Rotating Electrode Process) spraying technology is capable of producing high-quality powders that contain almost no defective particles [4]. However, for today, a number of issues regarding the development of plasma atomization technologies specifically using cast billets remain unresolved. Among the enterprises that possess specialized equipment and many years of experience in melting ingots from titanium, heat-resistant, nickel, and special alloys is the State Enterprise "Scientific and Production Center "Titan" of the PWI of the NAS of Ukraine" [5]. At the facilities of this enterprise, ingots were produced using electron beam technology and were used to manufacture the spherical powders studied in this work.

The equipment currently available in Ukraine for production of spherical powders (of the UTsR type) is more than 30 years old, and its technological capabilities at the time of its development were characterized by a rotational speed of the consumable electrode in the range of 15000–18000 min^{-1}, which allowed for the production of granules of approximately 200–250 μm in size [6]. Taking into account modern requirements for powders used in additive manufacturing, the equipment for plasma powder atomization needed extensive upgrading.

Multiflex LLC (Zaporizhzhia) is a leading manufacturer of spherical metal powders produced by rotary plasma atomization, where the equipment and manufacturing processes as a whole have been upgraded. The design of the new spherical powder production facility for this enterprise was carried out by the "Titanium Research and Design Institute" LLC (Zaporizhzhia), resulting in a complete upgrading and reconstruction of the facility. The production assignment set for this project was 100 kg per one cycle for titanium-based powders and 250 kg for nickel alloys, with a yield of at least 93 % of powder meeting the specified requirements to particle size distribution. Thus, the development of plasma atomization technologies using cast billets was carried out at Multiflex LLC.

The aim of the work was to determine the optimal process parameters for rotary plasma atomization of electron beam melted ingots made of titanium and heat-resistant nickel alloys for the production of dispersed (40–120 μm) spherical powders for additive manufacturing.

EQUIPMENT AND METHODS

The ingots (53 mm in diameter, L = 700 mm) were melted using the UE-208M multipurpose electron beam unit, equipped with gas-discharge electron guns and a cylindrical mould. A schematic of the UE-208M electron beam system is shown in Figure 1. The process equipment used for the electron beam remelting

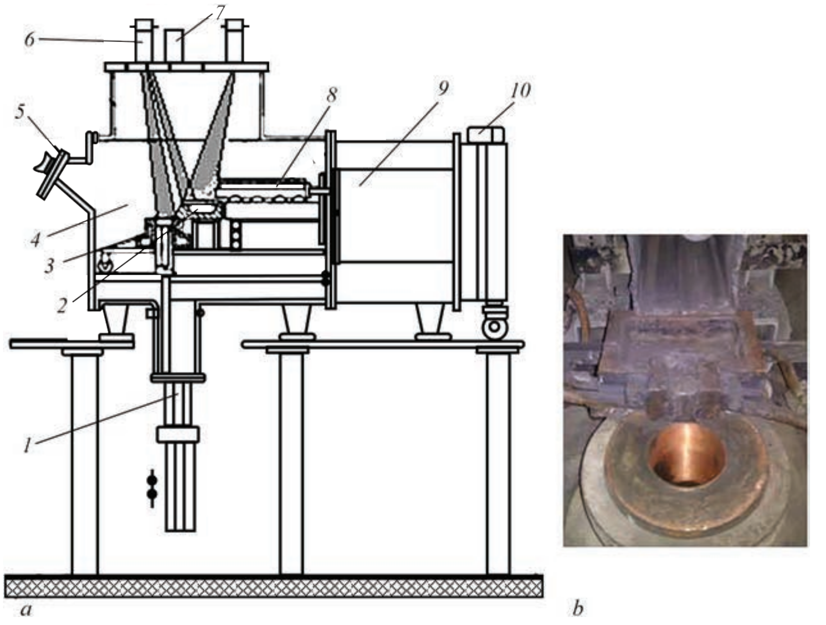

Figure 1. Schematic of the UE-208M electron beam installation (*a*) and appearance of the mould (*b*) [5]: *1* — ingot extraction mechanism; *2* — cold hearth; *3* — mould; *4* — melting chamber; *5* — viewing system; *6* — electron beam gun; *7* — water rheostat; *8* — feed billet; *9* — chamber; *10* — billet feed mechanism

