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DETERMINATION OF OPTIMAL MODES FOR THE PRODUCTION OF MICRO RANGE METAL POWDERS

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Abstract:
This paper presents the results of working out the modes of obtaining micro range powders according to the developed method. It is based on the navigation on the material of the vortex plasma flows of heterogeneous plasma and synchronized high-frequency impact on the electrode. The formation of powders is derived from individual particles heated and accelerated using a high-temperature gas jet - plasma. It is obtained in a special original plasma generator by blowing the plasma-forming inert gas into an electric arc resulting between two electrodes. The possibility of obtaining powders of various sizes and chemical composition by this method on the developed technology has been established. The design of the executive equipment that implements the studied generation process of the micro-size range is best suited for steel wires. Experimental studies made it possible to determine the optimal modes for wires of various chemical composition (steel wire, with a diameter of 1.2 mm, OK Autrod 347Si with a diameter of 0.8 mm). Range of parameters and modes of actuating equipment: current of 70-80A; Plasma-forming gas pressure 0.7-0.8 atm., Wire feed rate 20-40 mm/s, the frequency of ultrasonic oscillations of 50 MHz and 100 MHz. In these conditions, powders are obtained with a dimension of 50-100 μm.

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1. INTRODUCTION

The need to improve the scientific and technical level at the present stage of development of industrial production is most acutely manifested. To solve this problem, extensive scientific research is required, aiming at the massive introduction of new, progressive materials. Methods for obtaining such materials can be created on the basis of known technologies, such as surfacing and spraying, the improvement of which is carried out in the direction of providing a high gradient of properties along the thickness of the material [1,2], as well as the use of automated process control systems based on mathematical modeling [3].

Great advantages are provided by methods based on the introduction of new, for example, additive technologies, which allow directly, without intermediate stages, to obtain finished products from standardized blanks or prepared raw materials. For the manufacture of metal products, most often are used wire [4] or powders [5] from various metals.

According to forecasts of many authoritative organizations, priorities are works in the field of creating micro and nano range powders. These, among others will contribute to a significant increase in production efficiency in such areas as metallurgy, mechanical engineering, energy, construction, agriculture, medicine, etc. [6-8].

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In recent years, materials science has mainly focused on the development of spherical micro- and nano-range powders. The main advantage of these materials is their use in various fields of industry, since their structure gives improved material properties such as hardness, strength and ductility. This fact suggests that at the moment the issue of improving the existing methods for producing powders, as well as developing new ones, is topical.

Today, there are chemical-metallurgical methods for the production of powders. Method [9] consists in the reduction of nanosized iron hydroxide with hydrogen at high temperatures, and method [10] in the plasma-chemical production of metal oxide powders.

Also known is the method [11] of obtaining a finely dispersed metal powder by spraying an inert gas. This method provides powders with a highly spherical shape.

One of sufficiently new methods [12] is a hybrid method for producing spherical small metal powders by centrifugal spraying of mono-zero-dimensional droplets with a pulsating hole. This method obtained powders with a small size, narrow distribution in size and high sphericity.

In addition to some methods that use crushers for the production of powder, spraying liquid melt is the most common method of producing metal powders. Therefore, you can use various melting technologies. As well as spraying methods (water, gas). Depending on the melting and spraying technology used, the resulting powder may differ in size and form [13].

There are various technologies for the production of metal powders: gas, water spraying, centrifugal spraying, plasma spraying, mechanical grinding and doping, solid phase recovery, electrolytic and various chemical processes. All these methods make it possible to obtain metal powders, different in morphology, shape and size, with sphericity of particles and an uneven surface. When receiving a powder, they are repelled from its physical, chemical, volume properties. Usually high-quality metal powders with excellent granulometric composition and without oxide impurities used for the production, materials of structural and functional purposes [14-22].

In addition to metal powders, researchers confirm that it is possible to obtain composite micropowders of a spherical form of particles by means of plasma-chemical synthesis. Researchers used this method to obtain nanopowders of a W-C-Co system. Next, they used different methods to obtain micropowders with particles of a particular size. Among these methods, they mention spray dehydration with an average particle size equal to 40 microns and plasma treatment with an average spherical particle size of 20 microns [23-27].

