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# Effect of process parameters on nitriding rate in obtaining powder metal by plasma centrifugal atomization

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***Abstract***. The improved performance properties of metals are ensured by introducing into them certain set and amount of alloying elements. Nitrogen, which is an area of growing interest, is one such element. Publications show that nitriding with gaseous nitrogen is also used for plasma-arc remelting. They provide data on metal alloying with nitrogen at the granules and powders production stage. This paper studies the process of nitriding in obtaining metal microgranules from EP741NP alloy by means of plasma centrifugal atomization. Metal powders are obtained by melting the end face of a rotating workpiece with a stream of ionized gas (gas mixture). The technology allows for nitrogen-alloyed fine metal powders of multicomponent alloys of spherical shape with a minimum number of satellites, which do not differ in size or chemical composition, to be obtained. The study of the nitriding rate is of great interest, especially in production of powder metal. One parameter which affects the degree of metal saturation with nitrogen is the residence time of the liquid melt under the nitrogen-containing plasma, and the crystallization time of a metal droplet. This paper presents a methodology which allows quantification of the role of these parameters on the absorption of nitrogen by the metal in obtaining powder. The kinetic parameters of the nitriding process are influenced by the interface area of two metal – gas phases. In the case of obtaining powder, this parameter depends on the size of the powder particle. In this regard, this paper presents a calculation method which allows the average fractional composition of metal powders to be estimated depending on a number of process factors. The values obtained are compared with the data of semi-industrial melting. It is demonstrated that the fractional composition of microgranules depends on the rotation speed and diameter of the workpiece to be remelted, as well as the alloy density and the surface tension force. It has been established that by increasing the rotation speed of the consumable electrode it is possible to achieve a decrease in the dispersiveness of metal powders.

***Keywords:*** plasma centrifugal atomization, nitrogen in alloys, metal powder, plasma, nitriding

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*Original article*

# Исследование влияния технологических параметров на скорость азотирования при получении металлических порошков методом плазменного центробежного распыления

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***Аннотация***. Повышение эксплуатационных свойств металла обеспечивается введением в него определенного набора и количества легирующих элементов. К таким элементам относится и азот, интерес к которому постоянно растет. В публикациях отмечено, что азотирование газообразным азотом используется, в том числе, при плазменно-дуговом переплаве, приводятся данные легирования металла азотом на стадии получения гранул и порошков. В данной работе исследован процесс азотирования при получении металлических микрогранул из сплава марки ЭП741НП методом плазменного центробежного распыления. Металлические порошки получают путем оплавления торца вращающейся заготовки потоком ионизированного газа (смеси газов). Технология позволяет получать легированные азотом мелкодисперсные металлические порошки многокомпонентных сплавов сферической формы с минимальным количеством сателлитов, не отличающихся по размеру и химическому составу. Исследование скорости азотирования представляет большой интерес, особенно при получении порошкового металла. Одними из параметров, влияющих на степень насыщения металла азотом, являются время нахождения жидкого расплава под азотсодержащей плазмой и время кристаллизации металлической капли. В работе приведена методика, позволяющая дать количественную оценку роли данных параметров на поглощение азота металлом при получении порошка. Известно, что на кинетические параметры процесса азотирования определяющее влияние оказывает площадь контакта двух фаз металл – газ. В случае получения порошка, этот параметр зависит от размера порошинки. В связи с этим, в работе приведена методика расчета, позволяющая оценить средний фракционный состав металлопорошков в зависимости от ряда технологических факторов. Проведено сравнение полученных значений с данными полупромышленных плавок. Показано, что фракционный состав микрогранул зависит от скорости вращения и диаметра переплавляемой заготовки, плотности сплава и силе поверхностного натяжения. Установлено, что при увеличении частоты вращения расходуемого электрода можно добиться уменьшения величины дисперсности металлических порошков.

