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SPHERICAL TITANIUM POWDER PRODUCTION FOR 3D PRINTING BY PLASMA-ARC ATOMIZATION OF WIRE MATERIALS

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ABSTRACT

The possibility of spherical titanium powder production by plasma-arc atomization of solid Cp-Ti Grade 2 wire with a diameter of 1.0 and 1.6 mm has been experimentally confirmed. Analysis of the particle size distribution of the powder showed that in the case of atomization of titanium wire with a diameter of 1.0 mm, the main fraction is –140 μm, which is 96 % of the total mass of the powder, where the amount of the finely dispersed fraction of –63 μm is up to 60 wt. %, and in the case of wire with a diameter of 1.6 mm, the main fraction is –200 μm, which is 95 wt.%, while the amount of the finely dispersed fraction of –63 μm does not exceed 38 wt.%. A study of shape parameters of the titanium powder was performed, which showed that most particles have a regular spherical shape with an average sphericity coefficient close to 0.9, the number of particles with satellites and particles of irregular shape does not exceed 1 wt.%, which determines the high technological properties of the produced powder, which are on a par with other industrial technologies of spherical powder production by plasma and gas atomization methods. The chemical and phase composition of the atomized powder was investigated, and it was found that the phase composition consists of α-Ti, and the chemical composition corresponds to the ASTM B 348-05 standard. It was shown that application of the technology of plasma-arc atomization of titanium wire allows obtaining spherical powders that can be used as consumables for 3D printing of products for the aviation, rocket and space, medical, energy and chemical industries by the methods of electron beam melting (EBM), laser direct energy deposition (LDED) and plasma metal deposition (PMD).

KEYWORDS: plasma-arc atomization, wire, compact section, titanium, sphericity, powders, 3D printing

INTRODUCTION

Recently, due to the intensive development of aviation, rocket and space, medical, energy and chemical industries there has been a significant need in manufacturing volumetric complex-shaped parts from titanium and its alloys, predominantly using additive technologies of 3D printing [1, 2].

The main additive technologies of growing products from titanium and its alloys include: Selective Laser Melting (SLM), Selective Laser Sintering (SLS), Electron Beam Melting (EBM), methods of Laser Direct Energy Deposition (LDED) and Plasma Metal Deposition (PMD), etc. All these methods use specialized spherical powders (predominantly finely-dispersed) as consumable materials to form the additive layers, with strict requirements being made of the powder particle size distribution, shape, and physical-chemical and technological properties. So, for instance, powders of a narrow fraction of 15–45 μm are used for SLM process, powders of 45–106 μm are applied for EBM, those of 45–150 μm are used for LDED, and powders of 63–160 μm fraction are designed for PMD technology [3–5]. Moreover, the above powders should have a spherical shape with minimal number of external (satellites) and internal (pores) defects, good technological properties (high flowability, bulk density, coefficient of sphericity, etc.) and low content of gas mixtures, providing a high packing density of the additive layers, reduction of porosity and improvement of mechanical properties of the final product [6–8].

LITERATURE DATA ANALYSIS AND PROBLEM DEFINITION

At present, Vacuum Inert Gas Atomization (VIGA) is the most widespread technology for producing titanium powders for additive manufacturing. Alongside a number of advantages, described in works [9, 10], the above-mentioned technology has a number of essential drawbacks, namely [10-13]: presence of a large number of satellites and irregularly-shaped particles, lower coefficient of sphericity for the methods of gas atomization, of the melt resulting in low values of flowability (particularly for the fine fraction of <63 μm), and leading to defect formation in the deposited layers; presence of intragranular argon porosity for powders produced by gas atomization technology, which in some cases cannot be removed by further cold or hot pressure treatment; high cost of powder manufacture by gas atomization, as the gas-to-metal ratio (flow rate (m^3) of atomizing gas — argon, required to produce 1 kg of powder) can be equal to 26-110.

