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

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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 distilled water. 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 distilled water. 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 120–140 V; capacity of condensers 62.5–65.0 μF; impulse frequency 100–120 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, the following results have been established: the shape of the powder particles is mainly spherical and elliptical; oxygen is contained on the surface of the powder particles, while all other elements (iron, nickel and chromium) are distributed relatively uniformly; powder particles have sizes from 0.1 up to 100.0 μm with an average volumetric diameter 64.9 μm; powder particles include the phases Fe_3O_4, Fe_3Ni_2 and Cr. Analysis of the results of the conducted researches showed that the physico-chemical properties of the obtained iron-chromium-nickel powder are influenced by the chemical composition of the working fluid, as well as by the modes of electrodispersing. It is possible to obtain powder with various sizes by varying the technological parameters of the unit.
Key words: metal wastes, Kh25N20 alloy, electrodispersing, electroerosive dispersion, iron-chromium-nickel powder, structure, properties.
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Introduction

At present time, nickel superalloys 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]. One of the main problems of using superalloys and heat-resistant alloys (including the alloy Kh25N20) is connected with presence of the expensive components, such as Cr and Ni, in its composition and necessity of its secondary use [4–7]. The alloy Kh25N20 is widely distributed at present time for manufacture of elastic sensitive elements operating at the temperature up to 950 °C [8–11].

Putting into practice the recycling processes for expensive alloys, including the alloy Kh25N20, is one of the main indicators of saving and efficiently using a mineral and raw material potential both in the separate country and in the global scale [12–15].

The method on the base of electroerosion – electroerosive dispersion (ED) is considered as a prospective but incompletely examined recycling method for all current-conducting materials. 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 [16, 17]. Electroerosion processing method is characterized as waste-free, energy efficiency and ecological purity of the process. However, this method is rarely used in the industry due to insufficient knowledge of structure and properties of obtained raw materials.

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.

Recycling of high-alloy alloys, including iron-chromium-nickel alloy Kh25N20, will ensure resource saving, import replacement and providing RF technological sovereignty [1–4].

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 distilled water.

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**. Technological and cheap oxygen-containing liquid – distilled water according to the GOST R 58144-2018 – was applied as working liquid.

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

Obtaining of chromium-nickel powders of metal wastes of the alloy Kh25N20 was carried out in an experimental unit via the method of electroerosive dispersion [16, 17]. Bars of this alloy, which were cut by 2–3 pieces, then were charged in a reactor filled with working liquid (distilled water) (**Fig. 1**). Energy parameters of the unit were varied within the following ranges: voltage at the electrodes 120–140 V; capacity of condensers 62.5–65.0 μF; impulse frequency 100–120 Hz.

When electrodispersing of metal wastes of the alloy Kh25N20, impulse voltage of an impulse generator *1* was applied to the electrodes *2* and *3* and then to pieces of the alloy *6* (pieces of metal wastes of the alloy Kh25N20 are also used as electrodes). Electric breakdown of working liquid *5*, which was located in the space between electrodes, occurred with reaching the definite voltage and finalized in forming of discharge channel *7*. The alloy was melted and evaporated in a discharge point due to high concentration of thermal energy, while working liquid evaporated and encircled a discharge channel by gaseous decomposition products *9* (gas bubble). As a result of essential dynamic forces, which developed in discharge channel and gas bubble, drops of molten material *4* were thrown away in working liquid which encircled electrodes, outside the discharge area and then they solidified in this liquid with forming spherical powder particles of the alloy *4*.

Mathematical simulation of electrodispersing process for metal wastes of the iron-chromium-nickel alloy Kh25N20 was conducted via realizing of the complete factorial experiment of 2^3 type, where the following variable operating parameters of the unit were selected as factors having the influence on the optimization parameter (average size of particles): voltage at the electrodes, impulse frequency and capacity of discharge condensers. The values of selected levels for varying parameters are presented in the **Table 2**.