***Ключевые слова:*** плазменное центробежное распыление, азот в сплавах, металлический порошок, плазма, азотирование

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## Introduction

At the present time metal powders are actively used as the main resource for additive technologies [1, 2], and products made of them are widely used in a variety of fields: electrical power engineering, aircraft construction, automotive industry, health care, etc. [3]. The service properties of steel are largely determined by the presence therein of a particular set and amount of alloying elements, one of which is nitrogen. The influence of nitrogen as an alloying element on service properties has been proven by many papers. This also applies to powder metal, and thus obtaining nitrogen-alloyed powder metal at the stage of its production already is highly relevant.

Studies [4, 5] estimate a number of thermodynamic parameters which influence the powder nitriding process during plasma atomization of a rotating workpiece. The nitrogen content in the obtained powders has been noted as being quite far from thermodynamically justified concentrations. The paper focuses on the kinetics of this process. It provides calculated indices for the size of droplets – future powder particles, the contact time of liquid melt with plasma containing nitrogen and a number of other parameters.

The main factors of rapid development of layer-by-layer spraying processes are: the flexibility of the process; the possibility of manufacturing products of different geometric shape; a wide range of metallic materials; homogeneity of the chemical composition and product microstructure. Due to the high demand for the products manufactured by additive manufacturing methods, there are special requirements for manufacturing of metal powders aimed at improving the service properties of the end product. Powder metallurgy products are characterized by the best physical and mechanical properties compared to cast ones [6].

The most well-known and widespread methods of manufacturing metal powders are [7, 8]:

– atomization of a metal jet by a gas stream [9, 10];

– atomization of a metal jet by a water stream [11, 12];

– plasma centrifugal atomization [13].

As practice and the number of works show, the most promising method for obtaining metal granules is plasma centrifugal atomization of a workpiece. This method has several advantages. In particular the applied mechanism of liquid droplet formation and its subsequent crystallization in inert gas atmosphere create conditions for the formation of a dense structure with a minimum number of satellites. Furthermore, the high rate of crystallization makes it impossible for the liquid melt to contact other materials, e.g. lining. In this case, nitrogen mixed with argon can also be used as plasma-forming gas. This method is of interest both for the production of pure metals and alloys in powder form and for nitrogen alloying of metal powders [14 – 18].

The authors of work [4] presented data on semi-industrial melting with the production of nitrogen-containing metal powders of the EP741NP alloy by plasma centrifugal atomization. Granule production by this method involves melting the end face of a rotating workpiece to be melted by plasma which is a mixture of plasma-forming gases. The plasma melts the metal workpiece, and centrifugal forces move the liquid metal from the central axis of the workpiece towards the periphery, forming a crown. Overcoming the surface tension forces, metal droplets are torn from the workpiece, becoming spherical in flight and crystallizing. The variation of process parameters, such as rotation speed of the consumable electrode, workpiece diameter, and plasma unit power, may significantly influence the grain-size distribution of metal powders and the nitriding rate. For example, when obtaining metal powders of Ti-6Al-4V, 316-steel, Co-29Cr-6Mo alloys by plasma centrifugal atomization, the authors of paper [19] note that the fractional composition of metal granules is inversely proportional to the square root of the rotation speed of the consumable electrode. Works [20 – 24] reflect the effect of the rotation speed of the workpiece, the remelting speed and diameter of the electrode to be remelted on the size of the metal power obtained.

The process of metal saturation with nitrogen in the plasma centrifugal atomization unit depends on kinetic and thermodynamic parameters. Due to the high rotation speed of the workpiece being consumed, the residence time of the liquid melt under the nitrogen-containing plasma arc is limited, when compared with conventional plasma-arc remelting, and the microgranules obtained have a high rate of crystallization. In order to assess nitrogen solubility, it is necessary to consider a number of process parameters affecting the size of metal powders, the residence time of the liquid melt under the plasma column, and the granules crystallization time [5].

In order to analyze and study the above parameters and dependences, the authors of this work used the information given earlier in work [4].