For spheroidization of powders, plasma processing is widely used, which allows adapting powders for additive production. In addition, plasma processing reduces the average particle size in the range from 10 to 100 microns [28]. Powders are produced in the process of hydrogenation-dehydrogenation with subsequent plasma spheroidization, this allows to optimize the technological parameters of the process and grinding. Spherical powders of 40-70 μm [29] are obtained. Granulated powders obtained by spray drying were spherified using high-frequency inductively linked plasma. The application of plasma processing led to a decrease in particle size, microstructure sealing and improved density and flowability. Termically treated powders due to their good sphericity, improved density and fluidity are suitable for 3D printing [30].

Today, there are many ways to obtain powders; however, spherical micro-range powders required for additive production are a rather complex production task.

This study proposes hardware-technological conditions for obtaining such powders.

The aim of the work was to determine the optimal modes of obtaining metallic micro range powders according to the developed method, which is based on the effect on the material of the vortex plasma flows of heterogeneous plasma and synchronized high-frequency effects on the electrode. In this case, the formation of powders is derived from individual particles heated and accelerated with a high-temperature gas jet - plasma. It is obtained in a special original plasma generator by blowing the plasma-forming inert gas into an electric arc resulting between two electrodes [31]. Based on this method, a laboratory setup was developed [31], which allows you to control the parameters of the process and, accordingly, the properties of the powders obtained. This technology allows obtaining a wide range of powders of various metals and alloys. One of the advantages of the developed method is that a welding wire is used as a tool for obtaining powders. First, it provides the same chemical composition along the entire length, which makes it possible to obtain powders of the required chemical composition. Secondly, the welding wire is presented in a wide variety, both in the chemical composition and in diameter.

2. MATERIALS AND METHODS

The process of working out the modes was carried out on a developed experimental complex, which includes: a power source for a plasmatron, a plasmatron, a developed nozzle for a plasmatron, an industrial cooler, a cylinder with an inert gas, and a particle catcher. The main parameters of the process of obtaining microrange powders are:

- current strength (A);
- voltage (V);
- wire feed rate (mm/s);
- the consumption of plasma-forming gas (l/min);
- pressure of plasma-forming gas (atm.);
- the rate of expiration of the plasma-forming gas (mm/s);
- type of plasma-forming gas;
- wire diameter (mm);
- chemical composition of the wire (%);
- frequency of ultrasound oscillations (MHz);
- distance from the plasma exit from the plasmatron nozzle to the wire (mm);
- distance from wire to Laval nozzle (mm).

To carry out the approval of modes, a number of laboratory experiments were produced, i.e. optimal modes for wires of different diameters and chemical composition were installed. Development of modes was performed on the following types of wires: steel wire with a diameter of 1.2 mm - steel wire and OK Autrod 347Si with a diameter of 0.8 mm - stainless steel wire, the chemical composition of the wire is presented in Table 1.

Table 1. Chemical composition of electrodes

Wire type	Wire diameter, mm	Chemical elements, %										
		C	Si	Mn	Ni	S	P	Cr	N	Cu	Mo	Nb
Steel wire [32]	1.2	0.05 – 0.11	0.7 – 0.95	1.8 – 2.1	≤ 0.25	≤ 0.025	≤ 0.03	≤ 0.2	≤ 0.01	-	-	-
OK Autrod 347Si [33]	0.8	0.04	0.7	1.7	9.8	-	-	19	-	0.1	0.1	0.6

The main parameters of the powder production modes ranged in the following the current is 40-200 A, the pressure of the plasma-forming gas is 0.6-1 atm., the wire feed rate is 20-100 mm/s and the wire diameter of 0.8 and 1.2 mm.

A coil with a wire of the required diameter and chemical composition was installed on the laboratory setup, the power supply of the plasmatron and the compressor were switched on, the required current strength was set on the power supply of the plasmatron. Next, a cylinder with an inert gas was opened, on the reducer of which the required pressure of the plasma-forming gas was set, and then the cooler of the plasma torch nozzle was turned on. On the power supply, the Start button was pressed and the duty arc lights up. Next, the wire feed mechanism was launched, on which the required wire feed rate was set (in experiments there were two types of wire feed: with constant speed and impulse). After the wire reached the plasmatron nozzle the main arc is lights up. If the wire was moving at a constant speed, then the arc burned constantly, if the wire moved impulsively, then the main arc ignited when the wire moved, and extinguished when the wire stopped. As a result of these actions in the particles catcher, powders are condensed.