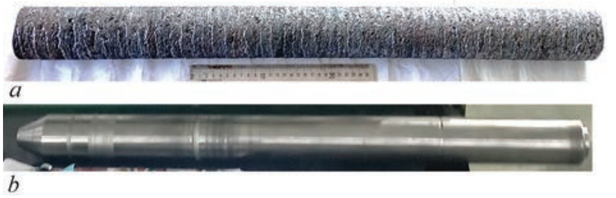

Figure 2. Appearance of an electron-beam-melted heat-resistant nickel alloy ingot before (*a*) and after (*b*) mechanical treatment

process consists of an cold hearth and a mould, which is a hollow closed loop equipped with a vertically movable bottom plate with a dovetail-shaped depression. The inner part of the mould, which comes into contact with the ingot metal, is made of copper and water-cooled (Figure 2). The unit's vacuum system creates and maintains a vacuum in the melting chamber (up to $6\cdot10^{-2}$ Pa at the start of melting and 10^{-1} Pa during melting).

After melting, the ingots were cooled in the melting chamber under vacuum; they had a light-colored, unoxidized surface (Figure 2, *a*). The depth of corrugation-type surface defects did not exceed 2 mm; no defects in the form of tears, cracks, or lack of fusion were detected. For subsequent rotary plasma atomization, the ingots underwent mechanical surface machining to a diameter of 50 mm (Figure 2, *b*).

Rotary plasma atomization of cast billets was performed using an upgraded UTsR-4 system (Figure 3); the plasma-forming gas mixture consisted of argon and helium in various ratios. Experimental spraying was conducted using Grade 5 titanium alloy (Ti–6Al–4V) and the heat-resistant nickel alloy INCONEL 718, whose chemical compositions are given in Table 1.

In the PREP process, the plasma arc acts on the end face of a rapidly rotating billet (consumable electrode), causing melting of the metal and forcing it toward the periphery of the consumable electrode by centrifugal forces. As the power of the plasma arc grows, the amount of molten material (spraying rate) increases due to a decrease in the viscosity of the molten material resulting from the rise in temperature and its faster movement toward the rim, where the molten material disperses into droplets. Experimental atomization using the PREP technology was carried out under vacuum conditions in the chamber at a level of 10^{-3} Pa, after which a mixture of argon and helium was introduced in a specific ratio. The consumption of the plasma-forming gas mixture per spraying pro-

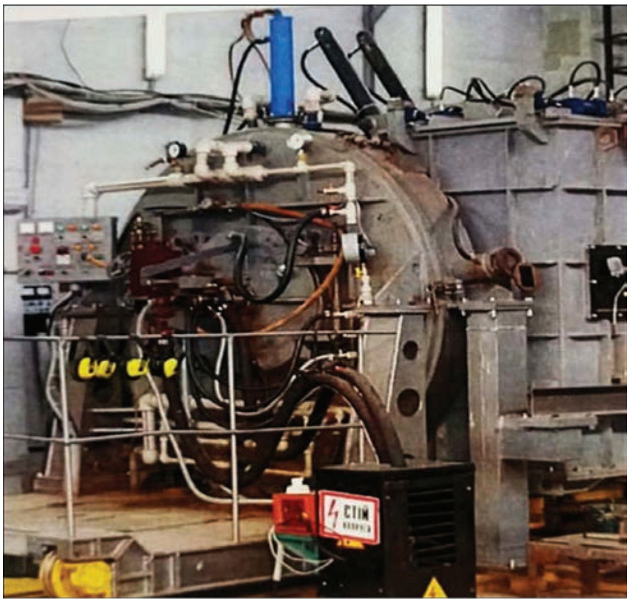

Figure 3. Appearance of the UTsR-4 equipment for the manufacture of metallic spherical powders by Multiflex LLC using the PREP technology

cess was approximately 12 m^3, with a gas flow rate of about 40 l/min, and this value remained constant for all experiments. Gas flow rates were measured using a rotameter and regulated via a special diaphragm in the area of the gas mixture inlet (16 mm in diameter). The plasmatron arc power depended on the Ar/He ratio in the gas mixture and the current, the specific value of which (within the range of 700–1200 A) was set for each spraying operation and remained unchanged during the process.

The resulting powders were tested in accordance with the national standard "Metal Powders" (DSTU 2049–92). The particle size distribution was determined using the standard sieve analysis method with sieves of various mesh sizes (from 40 to 200 μm), as well as through microscopic examination. The obtained fractions were analyzed to determine their particle size, shape, and percentage distribution in the sample. Statistical methods were used to process the analysis results, specifically to determine the average particle sizes and their size distribution.