When the plasma arc transmits heat to the workpiece, the process of metal melting begins, and some volume of liquid metal is formed at the end face of the electrode. The liquid metal formed under the influence of centrifugal forces moves from the central axis to the peripheral part of the rotating electrode. At the moment when the centrifugal forces exceed the surface tension forces, the droplet detaches. At the moment of detachment a thin “bridge” is formed between the droplet and the workpiece. It should be noted that in addition to the above forces the metal droplet is also affected by other forces, such as gravity forces, gravitational forces, the plasma arc pressure force and other forces. However, the influence of these physical parameters on the liquid metal retention on the workpiece or droplet detachment from it is very small [25].

## Research materials and methods

Nitrogen-containing metal granules were produced at an industrial plasma centrifugal atomization unit equipped with a consumable workpiece rotating mechanism, an atomization chamber, a heat source – plasma torch, a hopper for granules and a water cooling system.

The model alloy, for which nitrided metal powder production was studied, is high-temperature nickel alloy EP741NP as per GOST 52802-2007.

A remelted electrode obtained in a vacuum induction furnace (VIF), weighing about 20,000 g, was placed and fixed on a plasma centrifugal atomization unit. Then, the furnace body was closed and pressure was evacuated to $10^{-3}$ mmHg, after which the furnace chamber was filled with inert gas, argon. The pressure of the working gas mixture (argon, nitrogen, helium) in the plasma torch was 1.2 atm. The operating current of the plasma torch was 1.05 kA. The plasma torch voltage was 90 V, and the clearance between the plasma torch and the workpiece was 30 – 40 mm. The rotation speed of the workpiece for the first series of experiments was set to 15,000 rpm, and for the second series to 20,000 rpm. The workpiece diameter was 75 mm, length 670 mm, remelting time about 20 min. The nitrogen content in the plasma-forming gas mixture varied and was 15 and 20 %.

## Estimation of the residence time of liquid melt under plasma arc

It appears to be very difficult to experimentally determine the residence time of liquid metal under plasma in the process of heating, melting and atomization of electrode, as well as crystallization. In particular, in order to establish the residence time of liquid metal under the plasma flow and the crystallization time of the liquid granule, the most suitable methods are those of mathematical modeling, making a number of the following assumptions:

– the anode spot is located strictly in the center of the end face of the consumable electrode;

– the workpiece is heated and melted by the heat emitted by the plasma arc;

– metal saturation with nitrogen occurs only under the plasma column;

– due to the high crystallization rate of metal powder and melting conditions in the inert gas and nitrogen mixture atmosphere, nitrogen desorption does not occur.

It is possible to estimate the residence time of liquid metal from the moment of melting to droplet detachment from the end face of the workpiece on the basis of the mass melting rate of the unit. The resulting time will be taken as the residence time of the metal under plasma. In order to analyze this parameter, let us estimate the mass melting rate, i.e. the mass of molten metal per unit time.

Mass remelting rate:

$$v_{\mathrm{rem}} = \frac{m_{\mathrm{all}}}{t_{\mathrm{rem}}}, \tag{1}$$

where $m_{\mathrm{all}}$ is the mass of the workpiece to be remelted, g; $t_{\mathrm{rem}}$ is the time of remelting of the workpiece, s.

Let us assume that the thickness of the liquid film at the end face of the electrode is equal to the average diameter of the granules of the resulting metal powder. Then it is possible to estimate the instantaneous volume of liquid metal at the end face of the electrode. The area of the end face of the electrode to be remelted is found by the formula:

$$S = \pi r^2. \quad (2)$$

The volume of liquid metal at the end face of the electrode:

$$V = Sd, \quad (3)$$

where $S$ is the area of the end face of the electrode to be remelted, m$^2$; $d$ is the diameter of the powder particle formed, m.

Then the residence time of liquid metal under plasma, taking into account the remelting rate, will be:

$$\tau = \frac{m_{1.\,Me}}{v_{\text{rem}}}, \quad (4)$$

where $m_{1.\,Me}$ is the mass of liquid metal at the end face of electrode, g.