Plasma Rotating Electrode Process technology (PREP) has also become widespread, where the plasma jet melts the surface layer of the billet end face, and melt atomization is carried out due to off-center forces. However, this method also has several essential drawbacks, as PREP equipment operation involves considerable difficulties of producing –106 μm fraction: to achieve more than 40 vol.% yield of the above-mentioned fraction there is the need for a significant increase in the speed of billet rotation (more than 30 000 rpm) that complicates the already not at all simple kinematic diagram of the unit (lowering of the level of vibrations, designing complex bearing systems are required, etc.). We can also include here the difficulties associated with producing a cylindrical billet of precise dimensions, which should be ground with a high accuracy [12].

In this respect, prospects for manufacturing spherical powders open up, due to application of technologies, where wires or rods (ingots) are used as feedstock materials, and the processes of melt heating, melting and dispersion are performed by plasma jets, without using off-center forces, or high-speed equipment with a complex kinematic diagram. Among such technologies is the process of plasma-arc atomization, which is flexible, and can be used to produce powders from a wide class of materials (billets), which are either manufactured by industry (wires, rods), or can be produced by casting methods in the form of ingots [13, 14].

At present, in connection with the transition of additive manufacturing to a new level in Ukraine, its expansion and provision with consumables in full, particularly due to intensive progress of aviation and rocket and space industries, requires development of domestic technology and equipment to produce finely-dispersed spherical powders, particularly with the fractional composition of 63 μm, which is a relevant science and technology task.

RESEARCH OBJECTIVE AND TASKS

The objective of the work is analysis of the possibility and assessment of the prospects for application of the process of plasma-arc atomization of Cp-Ti Grade 2 titanium wire 1.0 and 1.6 mm in diameter for further development of the domestic technology and equipment to produce spherical powders for 3D printing. To achieve this goal, it is necessary to study the particle size distribution, morphology, microstructure, technological properties and chemical and phase composition of the powder manufactured using the technology of plasma-arc atomization of Cp-Ti Grade 2 titanium wire; provide conclusions regarding the possibility of application of the produced spherical powders for 3D printing by the methods of Selective Laser Melting (SLM) and Electron Beam Melting (EBM), Selective Laser Sintering (SLS), Laser Direct Energy Deposition (LDED) and Plasma Metal Deposition (PMD).

INVESTIGATION MATERIALS AND PROCEDURE

PROCEDURE OF WIRE ATOMIZATION AND DETERMINATION OF FRACTIONAL COMPOSITION OF THE POWDER

Experiments were performed using “PLAZER 50-PL-W” process equipment for plasma-arc atomization of wire materials (manufacturer is “SPC “PLAZER” Ltd, Ukraine) [15], with application of an experimental plasmatron with a copper hollow electrode operating by the scheme of “indirect action arc” (Figure 1, *a*, *b*) [16]. Technology of plasma-arc atomization of wire materials (Plasma Atomization (PA)) is well described in works [17–19], and in the general case it consists in melting the wire, fed into the zone of

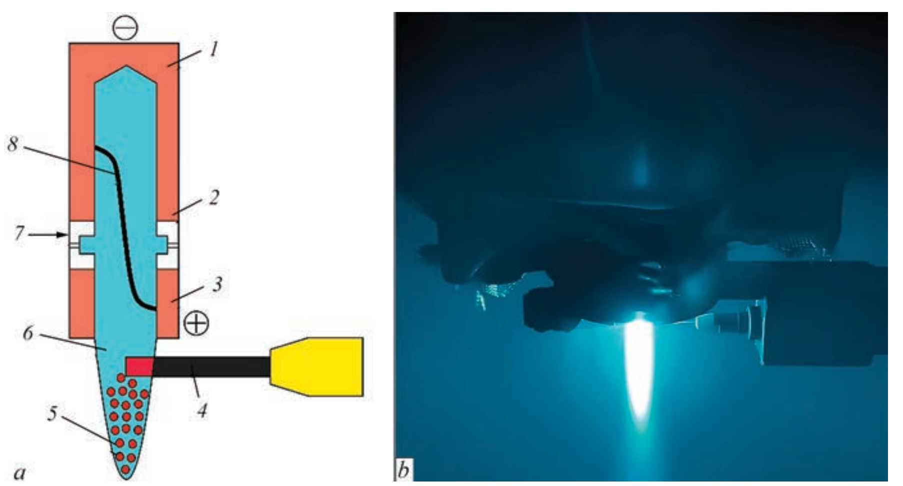

Figure 1. Scheme (*a*) and visualization (*b*) of the process of plasma-arc atomization of Cp-Ti Grade 2 titanium wire in the atomization chamber