Table 2. Intervals and levels of varying indicators

Levels of varying indicators	Code designation	U, V	f, Hz	C, μF
		X_1	X_2	X_3
Basic level	0	150	150	45.5
Varying interval	Δ_{xi}	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 for indicators, when maximal or minimal value of a final parameter is achieved.

The tasks for examination of structure and properties of iron-chromium-nickel powders, which were formulated in this research, were solved using up-to-date equipment and complimentary methods of physical material science. So, shape and surface morphology of powder particles were examined in the ion scanning (focused beam) microscope "QUANTA 600 FEG" (Netherlands) with field emission of electrons. Granulometric composition of powder particles were studied in the laser analyzer for sizes of particles "Analysette 22 NanoTec" (Germany). X-ray spectral microanalysis of powder particles was conducted in the energy dis-

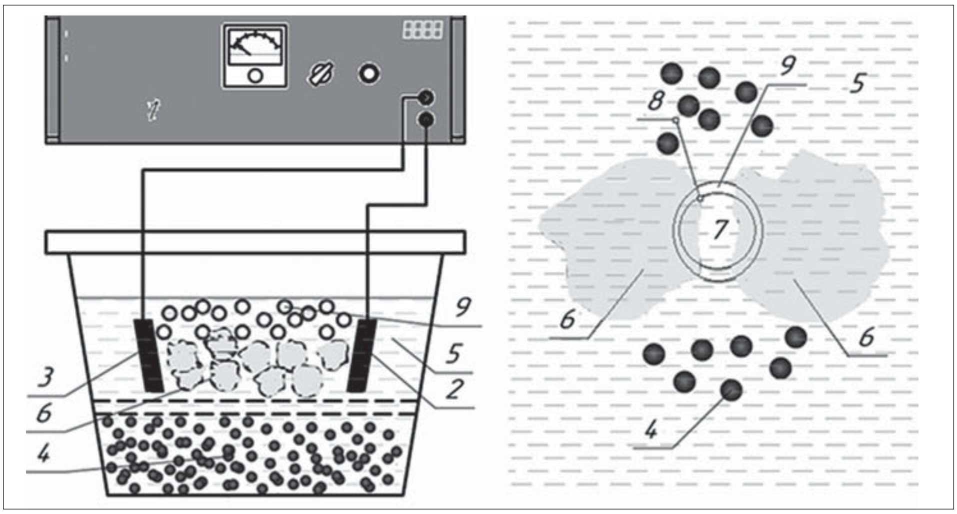

Fig. 1. The scheme of electric dispersion process for metal wastes of the alloy Kh25N20.
1 – impulse voltage of an impulse generator; *2*, *3* – electrodes; *4* – molten metal; *5* – working fluid; *6* – pieces of the alloy; *7* – discharge channel; *8* – discharge point; *9* – gaseous decomposition products (gas bubble)

persion analyzer of X-ray emission of the company "EDAX" (Netherlands), built-in a focused beam electron microscope "QUANTA 200 3D" (Netherlands). Phase analysis of powder particles was carried out in the X-ray diffractometer "Rigaku Ultima IV" (Japan).

The results of the research

The results of preliminary investigations of size parameters of electroerosive charge particles, fabricated of metal wastes of the alloy Kh25N20, displayed the influence of electric operating parameters of the unit (voltage at the electrodes, impulse frequency and capacity of discharge condensers) on charge dispersity. Optimization of electrodispersing process for metal wastes of the alloy Kh25N20 was required for dispersity stabilization. In this case, average size of 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:

$$\hat{y} = 32.9 + 14.3X_1 + 5.56X_2 + 8.83X_3 + 1.58X_1X_2 + \\ +2.21X_1X_3 + 1.31X_2X_3 + 2.68X_1X_2X_3 \quad (1)$$

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 other factors have maximal 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) (**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 bi	14.3	5.56	8.83	–
Varying interval ξi	50	50	20	–
bi · ξi	715	278	176.6	–
Step Δi	35.7	13.9	8.83	–
Rounded-off step	36	14	9	–
Experiment 1	186	164	54.5	50.2
Experiment 2	200	188	63.5	63.2
Experiment 3 (max)	200	200	65.5	64.9

According to the conducted calculations, the ultimate value of optimization indicator $\hat{y}$ (average size of particles) was determined; it made 64.9 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.