Using the above method, we estimated the residence time of liquid metal under the nitrogen-containing plasma. The results are presented below.

| Parameter | Value |
|---|---|
| Mass rate of remelting, $v$, g/cm | 16.898 |
| Electrode end face area, $S$, m$^2$ | $4.415 \cdot 10^{-3}$ |
| Volume of metal at the electrode end face, $V$, m$^3$ | $6.62 \cdot 10^{-7}$ |
| Mass of liquid metal at the end face, $m_{1.\,Me}$, g | 5.53 |
| Residence time of liquid metal under plasma, τ, s | 0.327 |

If we assume that the limiting stage is the convective diffusion of nitrogen atoms in the melt, the nitriding rate equation may be written as follows:

$$\frac{d[\text{N}]}{d[\tau]} = \alpha\left([\text{N}]_\text{p} - [\text{N}]\right). \quad (5)$$

By integrating the above equation, we obtain:

$$\ln \frac{[\text{N}]_\text{p} - [\text{N}]_0}{[\text{N}]_\text{p} - [\text{N}]} = \alpha t, \quad (6)$$

where $\alpha = \frac{\alpha' S}{V}$ is the mass transfer rate constant, cm/s; $\alpha'$ is a semi-empirical parameter determining the rate of mass transfer; $S$ is the area of the interphase surface, m$^2$; $V$ is the volume of the metal droplet, m$^3$; $[\text{N}]_\text{p}$ is nitrogen concentration in the surface layer of the metal – gas interface, close to equilibrium with the gas phase, %; [N] is nitrogen concentration in the metal volume at the moment of time τ, %; $[\text{N}]_0$ is the initial concentration of nitrogen in the metal volume, %; τ is the time of metal saturation with nitrogen, s.

Using the data of semi-industrial melting and calculated data obtained by the above method, we estimated the rate of metal nitriding in the process of atomizing the consumable electrode with nitrogen-containing plasma.

Fig. 1 shows the dependence of the nitriding rate on the partial pressure of nitrogen in the plasma-forming gas. It shows that an increase in the nitrogen pressure contributes to an increase in the nitriding rate.

However, the graph is not increasing constantly, and once a certain nitrogen concentration in the melt is reached, the nitriding rate ceases to depend on the nitrogen content. It should be noted that the calculated data is higher than the experimental data. This difference can be explained by the fact that a number of assumptions were made, in order to develop a mathematical model for estimating the residence time of metal under plasma and the rate of crystallization.

## Methodology of determining crystallization time of liquid droplet

The linear velocity of the melt flow can be determined by means of the following equation

$$V = \omega R, \quad (7)$$

where $\omega = 2\pi n$ is the angular rotation speed of the workpiece, rpm; $n$ is the rotation speed of the workpiece, rpm; $R$ is the radius of the workpiece, m.

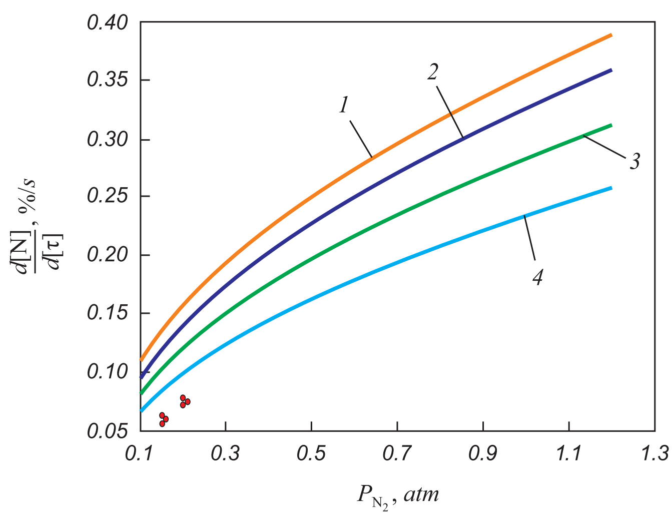


Fig. 1. Dependence of nitriding rate on partial pressure of nitrogen in the plasma-forming gas.
Points are experimental data, and lines are calculated data at different values of α′:
*1* – $5.2 \cdot 10^{-5}$; *2* – $4.9 \cdot 10^{-5}$; *3* – $4.3 \cdot 10^{-5}$; *4* – $3.5 \cdot 10^{-5}$