plasma jet outflow under the plasmatron nozzle edge and further fragmentation of the melt layer, formed at the atomized wire tip. This method provides formation of a high-speed (in some cases, supersonic) plasma jet, which greatly increases the dynamic pressure on the dispersed melt, and leads to its intensive fragmentation, that, in its turn, creates the conditions for producing an optimal particles size distribution of the dispersed phase.

Plasma-arc atomization of the wire was conducted in a laboratory atomization chamber in an argon atmosphere, according to the procedure of [20], where pumping down of the chamber inner volume to residual pressure of $5–7\times10^{-3}$ Torr was performed before filling it with gas. Residual pressure values were monitored, using thermocouple manometric transducer PMT-2 (Ukraine) and ionization-thermocouple vacuum gauge VIT-2 (Ukraine).

Parameters of the mode of plasma-arc atomization of the neutral wire were as follows: current of 300 A; arc working voltage of 140 V; plasma-forming gas flow rate of 15 m^3/h, wire feed rate of 3–7 m/min, distance from the plasmatron nozzle edge to the atomized wire of 4 mm. Plasma-forming gas was argon of the highest grade I1 to ISO 14175-2008 in both the cases.

Acceptable atomization modes were determined after achieving of the minimal angle of opening of the plasma jet had and the process stability during melting of the atomized wire in the axial zone of the plasma jet.

Particle size distribution of laboratory batches of the powders was determined by sieve analysis in keeping with the procedure of ISO 2591-1:1988 in shock sieve analyzer "AS 200U (ROTAP)" (Ukraine) with a set of sieves with hole dimensions of 25–400 μm, sample weight being not less than 200 g of powder.

ATOMIZATION MATERIALS

Solid wire from commercial Cp-Ti Grade 2 titanium 1.0 and 1.6 mm in diameter was used as atomization material. Its composition is given in Table 1.

DETERMINATION OF POWDER MORPHOLOGY AND MICROSTRUCTURE

The appearance of powder particles was studied in scanning electron microscope JEOL JSM-840 (Japan) at the following parameters: $U = 20$ kV, $I = 10^{-10}–10^{-7}$ A. Images were obtained in secondary electron mode of ZAF/PB computer program, designed for studying rough surfaces. Further analysis of the images for powder sphericity investigation was performed in MIPAR software product (USA) by the procedure of [21].

Table 1. Chemical composition of atomized Cp–Ti grade 2 wires according to the certificate of origin, wt.%

Ti	Fe	O	C	N	H
Base	0.2	0.18	0.05	≤0.03	0.012

DETERMINATION OF THE POWDER PHASE COMPOSITION

Phase composition of the powder and lattice parameters of the individual phases were studied by the method of X-ray diffraction phase analysis (XDPA). X-ray studies were conducted in DRON-M1 diffractometer (Ukraine) in monochromatic CuK_α-radiation. Used as a monochromator was a graphite single crystal mounted on a diffracted beam. Diffractograms were recorded by step-by-step scanning in the range of angles $2\theta = 10–120°$. Scanning step was equal to 0.05°, exposure time was 3–9 s. Diffractometric measurement data was processed using programs for full-profile analysis of X-ray spectra from a mixture of polycrystalline components PowderCell 2.4 (Germany).

DETERMINATION OF ELEMENTAL CHEMICAL COMPOSITION OF THE POWDER

The method of X-ray spectral microanalysis (XMA) with application of Link 860/500 detector (Great Britain) was used to determine the elemental chemical composition of the titanium powder.