The results of examination of shape and morphology of iron-chromium-nickel powders obtained via electrodispersing of metal wastes of the alloy Kh25N20 in distilled water are presented in the **Fig. 2**.

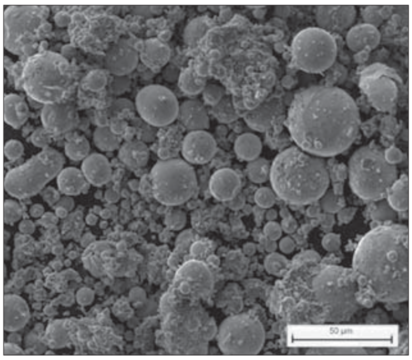

Fig. 2. Morphology of powder particles

It is noted that erosion products (particles of metal wastes) are thrown out in molten form during electrodispersing process from an electric discharge channel into a reactor filled by working fluid, and then they are crystallized very quickly. The process of quick crystallization of molten material in liquid working medium promoted acquiring of spherical and elliptical form by the particles. It was also noted that agglomerates, which were formed as a result of sticking together the particles with close temperature, also are presented in addition to spherical and elliptical particles.

Analysis of distribution of the particles of iron-chromium-nickel powders, which were obtained via electrodispersing of metal wastes of the alloy Kh25N20 in distilled water, according to their sizes is presented in the **Fig. 3**.

Each point at the integral curve (left scale) displays, how many percents of powder has size of particles less than the preset one of equal to it. The histogram (right scale) shows amount of powder with the preset size of particles.

It was noted that iron-chromium-nickel powders, which were obtained via electrodispersing, are characterized by

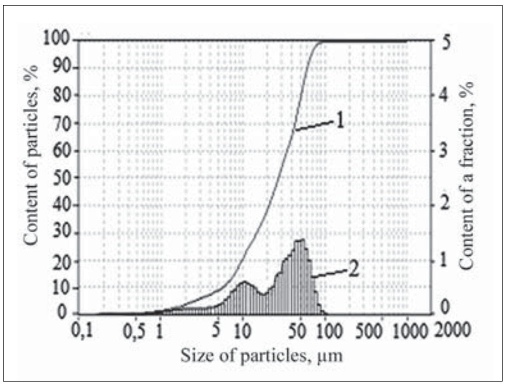

Fig. 3. Distribution of micro-particles according to their sizes: 1 – integral curve; 2 – histogram

rather wide range of distribution of the particles according to their sizes, depending on fabrication parameters (mainly impulse energy). This range varies from several hundreds nanometers to a hundred of microns, i.e. the particles of powder have sizes from 0.1 to 100 μm, with average volumetric diameter 64.9 μm.

It was established experimentally, that we can vary the average size of particles via varying discharge impulse energy via tuning of voltage at the electrodes and capacity of discharge condensers during electrodispersing of metal wastes. It was revealed that fine-dispersed particles of powder are forming during crystallization of vapour phase, while more coarse-dispersed particles – during crystallization of liquid phase.

It was also noted that average size of particles for powder obtained via electrodispersing depends on geometric parameters of a reactor (distance between the electrodes), size of dispersing material and its erosive resistance.

It was established that average size of particles for powder also increases due to increase of an impulse energy (charge-discharge energy for discharge condensers), which is used for partial melting of material; it is valid for other equal conditions, during increase of capacity of discharge condensers or voltage at the electrodes of the reactor.