Рис. 1. Зависимость скорости азотирования от парциального давления азота в плазмообразующем газе.
Точки – экспериментальные данные, линии – расчетные данные при различных значениях α′:
*1* – 5,2·10$^{-5}$; *2* – 4,9·10$^{-5}$; *3* – 4,3·10$^{-5}$; *4* – 3,5·10$^{-5}$

Let us calculate the volume of a droplet:

$$V' = \frac{4}{3}\pi r^3, \quad (8)$$

where $r$ is the droplet radius, m.

Let us find the mass of a liquid droplet:

$$m = V'\rho, \quad (9)$$

where ρ is the alloy density, kg/m$^3$.

The residence time of a liquid droplet from the moment of detachment up to complete crystallization can be found from the equation

$$\tau = \frac{C_\text{m} m_\text{d}}{\alpha S_\text{d}} \ln \frac{T_\text{n} - T_0}{T_\text{melt} - T_0}, \quad (10)$$

where $C_\text{m}$ is the specific heat capacity of the melt. We take it as equal to specific heat capacity of nickel (500 J/(kg·K)); $S_\text{d} = \pi d^2$ is the specific surface area of a liquid metal droplet, m$^2$; $T_\text{n}$ is the droplet temperature at the moment of departure from the electrode. We take it as equal to the alloy melting temperature +200 degrees of superheat (1860 K); $T_0$ is the temperature of a completely crystallized droplet, taken as equal to 300 K; $T_\text{melt}$ is the alloy melting temperature (1660 K); α is the heat transfer coefficient between the droplet and the ambient atmosphere, W/(m$^2$·K);

The heat transfer coefficient can be found from forced convective heat transfer equation [26]

$$\alpha = \frac{\text{Nu}\lambda_e}{d_\text{d}}, \quad (11)$$

where $d_\text{d}$ is the droplet diameter, m; $\lambda_e$ – is the coefficient of heat transfer of the gas medium, W/(m·K)). For argon, according to [27], we find $\lambda_e$ from equation:

$$\lambda_e = (4{,}923 + 0{,}0465T - 8{,}028\cdot 10^{-6}T^2)\cdot 10^{-3}; \quad (12)$$

Nu is the Nusselt criterion

$$\text{Nu} = 0{,}62\sqrt{\text{Re}}; \quad (13)$$

Re is the Reynolds number

$$\text{Re} = \frac{Vd_\text{к}}{\nu}; \quad (14)$$

ν is the coefficient of kinematic viscosity of argon, cm$^2$/s [27].

Be estimated the time of crystallization of the metal powder particle by substituting all coefficients determined by equations (11) – (14) in equation (10). The calculation results are given below.

| Parameter | Value |
|---|---|
| Alloy density, ρ, kg/m$^3$ | 8350 |
| Melt flow rate, $V$, m/s | 58.875 |
| Droplet volume, $V'$, m$^3$ | $2.679\cdot 10^{-13}$ |
| Droplet mass, $m$, kg | $2.237\cdot 10^{-9}$ |
| Droplet surface area, $S_\text{d}$, m$^2$ | $2.0096\cdot 10^{-8}$ |
| Heat transfer coefficient, α, W/(m$^2$·K) | 2818.954 |
| Nusselt criterion, Nu | 10.637 |
| Reynolds number, Re | 294.375 |
| Crystallization time, τ, s | $2.696\cdot 10^{-3}$ |

## Analysis of metal powder grain-size distribution

It is possible to determine the size of granules by examining the influence of the main process parameters on the mechanics of droplet formation. The main contribution to the detachment of liquid particles from the crown is made by centripetal acceleration force. This depends on the rotation speed and diameter of the workpiece to be consumed. The counteracting force is the surface tension force, the value of which depends on the density and surface tension coefficient of the alloy in the liquid state. On this basis and according to the data given in [22, 25, 28], we can use the formula that allows us to calculate the centrifugal force:

$$F_\text{c} = \frac{\pi d^3 \rho \omega^2 D}{12}, \quad (15)$$

where $d$ is the diameter of the liquid droplet, m; $D$ is the diameter of the consumable electrode, m; $\omega = 2\pi n$ is the angular rotation speed of the consumable electrode, rps; $n$ is the rotation speed, rps; η is the detachment coefficient; σ is the surface tension coefficient of the melt, N/m; ρ is the alloy density, kg/m$^3$.

