Structure and properties of iron-chromium-nickel powders obtained via electrodispersing of metal wastes of the alloy Kh25N20 in alcohol

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The paper considers the issues of obtaining iron-chromium-nickel powders via electrodispersing of metal wastes of the alloy Kh25N20 in isopropyl alcohol. The electroerosive processing method is characterized by waste-free, energy-efficient and environmental friendly process. However, this method is rarely used in the industry due to the little knowledge of the structure and properties of the obtained raw materials. The aim of this work was to study the structure and properties of iron-chromium-nickel powders obtained via electrodispersing of metal wastes of the alloy Kh25N20 in isopropyl alcohol. The pieces of the alloy Kh25N20 were preliminarily cut into 2–3 cm pieces at an experimental unit for consequent recycling of metal wastes of this alloy via the method of electroerosive dispersion. These pieces were loaded into a reactor filled with distilled water as a working liquid. The following electrical parameters of the unit were used: voltage at the electrodes 160–220 V; capacity of condensers 40–50 μF; impulse frequency 150–220 Hz. Based on the conducted researches aimed on studying the structure and properties of iron-chromium-nickel powders, which were obtained via electroerosive dispersion of metal wastes of the alloy Kh25N20 in isopropyl alcohol, the following results have been established: the shape of the powder particles is mainly spherical and elliptical; carbon and oxygen are found on the surface of the powder particles, while all other elements (iron, nickel, chromium and manganese) are distributed relatively uniformly; powder particles have sizes from 0.1 up to 100.0 μm with an average volumetric diameter 24.4 μm; powder particles include the phases Ni_3C, Fe_3C, Fe_3Ni_2 and Cr. Use of electroerosive dispersion technology makes it possible to solve the problem of obtaining small batches of spherical iron-chromium-nickel powders of the required fractional composition with minimal energy consumption and minimal damage to the environment. The resulting electroerosive iron-chromium-nickel powders can be effectively used in the production of heat-resistant alloys and coatings.

Key words: metal wastes, Kh25N20 alloy, electroerosive dispersion, alcohol, iron-chromium-nickel powder, properties.
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Introduction

At present time, nickel superalloys (including the alloy Kh25N20) are widely distributed in the aircraft and space industries for manufacture of thermally loaded components for gas turbine engines, such as nozzles, blades, discs etc. [1–3]. This alloy is mainly used for manufacture of elastic sensitive elements operating at the temperature up to 950 °C [4–6].

However, high cost of alloying elements, including the alloy Kh25N20 composition (such as nickel and chromium), is an obstacle for wide application of this alloy [7–9]. Processing of metal wastes of this alloy in powder material and its secondary use is one of the ways to solve this problem [10–12].

The method of electroerosive dispersion (EED) is considered as one of the most prospective but industrially unused technology for grinding of current-conducting metal wastes [13–15]. The grinding process of pieces of metal wastes via electric erosion is based on metal destruction due to local effect of electric discharge impulse energy [16–18]. Processing of metals up to the particles of micro- and nanofractions with preset complex of physical-chemical properties is possible using this method, with possibility of their secondary use, is provided via this method. Electroerosive dispersion allows rising of economical efficiency of recycling of metal wastes owing to small energy consunption of this process.

Additionally, the electroerosive method allows obtaining the powders without use of chemical reactants, what provides significant effect on powder cost and allows avoiding contamination of an operating liquid and the environment by chemical substances [19, 20]. Conduction of this process in a liquid operating medium, which provides the direct effect on composition, structure and properties of dispersion products, is the feature of EED process.

However, there is no complete information about the properties of iron-chromium-nickel powders obtained via electrodispersing of metal wastes of the alloy Kh25N20, what hampers their practical use. To develop the technological recommendations for processing of metal wastes of iron-chromium-nickel alloy Kh25N20 via electrodispersing in fine-dispersed particles for their secondary use, it is required to conduct the complex metallographic researches.

The aim of this work was to study the structure forming process for iron-chromium-nickel powders obtained in the electroerosion conditions for metal wastes of the alloy Kh25N20 in isopropyl alcohol.

Materials and methods of the research

To execute the planned researches, wastes of the alloy Kh25N20 in the form of bar pieces with diameter 6 mm and length 15 mm were chosen. Chemical composition of this alloy is presented in the **Table 1**. The formulated aim was achieved by conducting the experiments for grinding of wastes of the alloy Kh25N20 in the working medium — isopropyl alcohol (according to TU 6-09-402-87 (propanol-2)) via electrodispersing, with following optimization of this process and examination of the powder properties obtained at the optimal procedures.