Working out the modes began with minimal values, i.e. the strength of the current 40 A, the pressure of the plasma-forming gas of 0.6 atm., wire feed speed is 20 mm/s with a gradual increase in values. Variation interval of current was 40 A, pressure of plasma-forming gas – 0.1 atm., wire supply speed was 20 mm/s.

The task of laboratory experiments was the determination of optimal modes to obtain micro range powders from the wire of various chemical compositions, with a diameter of 0.8 and 1.2 mm.

Upon receipt of powders for the purity of the experiment, 125 laboratory experiments were performed on each wire. Total, 250 experiments on the development of modes were carried out.

Operating and adjusting the modes of particle production technologies in the laboratory was carried out according to the method of a full-factor experiment. One of the advantages of mathematical planning of a full-factor experiment is a fairly clear sequential distribution of operations performed (in this case, the process parameters in various combinations). When planning a full-factor experiment, all possible combinations of factors are implemented on all levels selected for research.

Statistical processing of experimental data was carried out by methods of dispersion and regression analyses, using Microsoft Office Excel packets. The determination of the sizes of the resulting powders was carried out by optical metallography on the LEICA DM 750M microscope with image fixation using the LEICA ICC50 W digital camera.

3. RESULTS AND DISCUSSION

The process of working out the modes is one of the main tasks of obtaining micro-range powders, since the technology for obtaining powders directly depends on the modes of their production.

Both the amount of time at which a wire breaks into droplets, and the size of particles we obtain, depend on current and voltage.. The higher the frequency of ultrasonic vibrations (MHz), the smaller the size of microroughnesses; therefore, in the study, a frequency of 50 MHz was used to obtain micro range powders, and 100 MHz was used to obtain powders with a dimension of up to 10 µm.

The flow rate and the rate of expiration of the plasma-forming gas directly depends on its pressure, so for ease of regulation as the main modes, the current, the pressure of the plasma-forming gas, the carrier feed rate, the wire diameter and the chemical composition of the wire are adopted.

An equally important parameter is the distance from the outlet of the plasma torch to the wire (l, mm) (Fig. 1). It should be such that the pilot arc, which lights up on the power source when the "start" button of the laboratory setup is pressed, quickly switches to the main arc, that is, lights up between the anode and the wire.

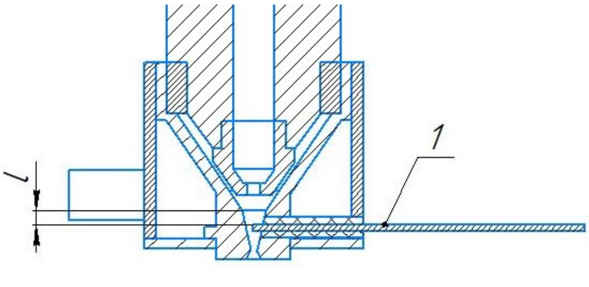


Fig. 1. Plasmatron diagram: *l* – distance from the plasma exit from the plasma torch nozzle to the wire, 1 – wire

Studies have shown [34, 35] that the optimal distance is 1-4 mm. At a distance of less than 1 mm, contact was observed between the plasma torch nozzle and the wire, which is unacceptable. At a distance of more than 4 mm, the main arc does not ignite. This parameter was taken into account in the developed design of the plasmatron nozzle [31], in which this parameter is l - 3 mm.

Optimum value of the distance from the wire to the Laval nozzle (L, mm) (Fig. 2) was found experimentally [31, 34, 35].