The surface tension n force can be calculated by the formula

$$F_\text{s.t.} = \sigma\pi d_1, \quad (16)$$

where $d_1$ is the diameter of the visible "bridge" between the workpiece and the droplet of metal at the moment of its detachment, m,

$$d_1 = \eta d, \quad (17)$$

where η is the detachment coefficient; according to [26, 29] we take it as equal to 0.85.

The presumed diameter of the liquid droplet can be estimated based on the equality of the centrifugal force and surface tension:

$$d = \frac{2\sqrt{3\eta}}{\omega}\sqrt{\frac{\sigma}{\rho D}}. \quad (18)$$

When the metal droplet detaches, it can have an arbitrary shape. However, due to the high rate of rotation of the workpiece, the metal droplet is globularized and crystallized in flight under the effect of surface tension forces.

Using the experimental data on the rotation speed, mass and geometric dimensions of the electrode to be remelted, we estimated the average size of metal powders formed using the above method. The results are shown in Fig. 2.

The dispersiveness of metal powders obtained is determined by the following main parameters:

– centrifugal force determined by the rotation frequency and diameter of the electrode;

– surface tension force which depends on the surface tension coefficient of the alloy and temperature.

The results of calculations agree conditionally with data obtained during semi-industrial tests at the plasma centrifugal atomization unit. Any difference in the data is due to the absence of all forces affecting the droplet size in the calculations, as well as the impossibility of estimating all physical parameters used in the calculations accurately. However, calculated and experimental data show that while maintaining the diameter of the workpiece to be consumed, the alloy grade and, consequently, the density, viscosity and the surface tension coefficient, the rotation speed of the electrode remains the key parameter.

According to equations (5) and (6), one parameter which affects the nitriding process rate is the mass transfer rate constant. The value of the mass transfer rate constant is influenced by the surface area of the powder particle, i.e. the interface area between the liquid metal and the gas phase, and the volume of the microgranule formed. These two parameters depend on the diameter of the microgranule formed. As shown above, the powder diameter can be affected by changing the rotation speed of the workpiece. Therefore, the rotation speed of the workpiece influences the final nitrogen concentration in the powder particle.

Figure 3 shows the calculated dependence of the nitrogen content in the microgranule on the metal – gas interface to microgranule volume ratio. Increasing the rotation speed of the workpiece allows powders of smaller diameter to be obtained. This in turn increases the metal – gas interface area to microgranule volume ratio.

The data in Fig. 3 suggests that by reducing the size of the metal granules, it is possible to achieve an increase in the nitrogen concentration. However, it should be noted that a constant increase in the rotation speed of the workpiece, thereby obtaining more finely dispersed granules, will make the nitriding process impossible due to the small surface area of the metal – gas interface and a high rate of crystallization.

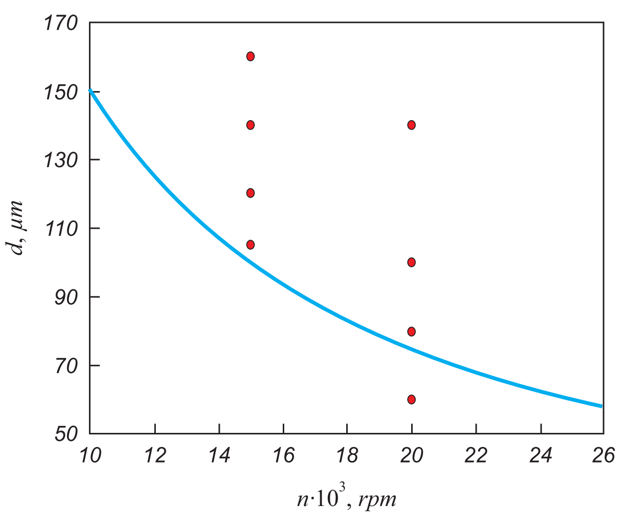


Fig. 2. Dependence of the granules diameter on the workpiece rotation speed. Points are experimental data, and the line is calculated data