The processes occurring during electrodispersing (ED) of metal wastes, takes place in the space between electrodes, which is filled by working liquid; this liquid provides the physical, chemical, rinsing and mechanical effect on the process, electrodes, granules and products of alloy erosion, what finalizes in influence of all process stages. Decomposition of working liquid occurs at the stage of electric discharge, and products of its pyrolysis enter in chemical interaction with products of electroerosive dispersing with forming of various chemical compounds. Working liquid cools a working area during electrodispersing. Passing od electric current through a working liquid causes its decomposition with forming of hydrogen oxygen and carbon (depending on fluid nature). Hydrogen appears on the surface of working liquid during ED, while oxygen and carbon react with erosion products (Me′), i.e. $Me \leftrightarrow Me' + H_2O = Me_xO_y + H_2\uparrow$.

X-ray spectral microanalysis of powder particles, which was conducted in the energy dispersion analyzer of X-ray emission of the company "EDAX", built-in a focused beam electron microscope "QUANTA 200 3D", displayed that oxygen is not revealed on the surface of powder particles, which was obtained via electroerosive dispersing (EED) from wastes of the hard alloy Kh25N20; all other elements (Fe, Ni, Cr) are distributed relatively homogeneously (**Fig. 4**).

The conducted researches showed that it is possible to obtain iron-chromium-nickel powder with homogeneous distribution of the alloying elements via the method of electroerosive dispersing of wastes of the alloy Kh25N20 in distilled water. Diffraction pattern of phase composition of the particles of iron-chromium-nickel powder are presented in the **Fig. 5**.

Phase analysis of composition of powder particles, which was carried out in the X-ray diffractometer "Rigaku Ultima IV", displayed that presence of oxygen in a working liquid leads to forming of the phases Fe_3O_4, Fe_3Ni_2 and Cr.

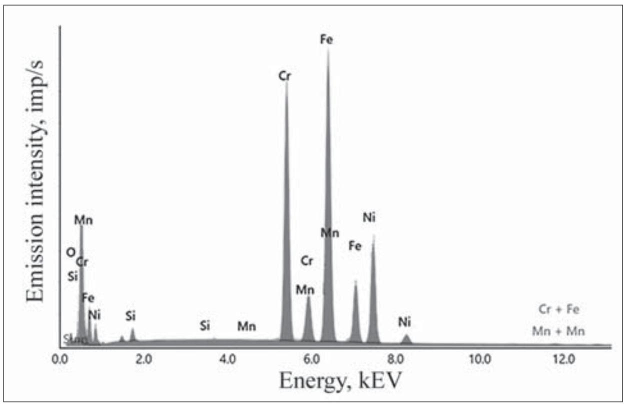

Fig. 4. The results of X-ray spectral microanalysis of the particles of iron-chromium-nickel powder

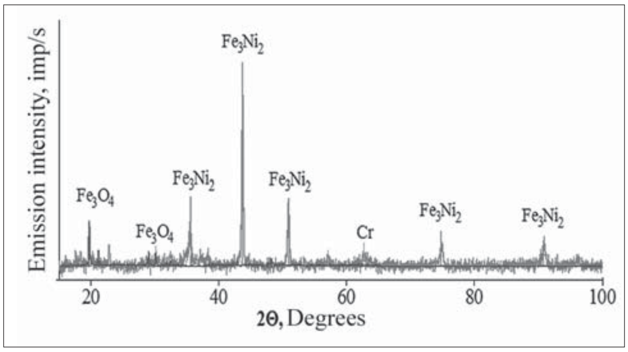

Fig. 5. Diffraction pattern of phase composition of the particles of iron-chromium-nickel powder

Forming of the particles of iron-chromium-nickel powder with spherical shape in the process of electrodispersing of metal wastes of the alloy Kh25N20 is a very important feature of this process, because spherical particles both in the alloys and in the coatings are minimal stress concentrators and provide high mechanical properties (including impact strength) for these materials.