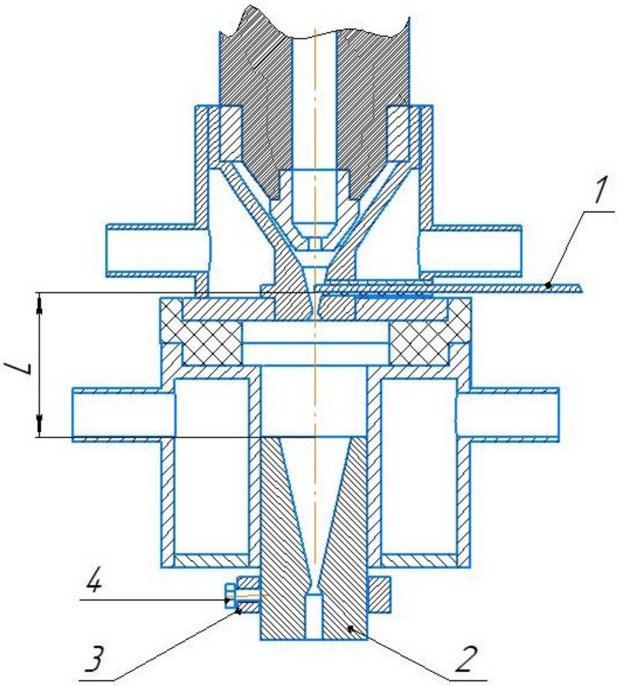


Fig. 2. Plasmatron nozzle diagram: *L* – distance from wire to Laval nozzle, 1 – wire, 2 – Laval nozzle, 3 – fixed ring, 4 – stopper

The dimensions of the resulting particles were chosen as optimization parameters. It was necessary to obtain particles with the smallest size. To determine the optimal distance, laboratory studies were carried out, which were as follows. The Laval nozzle was placed in the lower body of the plasmatron nozzle and fixed in a stationary ring with a stopper at the required distance. The studies were started with a minimum distance of 1 mm from the wire. The process was started, powders were obtained, the process was turned off. Then the distance was gradually increased and the process was repeated until the desired result was achieved. After research, it was found that the optimal distance for obtaining powders with a minimum size of up to 10 microns is 40-45 mm.

A distance of less than 40 mm did not ensure the production of a powder, since the Laval nozzle was heated strongly, which is unacceptable. At a distance of more than 45 mm, the correct formation of the necessary directions of plasma flows for the formation of microroughnesses on the surface of a liquid metal droplet does not occur. In the manufacture of the plasmatron nozzle, the parameter of the distance from the wire to the Laval nozzle (L = 45 mm) was taken into account.

Another of the main parameters is the chemical composition of the wire. To obtain powders that are cleaner in chemical composition, it is necessary to create an inert environment; therefore, argon and helium were used as a gas. The size of the powder depends on the modes and diameter of the wire, the chemical composition of the powder depends on the chemical composition of the wire.

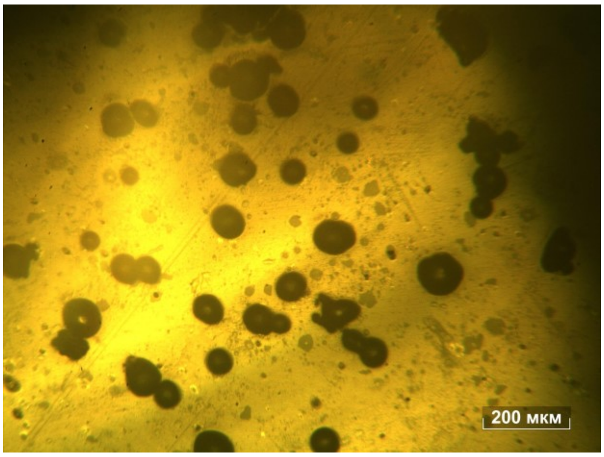
By processing the statistical data of comparative laboratory studies, we obtained the numerical values of the parameters of the micro range droplet generation process (current strength, plasma-forming gas pressure, wire feed rate) (Table 2).

Table 2. Powder production modes

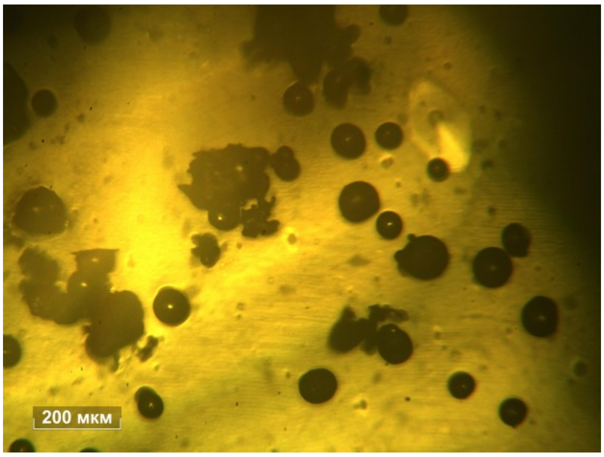
Wire	Current strength, A	Plasma-forming gas pressure, atm.	Wire feed rate, mm/s
Steel wire, diameter 1.2 mm	80±10	0.8±0.1	20±5
OK Autrod 347Si, diameter 0.8 mm	80±10	0.7±0.1	20±5