The grinding nprocess of wastes of the alloy Kh25N20 (**Fig. 1**) was carried out due to impulse voltage of an impulse generator *1* applied to the electrodes *3*, which were manufactured of the same alloy *4*, in isopropyl alcohol *6*. The pieces of metal wastes 4 of the alloy Kh25N20 were melted and liquid metal was thrown out of discharge channel under the effect of dynamic forces. Then this metal solidified snd crystallized rapidly in this liquid with forming spherical and elliptic powder particles and formed a sediment *7*. Working liquid in this case encircled discharge channels with gas bubbles *2* [21].

The following parameters were used during experiments for grinding of metal wastes of the alloy Kh25N20 in isopropyl alcohol for the original patented unit: voltage at the electrodes 160–220 V; capacity of discharge condensers 40–50 μF; electric impulse frequency 150–220 Hz. The cahege mass was 500 g in this case.

Selection of these parameters for electroerosive dispersion procedure is based on the requirement to provide stable spark forming between granules of dispersed metal waste.

Average specific electric energy consumprion consituted about 4.2 kg/kWt·h.

Dried powder was examined via various methods. Microanalysis of particles of the tltctric erosion iron-chromium-nickel powder was carried out using a scanning electron microscope S-3400N of «Hitachi High-Technologies Corporation» (Japan). Granulometric composition of powder particles was studied in the laser analyzer for sizes of particles «Analysette 22 NanoTec» (Germany). Local X-ray spectral microanalysis of powder particles was conducted in the X-ray energy dispersion spectrometer«NORAN», which is built-in a scanning electron microscope S-3400N of «Hitachi High-Technologies Corporation» (Japan). Phase analysis of powder particles was carried out using X-tay diffraction in the diffractometer «Rigaku Ultima IV» (Japan).

Mathematical simulation of grinding of wastes of the alloy Kh25N20 in isopropyl alcohol was conducted via preparing of the complete factorial experiment of 2^3 type, where the following variable indicators were chosen as the indicators having the effect on the optimization parameter (an average size of particles): voltage at the electrodes (X_1), impulse frequency (X_2) and capacity of condensers (X_3). The values of chosen levels for variuable factors are presented in the **Table 2**.

Table 2. Intervals and levels of the variable factors

Level of the variable indicators	Encoded designation	*U*, V	*f*, Hz	*C*, μF
		X_1	X_2	X_3
Basic level	0	150	150	45.5
Varying range	Δx_i	50	50	20
Upper level	+1	200	200	65.5
Lower level	–1	100	100	25.5

Achieving of the optimal value for average size of particles was carried out via the Box-Wilson path-of-steepest-ascent method. Optimization task was concluded in pilot determination of such combination of levels of indicators, when optimal values of a final parameter are achieved.

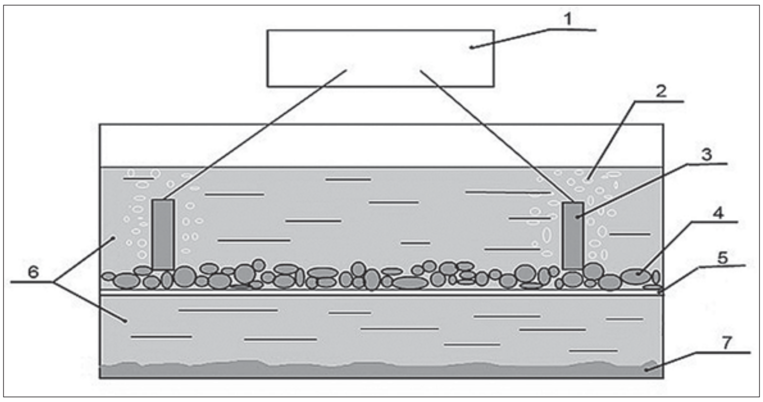

Fig. 1. Technological scheme of electroerosive dispersion of the alloy Kh25N20: *1* – impulse generator; *2* – gas bubbles; *3* – electrodes; *4* – dispersed material; *5* – perforated grid; *6* – working liquid; *7* – powder sediment

Table 1. Chemical composition of the alloy Kh25N20, %

Fe	C	Si	Mn	Ni	S	P	Cr
49.8–59	up to 0.15	up to 1	up to 2	17–20	up to 0.02	up to 0.03	24–27

The results of the research

The results of preliminary investigations of size parameters of powder particles, fabricated via electroerosion ndispersion of metal wastes of the alloy Kh25N20 in isopropyl alcohol, displayed the influence of electric operating parameters of the unit (voltage at the electrodes, impulse frequency and capacity of discharge condensers) on their average size. Optimization of electrodispersing process for metal wastes of the alloy Kh25N20 was required for dispersity stabilization of powder particles. In this case, average size of the particles is considered as an optimization parameter, while voltage at the electrodes, impulse frequency and capacity of discharge condensers are the basic indicators.