The bulk density and flowability of the powder were determined using the Hall funnel method in accordance with ISO 4490–78. For metallographic analysis, the powder was embedded in an electrically conductive resinous material and polished using diamond suspensions. Microstructural analysis was per-

Table 1. Chemical composition of alloys for the manufacture of spherical powders, %

Alloy grade	C	O	Si	Mn	Cr	Ni	Ti	V	Al	Nb
Ti–6Al–4V (Grade 5)	≤0.10	≤0.20	≤0.1	–	–	–	Base	3.5–4.5	5.5–6.75	–
INCONEL718	≤0.10	0.02–0.05	≤0.30	≤0.6	14.0–16.0	43.0–47.0	1.9–2.4	–	0.9–2.4	0.8–1.5

formed using an inverted reflected-light microscope (Carl Zeiss).

Microhardness was measured using a BUEHLER MM7T device. To measure microhardness of powder particles, microsections with polished surface were prepared; the powder was pressed into Bakelite using a SIMPLIMET 2000 automatic pressing machine. The measurements were taken on powder particles that were fully visible in the polished section. Microhardness in the surface area of the powder particles was measured at a distance of ~15–18 μm from the edge, and in the central area of the particles — at a distance of ~1/2 of the diameter; at least five measurements were taken.

The chemical composition of the studied samples was determined by spectral analysis using a SPECTROMAX instrument manufactured by SPECTRO Company, in which the analysis is based on determining the intensities of the excited spectrum from each chemical element, which is directly proportional to its weight fraction in the alloy. The oxygen content in the powder particles was determined using an ON900 gas analyzer manufactured by ELTRA Company. The principle for determining gaseous impurities involved burning the specified samples (or a portion of powder weighing the same as a sample of 3.5 mm length and 5 mm diameter for titanium alloys, and 3 mm diameter for nickel alloys) and determining the content of the released oxygen.

RESEARCH RESULTS

Gases that are inert with respect to titanium and nickel — argon and helium — were used as plasma-forming gases. Selecting the optimal composition of the plasma-forming gas mixture and the optimal rotational speed of the consumable electrode is a significant part of improving the atomization technology for nickel heat-resistant and titanium alloys. For this purpose, experimental centrifugal spraying processes were conducted at various levels of these parameters (Table 2). To determine the optimal process parameters for rotary plasma atomization of a consumable electrode made of heat-resistant aviation alloys to produce powders with different particle size distributions, a series of experimental spraying tests was conducted. For each spraying, the following process parameters were varied: the rotational speed of the consumable electrode (X_1); and the helium content He(X_2) % in the plasma-forming gas mixture (argon + helium).

Table 2. Numerical characteristics of the plasma atomization process parameters that were varied during the study

Experiment	X_1 (min^{-1})	X_2 (He, %)
1	20 000	30
2	30 000	
3	20 000	60
4	30 000	
5	25 000	45

The response variable is the particle size of the powder (Y, μm).

A helium content of 30 % was selected based on research data [7, 8] as the lower limit, and a value twice as high was chosen as the experimental value, which became the upper limit of the variation range. The higher value (60 % He) was not considered because helium increases the plasma's melting capacity and, as a result, a deep crater may form on the end surface of the melting consumable electrode, which negatively affects the spraying process and powder quality. Other process parameters were as follows: voltage — 50–70 V; plasmatron power — within the range of 65–100 kW.

For Grade 5 titanium alloy (Ti–6Al–4V) and INCONEL 718 nickel alloy, the specified powder size was 40–110 μm (particle size distribution as per the customer's requirements). The introduction of helium into the plasma-forming gas mixture with argon had a positive effect on reducing the particle size of the Grade 5 alloy powder, with the greatest effect observed when the He content reached 60 % in the mixture with Ar at a maximum billet rotational speed of 30000 min^{-1}. Based on experimental spraying conducted according to the experimental design matrix (where X_1 — rotational speed and X_2 — helium content in the plasma-forming mixture were varied). The obtained dependencies demonstrate the combined effect of these two parameters on the particle size of the powders. The particle size dependencies (Y_1, Y_2, μm) for powders made of Grade 5 alloys (1) and INCONEL 718 (2) were linear and took the following form:

$$Y_1 = 212.90 - 0.00283 \cdot X_1 - 0.833 \cdot X_2; \quad (1)$$

$$Y_2 = 291.04 - 0.00563 \cdot X_1 - 1.375 \cdot X_2; \quad (2)$$

The obtained equations demonstrate that an increase in rotational speed and helium content reduces the average particle size and allows for the prediction of process parameters for the production of spherical powders of the desired size.