It should be noted that the Ed method starts to compete successfully with other fabrication methods for iron-chromium-nickel powders, including those suitable for additive technologies. Use of metal wastes as initial metal waste materials (which are significantly cheaper than pure components using in conventional technologies), good controllability, low energy intensity and ecological acceptability of the process are considered as advantages of the suggested electrodispersing technology.

Obtaining of the new data about technological features of manufacturing cheap iron-chromium-nickel powder with spherical shape and required fractional composition, with fabrication of small batches from secondary raw material and at minimal energy expenses and minimal damage to the environment, is considered as scientific and practical importance of solving the proclaimed problem in the field of powder metallurgy of refractory alloys.

Conclusion

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, the following results have been established: the shape of the powder particles is mainly spherical and elliptical; oxygen is contained on the surface of the powder particles, while all other elements (iron, nickel and chromium) are distributed relatively uniformly; powder particles have sizes from 0.1 up to 100.0 μm with an average volumetric diameter 64.9 μm; powder particles include the phases Fe_3O_4, Fe_3Ni_2 and Cr. Analysis of the results of the conducted researches showed that the physico-chemical properties of the obtained iron-chromium-nickel powder are influenced by the chemical composition of the working fluid, as well as by the modes of electrodispersing. It is possible to obtain powder with various sizes by varying the technological parameters of the unit.

2. Use of electrodispersing technology for processing of metal wastes of the alloy Kh25N20 and obtaining iron-chromium-nickel powders on its base is rather actual and requested scientific and practical direction. This recycling method for metal wastes allows to obtain powder particles with the required set of properties, which can be used in creation of superalloys and heat-resistant alloys and coatings. Forming of the particles with spherical shape during electrodispersing of metal wastes of the alloy Kh25N20 is a very important feature of this process, which allows to recommend it for use in various technologies, including the additive ones, where spherical shape is the main requirement to powders for additive production units.

3. Organization of the complete factorial experiment allowed to simulate mathematically electrodispersing process for metal wastes of the alloy Kh25N20 and to improve it via determination of the optimal procedure of powder manufacture. The model of ED process for metal wastes of the alloy Kh25N20 in distilled water was obtained, which establish correlation relationships between dispersed composition of products manufactured in electrodispersing of alloys and energy parameters of electrodispersing process itself (voltage at the electrodes, impulse frequency and capacity of discharge condensers). It provides the required dispersity of iron-chromium-nickel powders.

4. Use of electroerosive dispersing technology increased economical efficiency of recycling of metal wastes due to small energy intensity of the process of powder manufacture.