DETERMINATION OF THE CONTENT OF OXYGEN AND NITROGEN IN THE POWDER

Investigations of the content of oxygen and nitrogen in the atomized powders were performed in gas analyzer LECO TC-436 Nitrogen Oxygen Analyzer (USA).

DETERMINATION OF THE TECHNOLOGICAL PROPERTIES OF THE POWDER

Powder flowability was determined in Hall instrument according to ASTM B213 standard. Measurements of bulk density were conducted according to ASTM B212 standard.

INVESTIGATION RESULTS AND DISCUSSION

Studies of particle size distribution of the produced powder (Figure 2) showed that the largest quantity of the powder finely-dispersed fraction is observed at atomization of 1.0 mm titanium wire, where the main powder fraction is 140 μm, making up 96 wt.%, and the proportion of the finely-dispersed fraction of 63 μm is 59 wt.%. In the case of atomization of 1.6 mm titanium wire, the main powder fraction is 200 μm, which is 95 wt.%, and the quantity of fine-

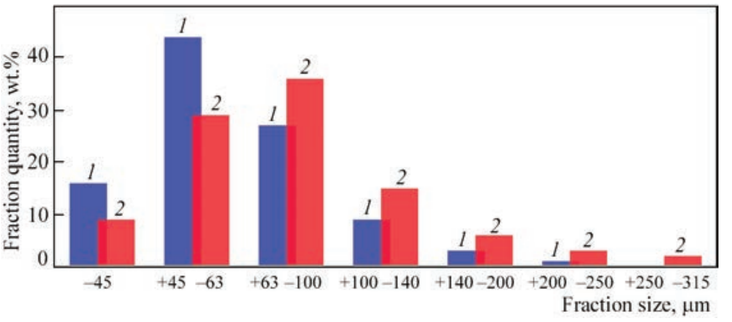

Figure 2. Distribution of the particle size distribution of powder produced in plasma-arc atomization of Cp–Ti Grade 2 titanium wire with a diameter of 1.0 (*1*) and 1.6 mm (*2*)