As a result of optimizing the process parameters, an improved technology for rotary plasma atomization of nickel heat-resistant and titanium alloys was developed by increasing the plasma power in combination with an increase in the rotational speed of the consumable billet. The plasma power was increased by altering its thermophysical properties through the introduction of helium, an inert gas, into the argon

Table 3. Technological properties of different batches of powders manufactured using upgraded centrifugal plasma atomization equipment

Alloy grade	Powder particle size distribution, μm	Flowability (per 50 g sample), s	Bulk density, g/cm^3
Ti–6Al–4V (Grade 5)	–100+50	15	1.5
	–60+30	19	2.0
	–40	21	2.5
INCONEL 718	–50+30	15	4.4
	–30	11	5.1

at an optimal ratio. The ability to predict the particle size distribution of powders using regression relationships between plasma atomization process parameters made it possible to produce powders of various sizes, optimized for specific 3D printing technologies used to fabricate products from them. Thus, the optimal spraying conditions for obtaining the specified powder particle size for the studied alloys were determined as follows: rotational speed — 30000 min^{-1}; gas ratio in the plasma-forming mixture — 60 % He/4 % Ar.

In addition to particle size, additive powders must meet a number of quality requirements, the key among which are sphericity, the absence of satellites, the absence of surface oxidation, the absence of internal porosity, uniformity of chemical composition and microstructure, as well as the required level of the powder's processing properties (Table 3).

The Grade 5 (Ti–6Al–4V) titanium alloy powder exhibited higher flowability values than nickel alloys, but its bulk density is higher than that of the titanium alloy. This difference in technological properties can be explained by the different specific weight of the materials under study, as well as by the influence of particle size on powder quality.

Table 4. Microhardness of spherical powder particles

Alloy grades	Powder particle size distribution, μm	Microhardness of powder particles, $HV^{0.05}$	
		Surface	Core
Ti–6Al–4V (Grade 5)	40–100	275–311	220–295
INCONEL 718	60–110	370–392	395–400

Microanalysis revealed that the manufactured powders had a regular spherical shape, without satellites, micropores, or structural changes on the particle surface (Figure 4), which met the requirements of additive manufacturing technologies for the starting powder.

The microstructure of the powder particles was distinguished by ultrafine grains and a fine crystalline structure, which can provide the optimal combination of strength and ductility — that is, maximize the potential of the alloys under study.

During the crystallization of spherical powder particles, a more significant deviation from the phase equilibrium was observed in the alloys under study than during the crystallization of ingots. Conditions were created for the formation of highly dispersed, pseudo-homogeneous mixtures, which improved the mechanical properties and may also enhance the effectiveness of subsequent strengthening heat treatment. The homogeneity of the microstructural state was confirmed by microhardness measurements of spherical powder particles carried out on transverse micro-polished sections (Table 4).

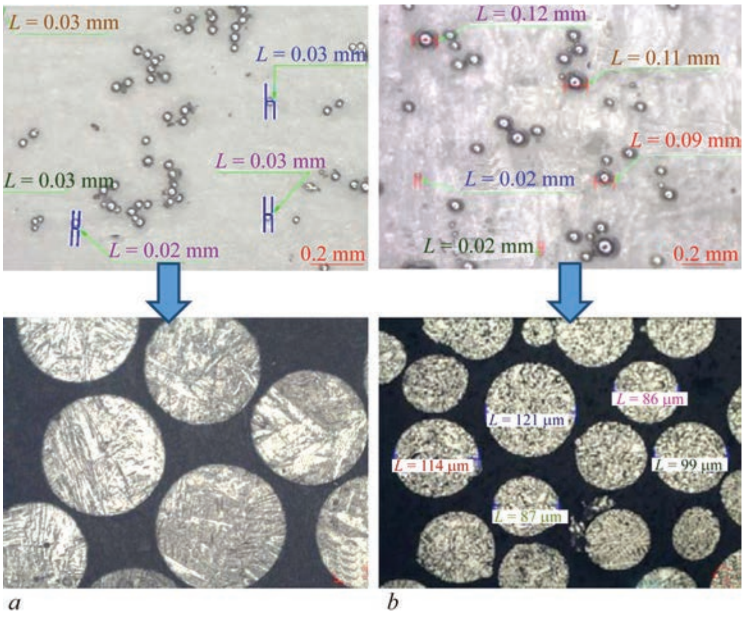

Figure 4. Surface condition (×20) and microstructure of powder particles manufactured using the upgraded equipment of LLC "Multiflex" from Ti–6Al–4V (*a*) and Inconel 718 (*b*) alloys, ×500