According to the conducted calculations, the following regression equation for mathematical description of electrodispersing process for metal wastes of the iron-chromium-nickel alloy Kh25N20 was obtained:

$$\hat{y} = 50.4 + 13.6X_1 + 3.8X_2 + 8.1X_3 - 0.2X_1X_2 + \\ + 0.08X_1X_3 - 0.7X_2X_3 + 0.8X_1X_2X_3 \quad (1)$$

where X_1, X_2, X_3 — encoded values of indicators (voltage at the electrodes, impulse frequency and capacity of discharge condensers respectively) and $\hat{y}$ — optimization indicator (average size of particles, μm).

It can be seen from the equation (1), that maximal effect on the optimization indicator is provided by voltage at the electrodes and capacity of discharge condensers, because these factors have minimal coefficients. The obtained equation was used for calculation of a path-of-steepest-ascent on the surface of response. This ascent was started from the zero points (basic levels) (see **Table 3**).

Table 3. Calculation of a path-of-steepest-ascent

Name of parameters and steps	X_1 (U, V)	X_2 (f, Hz)	X_3 (C, μF)	$\hat{y}$, μm
Basic level	150	150	45.5	–
Coefficient b_i	13.6	3.8	8.1	–
Varying interval ξ_i	50	50	20	–
$b_i \cdot \xi_i$	680	190	162	–
Step Δ_i	34	9.5	8.1	–
Rounded-off step	34	10	8	–
Experiment 1	184	160	53.5	63.6
Experiment 2	200	170	61.5	71.8
Experiment 3	200	180	66.5	74.1
Experiment 4	200	190	65.5	74.7
Experiment 5 (max)	200	200	65.5	75.4

According to the conducted calculations, the ultimate value of optimization indicator $\hat{y}$ (average size of particles) was determined; it made 75.4 μm with capacity of discharge condensers 65.5 μF, voltage at the electrodes 200 V and impulse frequency 200 Hz.

Further examination of structure and properties of iron-chromium-nickel powders was carried out for the particles obtained at the optimal electrodispersing procedures for metal wastes of the iron-chromium-nickel alloy Kh25N20 in isopropyl alcohol.

The results of examination of shape of iron-chromium-nickel powders obtained via electrodispersing of metal wastes of the alloy Kh25N20 in isopropyl alcohol testify that electroerosion particles have spheric and ellyptic shape, the same as agglomerates (**Fig. 2**).

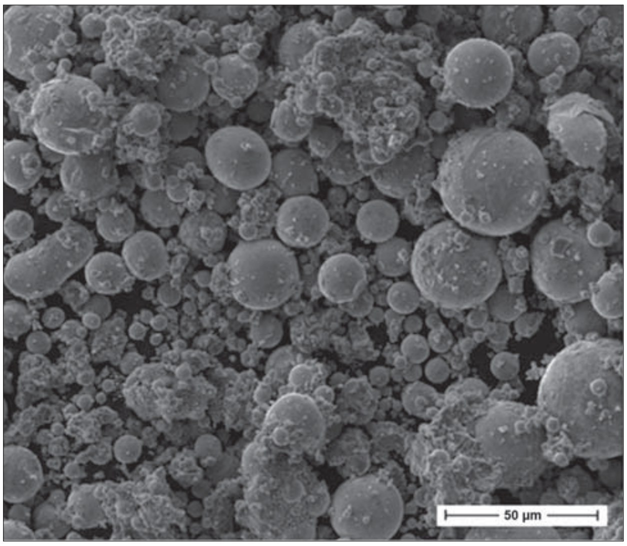

Fig. 2. Morphology of powder particles

It is noted that pieces of metal wastes of the iron-chromium-nickel alloy Kh25N20 were melted in isopropyl alcohol and liquid metal was thrown out from an electric discharge channel and crystallized very quickly in the points of impulse electric discharge. The process of quick crystallization of molten material in isopropyl alcohol promoted acquiring of spherical and elliptical form by the particles [21]. Essential dynamic forces lead to frequent contact with liquid metal, which is thrown out from an electric discharge channel.