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REFERENCES

1. Skvortsov A. A., Rybakova M. R., Burukin I. V., Gnatyuk E. O. Methods for hardening and improvement of fatigue characteristics of titanium and iron-chromium nickel alloy samples. *Periodico Tche Quimica*. 2020. Vol. 17. No. 35. pp. 425–436.
2. Skvortsov A. A., Gnatyuk E. O. Hardening of samples made of titanium and iron-chromium-nickel alloys to improve their strength, resource and fatigue characteristics. *Asia Life Sciences*. 2019. No. 2. Suppl. 21. pp. 749–758.
3. Allen T. R., Kenik E. A., Was G. S. Variability of radiation-induced segregation in iron-chromium-nickel alloys. *Journal of Nuclear Materials*. 2000. Vol. 278. No. 2. pp. 149–163. DOI: 10.1016/S0022-3115(99)00263-9.
4. Bilchugov Yu. I., Makarova N. L., Nazarov A. A. On limit of molybdenum content of pitting-corrosion-resistant austenitic steels. *Protection of Metals*. 2001. Vol. 37. No. 6. pp. 597–601. DOI: 10.1023/A:1012879916686.
5. Isaev S. L., Baranov D. A., Shchedrin E. Yu. et al. Structure and properties of welded seams in electron beam welding of iron-chromium-nickel alloy EP718. *Izvestiya vysshikh uchebnykh zavedeniy. Tsvetnaya metallurgiya*. 2023. Vol. 29. No. 6. pp. 44–53. DOI: 10.17073/0021-3438-2023-6-44-53.
6. Zahs A., Spiegel M., Grabke H. J. H. J. Chloridation and oxidation of iron, chromium, nickel and their alloys in chloridizing and oxidizing atmospheres at 400–700 ^{o}C. *Corrosion Science*. 2000. Vol. 42. No. 6. pp. 1093–1122.
7. Wang X., Geysen D., Gerven T. Van et al. Characterization of landfilled stainless steel slags in view of metal recovery. *Frontiers of Chemical Science and Engineering*. 2017. Vol. 11. No. 3. pp. 353–362. DOI: 10.1007/s11705-017-1656-9.
8. Krukovich M. G. Technology to improve the performance properties of heterogeneous boronized layers. *Materials Performance and Characterization*. 2020. Vol. 9. No. 3. pp. MPC20190091. DOI: 10.1520/MPC20190091.
9. Bigeev V. A., Potapova M. V., Potapov M. G. On the problem of ferronickel manufacture via selective reduction of depleted iron-chromium-nickel ores. *Liteinye protsessy*. 2015. No. 14. pp. 23–29.
10. Bigeev V. A., Arslanova M. V. Establishment of the main parameters in the process of ferronickel metallurgical concentration obtained of iron-chromium-nickel ores from Khalilovskoe deposit. *Izvestiya Chelyabinskogo nauchnogo tsentra UrO RAN*. 2004. No. 1. pp. 132–136.
11. Ishkinyaev E. D., Osintsev A. V., Petrovskiy V. N. et al. Study of deformation of composite materials on the base of metastable alloys of the Fe–Cr–Ni system via the method of correlation of digital images. *Zhurnal tekhnicheskoy fiziki*. 2023. Vol. 93. No. 8. pp. 1152–1157. DOI: 10.21883/JTF.2023.08.55977.84-23.
12. Muravyev V. V., Muravyeva O. V., Vladykin A. L. Acoustic and electromagnetic properties of a martensite-ageing iron-chromium-nickel alloy with copper addition in mechanical extension. *Defektoskopiya*. 2023. No. 5. pp. 12–20. DOI: 10.31857/S0130308223050020.
13. Amirkhanova N. A., Galiev V. E., Ustyuzhanina S. V. Features of high-speed dissolution of the iron-chromium-nickel alloy KhN35VTYu applying to electrochemical treatment. *Vestnik Ufimskogo gosudarstvennogo aviatsionnogo tekhnicheskogo universiteta*. 2012. Vol. 16. No. 5 (50). pp. 132–136.
14. Iskalieva A. T., Bigeev V. A., Potapov M. G., Potapova M. V. Determination of reducing agent consumption for ferrosilicochromium production from oxide melt at the second processing stage of iron-chromium-nickel ores from Khalilovskoe deposit. *Tekhnologii metallurgii, mashinostroeniya i metalloobrabotki*. 2019. No. 18. pp. 17.
15. Plaskeev A. V., Feldgandler E. G. Influence of copper, molybdenum and silicon on corrosion-electrochemical behaviour of austenite chromium-nickel steel. *Zashchita metallov*. 2000. Vol. 36. No. 4. pp. 375-382.
16. Ageev E. V., Ageeva E. V., Khardikov S. V. Structure and properties of sintered corrosion-resistant steel manufactured from electroerosive powders. *CIS Iron and Steel Review*. 2021. Vol. 22. pp. 88–91.
17. Ageev E. V., Ageeva E. V., Altukhov A. Yu. Structure and properties of additive products manufactured from electroerosion powders. *CIS Iron and Steel Review*. 2022. Vol. 23. pp. 92–97.