Table 5. Comparison of the basic parameters of Ti–6Al–4V alloy powders (Grade 5, Grade 23) from different manufacturers [9–12]

Manufacturer	Hoganas (Sweden)	Sino-Euro Materials Technologies of Xi'an Co., Ltd (China)	AP&C a Colibrium Additive Company (USA)	Pyrogenesis (Canada)	"Multiflex" LLC (Ukraine)
Particle size distribution, μm	45–106	85–90 125–130	45–150	45–106	40–100
Bulk density, g/cm³	2.35	2.5–2.7	2.59	2.51–2.65	2.2–2.5
Flowability, s, per 50 g	29	20–25	25	21–28	15.0–21.0
Oxygen content, %	0.17	0.08–0.10	0.13	0.13	0.15

The microstructural state and chemical homogeneity of the particles, as confirmed by microhardness measurements across the surface and in the cross-section of the powder section, as well as the absence of gas saturation in the powders and nonmetallic inclusions, demonstrated that the upgraded equipment and the developed process parameters for atomizing ingots enable the production of high-quality spherical dispersed powders for use in additive manufacturing technologies for various parts. As a result of the carried out research, the Company Multiflex has drawn up the following technical specifications: "Spherical titanium alloy powders for additive manufacturing processes" (TS U 24.4-31914753-001:2018), "Nickel-based and iron-nickel-based alloy powders" (TS U 24.4-31914753-002:2020).

A comparative analysis of metal powders produced by the Ukrainian Company Multiflex LLC with those from foreign manufacturers has shown that both foreign and Ukrainian powders have stable and narrow particle size distributions, which enables achieving reproducible results in laser and electron beam printing, among other applications. The oxygen content in foreign powders (which has a critical influence on ductility and susceptibility to brittle fracture) is close to the oxygen content in powders manufactured by Multiflex LLC; and in terms of the considered quality indices, Ukrainian powders can compete with their foreign counterparts (Table 5) [9–12].

In terms of technical specifications and oxygen content, the powders manufactured by Multiflex LLC are comparable to those of a number of industrial manufacturers, such as "Hoganas" (Sweden), "Sino-Euro Materials Technologies of Xi'an Co., Ltd" (China), "AP&C, a Colibrium Additive Company" (USA) and "Pyrogenesis" (Canada).

It should also be considered that domestic powders offer advantages in terms of cost and availability. Considering the current level of production organization, the production cost of Grade 5 (Ti–6Al–4V) alloy powder from Multiflex LLC is by 40–50 % lower than that of European or Canadian equivalents. Furthermore, local production allows the chemical composition and particle size distribution to be adapted to the customer's specific requirements. The wholesale price of Ti–6Al–4V alloy powders on the international market is approximately 200 Euros per kg, whereas Ukrainian companies implementing additive manufacturing methods have to pay twice as much to acquire them, resulting in a high production cost for parts manufactured from these powders. Furthermore, this creates a dependence on foreign suppliers for raw materials. The use of electron-beam melting ingots as consumable electrodes has made it possible to establish a closed production cycle without relying on foreign supplies of deformed rods.

CONCLUSIONS

1. The results of the practical implementation of electron beam-melted ingots of 50 mm diameter and 700 mm length as consumable electrodes for the manufacture of dispersed spherical powders using the PREP process from the titanium alloy Ti–6Al–4V (Grade 5) and the heat-resistant nickel alloy INCONEL 718 are shown.

2. Optimal process parameters have been determined for the rotational plasma atomization of ingots made from Grade 5 alloy and INCONEL 718 nickel alloy to ensure a yield of the specified particle size range of 40–100 μm, which can be used in additive manufacturing processes such as electron beam melting (EBM), direct plasma metal deposition (PMD) and laser direct energy deposition (LDED). The optimal process parameters were 30000 min^{-1} and a gas ratio of 60 % He/40 % Ar.

3. The powders manufactured in accordance with optimized technological parameters, met the quality criteria for powders intended for additive manufacturing: the powder particles had a regular shape without satellites, were characterized by the absence of internal porosity and nonmetallic inclusions, and their particle size fell within the specified range. As a result of the conducted research, technical specifications have been developed for spherical powders made from ti-

tanium alloys (TS U24.2431914753-001:2018) and for powders made from nickel-based and iron-nickel-based alloys (TS U24.431914753-002:2020).

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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