Рис. 2. Зависимость диаметра гранул от скорости вращения заготовки. Точки – экспериментальные данные, линия – расчетные данные

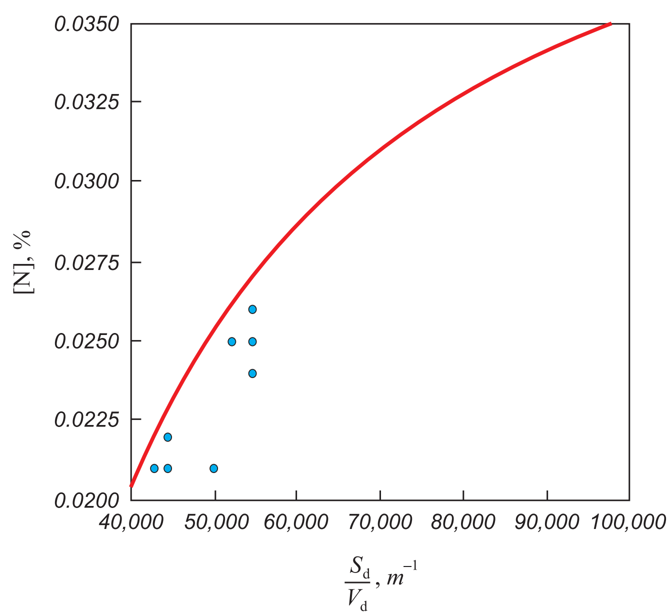


Fig. 3. Dependence of nitrogen content on the metal – gas interface to the microgranule volume ratio. Points are experimental data, and the line is calculated data

Рис. 3. Зависимость содержания азота от отношения поверхности взаимодействия металл – газ к объему микрогранулы. Точки – экспериментальные данные, линия – расчетные данные

## Conclusion

In this paper we studied the effect of a number of kinetic parameters on the process of metal nitriding in the production of metal powders by plasma centrifugal atomization. In particular, we examined the contact time of the nitrogen-containing plasma with the liquid phase, the crystallization time of microgranules formed, the rotation speed of the workpiece to be remelted, and the size of powder particles – granules obtained.

The nitriding rate of metal powders depends on the partial pressure of nitrogen in the plasma-forming gas. For example, at a partial pressure of nitrogen equal to 0.15 atm, the nitriding rate may be 0.08 – 0.14 %/s depending on α′ (adopted parameter determining the mass transfer rate). The above dependence shows that an increase in the partial pressure in the plasma-forming gas contributes to an increase in the nitriding rate. The quantitative dependence between the process parameters and indices that characterize the process of metal saturation with nitrogen has been demonstrated.

The calculation procedure was presented, allowing for prediction of the average size of metal powders obtained by plasma centrifugal atomization, depending on a number of process parameters of the unit. It was shown that in the plasma centrifugal atomization unit, it is possible to adjust the size of granules by changing the rotation speed of the electrode to be remelted. The data calculated in the work shows that by increasing the rotation speed from 15,000 to 20,000 rpm, the average diameter of microgranules can be reduced by 25 µm. The validity of these dependences established mathematically has been confirmed by the experimental data.

The dependencies thereby obtained support metal nitriding conditions at the stage of powder production by plasma centrifugal atomization, also by varying the partial pressure of nitrogen in the plasma-forming gas mixture, the atomization rate and the related fractional composition of granules.

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***O. A. Komolova*** – scientific guidance, revision of the text, formulation of the conclusions.
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