When crystalluzation of liquid metal is completed, ther typical footprints of strikes and net surface remained on the particles of forming powder. If this crystallization is not completed, sticking of separate powder particles with forming of agglomerates with inproper shape occurs.

Spheric and ellyptic shape of powder particles , i.e. obtained via grinding of metal wastes of the alloy Kh25N20 in isopropyl alcohol, is considered as a special technological feature of the process of electroerosion dispersion of current-conducting materials in a liquid operating medium.

Analysis of distribution of the particles of iron-chromium-nickel powder, which was obtained using the laser analyzer «Analysette 22 MicroTec», according to their sizes, displayed the size of particles from 0.1 to 100.0 μm with the average volumetric diameter 75.4 μm (**Fig. 3**).

Each point at the integral curve *1* displays, how many percents of powder has size of particles less than the preset one of equal to it. The histogram *2* shows amount of powder with the preset size of particles.

Special features of electrodispersing process for current-conducting materials in a liquid operating medium is also

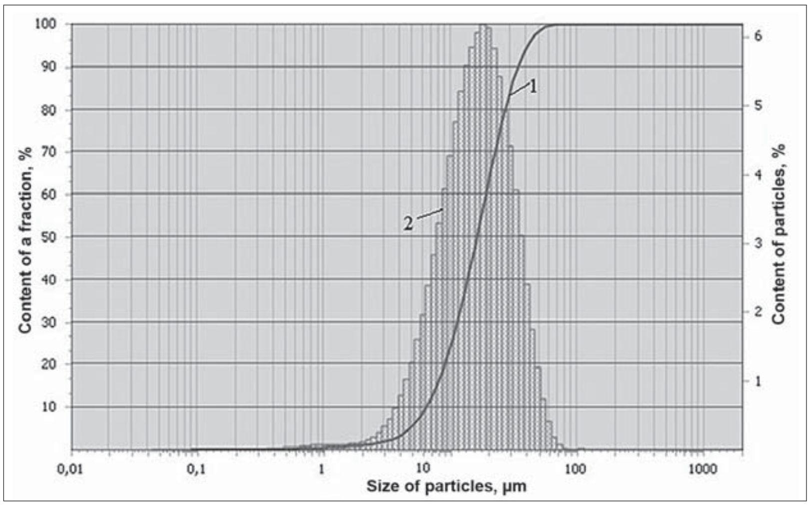

Fig. 3. Distribution of micro-particles according to their sizes: *1* — integral curve; *2* — histogram

connected with origination of powder particles via crystallization of drops of molten metal with forming of a large dispersion fraction and via crystallization of vapours of dispersing metal wastes with forming of a small dispersion fraction.

It was established experimentally, that about 7 % of powder particles have the size from 0.1 to 1.0 μm. It was revealed that fine-dispersed powder fraction is forming during crystallization of a metal vapour phase, while this metal is thrown out from the point of impulse electric discharge. Other 93 % of powder particles have the size from 1.0 to 100 μm. This powder fraction is forming during crystallization of the liquid phase of metal wastes.

The relationship displaying increase of the average size of particles with inrease of an impulse energy (i.e. voltage and capacity) is also established.

Local X-ray spectral microanalysis of powder particles, which was conducted using the X-ray energy dispersion spectrometer "NORAN", built-in a scanning electron microscope S-3400N of "Hitachi High-Technologies Corporation", has shown that carbon and oxygen are revealed on the surface of powder particles obtained from metal wastes of the alloy Kh25N20 of the alloy Kh25N20 via electroerosive dispersion (EED). All other elements, such as Fe, Ni, Cr and Mn are distributed relatively homogeneo suly (**Fig. 4**).

The conducted researches showed that it is possible to obtain iron-chromium-nickel powder with homogeneous distribution of wastes via the method of electroerosive dispersing of wastes of the alloy Kh25N20 in isopropyl alcohol [21].

Phase analysis of composition of powder particles, which was carried out in the X-ray diffractometer "Rigaku Ultima IV", displayed that presence of the basic phases Ni_3C, Fe_3C, Fe_3Ni_2 и Cr (**Fig. 5**).