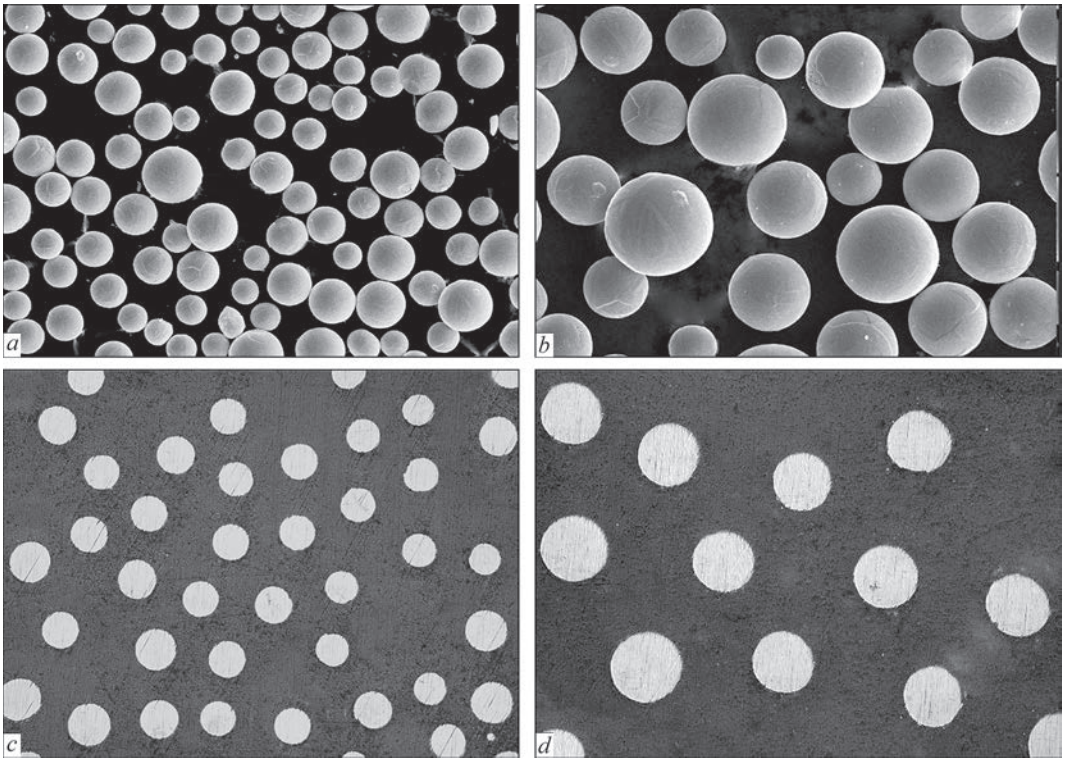

Figure 3. SEM images of the morphology (*a, b*) and microstructure (*c*, *d*) of Cp-Ti Grade 2 titanium powder of 106 μm fraction; *a*, *c* — ×100; *b*, *d* — ×200

ly-dispersed powder fraction of 63 μm does not exceed 38 wt.%.

Formation of finer powders during atomization of 1.0 mm titanium wire with other unchanged process

Table 2. Comparison of the technological properties of Cp-Ti Grade 2 titanium powder produced by SS-PREP, GA and PA technologies

Method	Fraction size, μm	Flowability, s/50 g	Bulk density, g/cm^3
SS-PREP (Sino-euro Ltd, China) [23]	15–45	≤30	≥2.5
	45–106	≤27	≥2.5
	45–150	≤27	≥2.5
GA (Hoganas, Sweden) [24]	15–45	40	2.34
	45–106	35	2.37
	45–150	33	2.37
PA (AP&C, USA) [25]	15–45	29	2.55
	45–106	25	2.61
	45–150	23	2.65
PA (PWI, Ukraine)	15–45	32	2.46
	45–106	28	2.53
	45–150	25	2.57

Table 3. Chemical composition of the feedstock wire and titanium powder Cp-Ti Grade 2, wt.%

Studied zone	Ti	Fe	O	N
ASTM Standard B 348-05	Base	≤0.30	≤0.25	≤0.03
Initial wire		0.18	0.23	0.02
Powder of 106 μm fraction		0.19	0.20	0.01
Powder of +106, –106 μm fraction		0.21	0.22	0.02

Table 4. Results of XDA of Cp-Ti Grade 2 titanium powder of –106, +106; –160 μm

Sample	Phases	Phase content, vol.%	Lattice parameters, Å
Powder fraction of –106 μm	α-Ti	100	a = 2.9487; c = 4.6850; c/a = 1.5888
Powder fraction of +106; –160 μm			a = 2.9501; c = 4.6871; c/a = 1.5887

parameters is attributable to smaller dimensions of the liquid interlayer, compared to those of the interlayer forming at the tip of atomized 1.6 mm wire. This, in its turn, promotes reduction in the dimensions of the fragments forming at atomization [16].

Analysis of titanium powder morphology in MIPAR software (Figure 3) in the case of 106 μm fraction showed that the powder is of a spherical shape, with average coefficient of sphericity S = 0.87–0.91. External defects in the form of satellites and irregularly-shaped particles are practically absent, their proportion not exceeding 1 wt.%, and dendrites and grain boundaries coming to the powder surface are observed. Investigations of powder microstructure showed absence of internal defects in the form of cavities, cracks, etc.

High indices of sphericity of the studied powders determine their high technological properties (Table 2), which can be compared to the properties of powders produced by the SS-PREP technology ([22]) at the speed of more than 30 000 rpm.

XMA and gas analysis methods were used to establish the chemical composition of powders (Table 3), produced during plasma-arc atomization of Cp-Ti Grade 2 titanium wire, and to determine that it corresponds to the chemical composition of the feedstock material — the wire (ASTM B 348-05 standard).

Proceeding from the results of X-ray studies (Figure 4, Table 4), it was established that the powder phase composition consists of α-Ti with the following FCC lattice parameters: a = 2.95 Å; c = 4.69 Å.