Powders of various sizes and shapes from the above wires were obtained on the obtained modes. Powders obtained from steel wires have a regular shape close to a sphere and an average size of 50-100 microns (Fig. 3).

Also, in the process of experimental studies, powders were obtained in which the particle has an irregular shape, such powders can be used only after the spheroidization process. The irregular shape of the powder particles is justified by the fact that all metals have different physical, mechanical properties and melting points; therefore, it is impossible to take into account all the features and develop an optimal design of the plasma torch nozzle to obtain powders of the correct shape from all metals. The developed design of the nozzle of the plasmatron [20] is the most optimal for metals with low fluidity (wire made of low-carbon steel, high-alloy austenitic steel).



(a)



(b)

Fig. 3. Optical microscopy of steel particles: a particle of steel wire (a), a particle of stainless steel wire (b)

The design and geometrical parameters of the developed nozzle of the plasmatron allows the formation of the necessary directions of plasma flows for these materials for the formation of microroughnesses on the surface of a liquid metal droplet. The powders obtained from steel wires have the correct shape and size of 50-100 microns.

In order to obtain powder particles of the correct shape from metals with increased fluidity (aluminum, titanium, copper), it is necessary to change the following parameters: angle α, diameter of the inlet and outlet of the gas channel in which the plasma is formed [20], that is, in the nozzle plasmatron to create the necessary directions of plasma flows for the formation of microroughnesses on the surface of a liquid metal droplet. To obtain powders up to 10 microns, it is necessary to place a Laval nozzle in the lower housing of the plasma torch.

4. CONCLUSION

Based on the conducted research, the following conclusions were drawn:

1. Experimentally, the optimal distance from the plasma outlet from the plasma torch nozzle to the wire (*l*, mm) has been established; it is equal to 1-4 mm. In the manufacture of the plasmatron, the distance *l* is taken to be 3 mm. This distance allows an instant transition of the pilot arc to the main one;

2. Experimentally, the optimal distance from the wire to the Laval nozzle (*L*, mm) has been established; it is equal to 40–45 mm. In the manufacture of the plasmatron, the distance *L* is assumed to be 45 mm. This distance makes it possible to form the necessary directions of plasma flows for the formation of microroughnesses on the surface of a liquid metal droplet and to obtain powder particles up to 10 µm in size;

3. The modes of obtaining powders in laboratory conditions have been worked out. Laboratory experimental studies made it possible to determine the optimal modes for wires of various chemical compositions (steel wire, 1.2 mm in diameter, OKAutrod 347Si, 0.8 mm in diameter). The range of parameters and modes of the executive equipment: Current 70-80A; plasma-forming gas pressure 0.7–0.8 atm., wire feed speed 20–40 mm/s, ultrasonic vibration frequency 50 MHz. Powders of 50-100 µm were obtained in these modes;

4. The possibility of obtaining powders of various sizes and chemical composition by this method according to the developed technology has been established. The design of the executive equipment, which implements the investigated process of generation of drops of the microsize range, is most optimal for steel wires. The manufactured nozzle of the plasmatron forms the necessary directions of plasma flows for the formation of a drop of liquid metal from the steel wire of microroughness on the surface. In order to obtain powders of the correct shape from other metals, it is necessary to correct the design features of the plasma torch nozzle and the dimensions of the gas channel in which the plasma is formed. These parameters need to be adjusted depending on the physical, mechanical properties and chemical composition of metals;

5. The resulting powders can be used in three directions: in plasma spraying - powder particles should have the shape of a sphere and a size of 100-300 µm, in additive manufacturing - powder particles should have the shape of a sphere and a size of 20-30 µm, in welding production (modification of the deposited metal) - powder particles should have the shape of a sphere and not more than 10 µm.

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