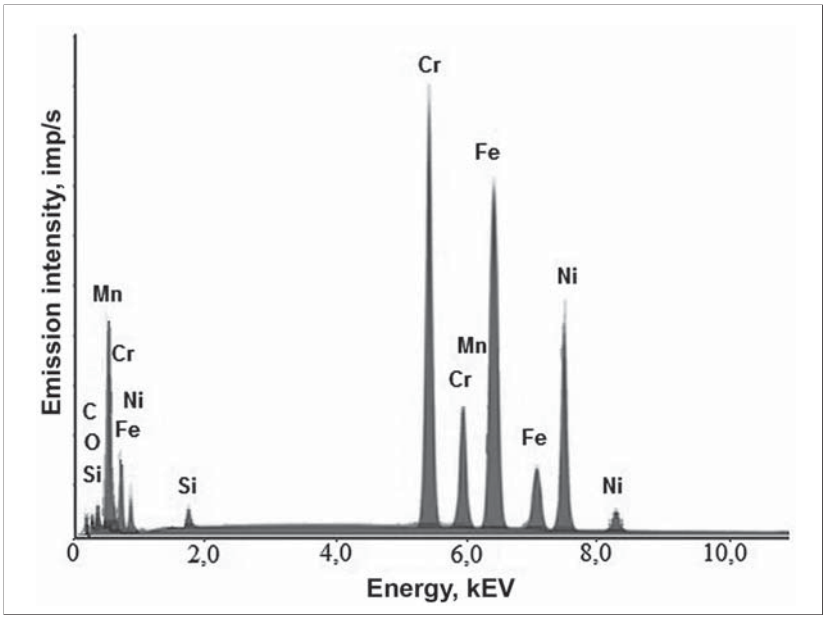

Fig. 4. The results of X-ray spectral microanalysis of the particles of iron-chromium-nickel powder

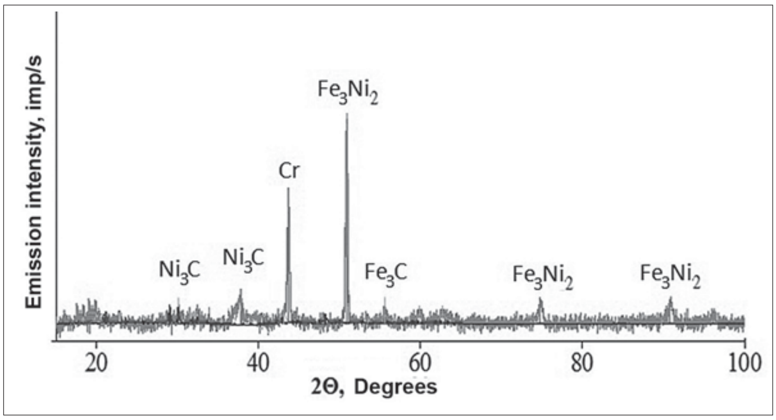

Fig. 5. Diffraction pattern of phase composition of the particles of iron-chromium-nickel powder

It was revealed experimentally that isopropyl alcohol was subjected to pyrolysis in the ptocess of grinding of pieces of metal wastes of iron-chromium-nickel alloy Kh25N20 via electroerosion and participated in structure forming of powder particles, forming various chemical compounds with them [21].

The processes occurring during electrodispersing of metal wastes of iron-chromium-nickel alloy Kh25N20 conducy in intra-electrode space, which is filled with the working fluid — isopropyl alcohol. This liquid provides physical, chemical, washing and mechanical effect on the process, electrodes, granules and nerpsion products of the alloy, what influences on all technological stages. At the stage of electric discharge, decomposition of the working fluid takes place, and its pyrolysis products have chamical interaction with products of electroerosive dispersion, with forming various chemical compounds. Hydrogen moves to the cover of the working fluid during ED process, while carbon interacts with the erosion products (Me′), I.e. $Me \leftrightarrow Me' + C_nH_m = MeC_n + m/2H_2\uparrow$.

Conclusions

1. Based on the conducted researches aimed on studying the structure and properties of iron-chromium-nickel powders, which were obtained via electroerosive dispersion of metal wastes of the alloy Kh25N20 in isopropyl alcohol, the following results have been established: the shape of the powder particles is mainly spherical and elliptical; carbon and oxygen are found on the surface of the powder particles, obtained via EED method from the metal wastes of the alloy Kh25N20, while all other elements (iron, nickel, chromium and manganese) are distributed relatively uniformly; powder particles have sizes from 0.1 up to 100.0 μm with an average volumetric diameter 24.4 μm; powder particles include the phases Ni_3C, Fe_3C, Fe_3Ni_2 и Cr.

2. Use of electroerosive dispersion technology makes it possible to solve the problem of obtaining small batches of spherical iron-chromium-nickel powders of the required fractional composition with minimal energy consumption and minimal damage to the environment.

3. The resulting electroerosive iron-chromium-nickel powders can be effectively used in the production of heat-resistant alloys and coatings.

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