Analysis of the results of studying the chemical and phase composition, technological properties and particle size distribution of spherical titanium powder Cp-Ti Grade 2, produced during plasma-arc atomization, showed that the above-mentioned powders can be used for 3D printing by different methods. Plasma-arc atomization of current-conducting 1.0 mm wire allows producing sound titanium wires, where the proportion of the fraction of 45–106 μm is up to

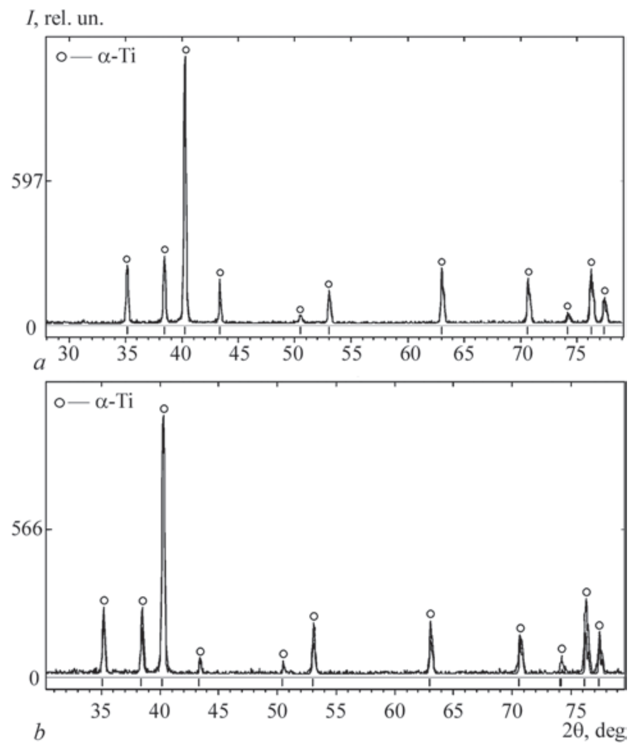

Figure 4. Diffractograms of Cp-Ti Grade 2 titanium powders of the fractions: –106 μm (*a*), +106; –160 μm (*b*)

70 wt.%, which it is rational to use predominantly in the electron beam melting (EBM) process; plasma-arc atomization of current-conducting 1.6 mm wire allows producing sound titanium powders, where the proportion of the fraction of 45–140 μm makes up to 80 wt.%, which it is more rational to use for direct laser deposition (LDED) and plasma metal deposition (PMD) processes.

CONCLUSIONS

1. It was determined that during plasma-arc atomization of 1.0 mm Cp-Ti Grade 2 titanium wire the main powder fraction is 140 μm, making up 96 wt.%, of the total powder weight, where the proportion of the finely-dispersed fraction of 63 μm is 59 wt.%. In the case of atomization of 1.6 mm titanium wire, the main

fraction is 200 μm, which is 95 wt.%, and the quantity of finely-dispersed powder fraction of 63 μm does not exceed 38 wt.%. On the whole, the powders are of a regular spherical shape with the coefficient of sphericity close to 0.9, with a small quantity of satellites and irregularly-shape particles (<1 wt.%). Investigations of the chemical and phase composition of the titanium powder showed that the phase composition of the atomized powder, irrespective of the fraction size, consists of α-Ti, and its chemical composition corresponds to ASTM B 348-05 standard for rods and billets from titanium and its alloys.

2. Derived results allow considering the process of plasma-arc atomization of wire materials as an effective technology, which allows producing sound domestic spherical powders, the technological properties of which are at the same level with another industrial method of producing spherical powders for 3D printing: SS-PREP (with more than 30000 rpm speed), and have higher characteristics, compared to gas atomization processes (VIGA, etc.). The particle size distribution of the produced powders determines the good prospects for their application exactly in the processes of electron beam melting (EBM), direct laser deposition (LDED) and plasma metal deposition (PMD). In the SLM process a relatively low yield of the required 45 μm fraction at the level of 15 wt.% is achieved, so further research is required to increase the yield of the above powder fraction during plasma-arc atomization of wire materials.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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