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Key words: *additive products, cobalt-chromium alloys, electroerosive dispersion, powder, certification of the properties of cobalt-chromium powders, spherical shape of particles*

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STRUCTURE AND MECHANICAL PROPERTIES OF POWDERS OBTAINED BY ELECTRODISPERGING COBALT-CHROMIUM ALLOY

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The work presents the results of attestation of powders that were obtained from the KHMS "Cellite" alloy (Co-63%, Cr-27%, Mo-5%, Ni-2%, Fe-2%) by electroerosive dispersion under various technological conditions (voltage from 100V to 220V, the capacitance of condenser from 15μF to 50μF, pulse frequency from 100Hz to 200Hz), and with using working fluids of different chemical composition and properties (water, kerosene, butyl alcohol). The study of the dispersion of the obtained powders, based on the results, established: the range of particle sizes is from 20μm to 110μm depending on the production modes. The results show various particle sizes, both a few nanometers and hundreds of microns. Depending on the technological modes of production, various mechanisms of the formation of powder particles can occur. Flake particles ranging in size from a few nanometers to (as a rule) one micron are obtained by the crystallization of the material vapor. They usually form agglomerates or stick to larger particles. Spherical and elliptical particles with a diameter from tens of nanometers to hundreds of microns were formed in crystallized material upon melting. The result of thermal and mechanical action during electroerosive dispersion was fragmentation grains with an average size from units to hundreds of microns. To meet the requirements for powders used in additive machines, it is necessary to select modes that exclude brittle destruction of the particles of the powder material and ensure the production of spherical or elliptical particles in the required particle size ranges. As a result of the experiment during the study of the phase composition of powders, using various technological modes and the composition of working fluids, the following phases were revealed: Cobalt (Co) with a cubic crystal lattice, a=b=c=3.561079 Å; Chromium (Cr) with a hexagonal crystal lattice a=b=2.738459 Å, c=4.55078 Å; Nickel (Ni) with a hexagonal crystal lattice, a=b=2.652590 Å, c=4.380519 Å; sigma-Cr7Co3 (Cr7Co3 with a tetragonal crystal lattice, a=b=8.656172 Å, c=4.484030 Å; Cobalt Iron (CoFe), with a cubic crystal lattice, a=b=c=2.846754 Å; Chromium Carbide (Cr_3C_2) with an orthorhombic crystal lattice: a=2.821Å, b=5.53Å and c=11.47Å; Iron (Fe) with a cubic crystal lattice, a=b=c=3.604293 Å; Cobalt Carbide (Co_3C), with an orthorhombic crystal lattice, a=b=4.455931 Å, c=6.86598Å; Cobalt Oxide (CoO) with a cubic crystal lattice a=b=c=4.563279 Å; Magnetite (Fe_3O_4) with a cubic crystal lattice a=b=c=8.4774342 Å.

Key words: additive products, cobalt-chromium alloys, electroerosive dispersion, powder, certification of the properties of cobalt-chromium powders, spherical shape of particles

INTRODUCTION

One of the rapidly developing and promising industries is the manufacture of products based on metals and their alloys using additive manufacturing technologies (AT). All developed countries of the world are actively conducting research and applied work to improve the technologies for the production of powders, so the development of ATs themselves [1-4]

Important tasks in the production of parts, in addition to maintaining the accuracy of the shape, is the control of such material properties as: strength and wear resistance, plasticity and elasticity, etc. For example, any of the processing processes using additive technologies negatively affects the material of the workpiece, due to high-temperature exposure. Therefore, with a multiple increase in production capacity, for additive technologies, the primary goal is to ensure high operational properties of the product and ensure its high-quality structure [5-8].

Particles with a spherical shape and imparting "fluidity" to the powder composition in material supply systems are placed in a given volume most efficiently, and therefore, for three-dimensional additive technologies, are the main requirement for powders. The powder should have a microdisperse and homogeneous structure, with a uniform distribution of the phase composition. In this case, the amount of gas in the powder should be minimal.

Electroerosive dispersion (EED) is a technology characterized by low energy consumption and environmental friendliness. It is proposed, based on technological features, for use in additive machines of spherical powder particles [9-10]. The main advantage of the EED technology is the possibility of using production wastes, which are cheaper in comparison with pure components. Also, it makes it possible to obtain powder particles from multicomponent alloys.

The tasks of improving the properties of products can be solved by using new materials. These materials include, first of all, alloy powders. When working with them, it is necessary to study in detail their characteristics, properties, and structure.

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Powdered products have the disadvantage that the components at different points in the powder are in unequal concentration. This disadvantage can manifest itself, both in the initial state, and persist until the end of production. The preservation of stable and high mechanical properties impose restrictions on the use of these materials in the manufacture of a wide range of parts for various branches of mechanical engineering.

To eliminate the indicated disadvantages for additive technologies, it is proposed to use alloy powders obtained from metal alloy waste by electroerosive dispersion. The work will contribute to solving the problem of expanding the range of materials suitable for additive technologies.

The purpose of this work is to certify the properties of powders obtained from waste cobalt-chromium alloy by electroerosive dispersion in distilled water and butyl alcohol.

RESEARCH MATERIALS AND METHODS

The process of electroerosive dispersion (Fig. 1) of the KHMS "Cellite" alloy (Co-63%, Cr-27%, Mo-5%, Ni-2%, Fe-2%) by electroerosive dispersion was carried out on the original installation shown in Fig. 1 As a result of the dispersion of the wastes of the cobalt-chromium alloy, its destruction occurred in the working fluid, under the influence of short-term electric discharges between the electrodes.

To electrodes 2 and 3, and then to the waste of alloy 6, the pulse voltage of generator 1 is applied. An electrical breakdown in the working fluid 5, which is located in the interelectrode space, occurs, with the formation of a discharge channel 7, upon reaching a certain voltage. At discharge point 8, the material evaporates and melts under the influence of concentrated heat energy. Decomposition products 9 in the form of gas, due to the evaporation of the working liquid, surround the discharge channel.

As a result of significant dynamic forces developing in the discharge channel and the decomposition products of the working fluid, drops of molten material 4 are thrown out of the discharge zone into the working fluid surrounding the electrodes and freeze in it, forming drop-like particles of hard alloy.

Distilled water and butyl alcohol were used as the working fluid. The dispersion was carried out on an installation for EED of conductive materials.

Electrodispersion modes are presented in Table 1.

The obtained powders were certified using modern complementary methods of physical materials science. The

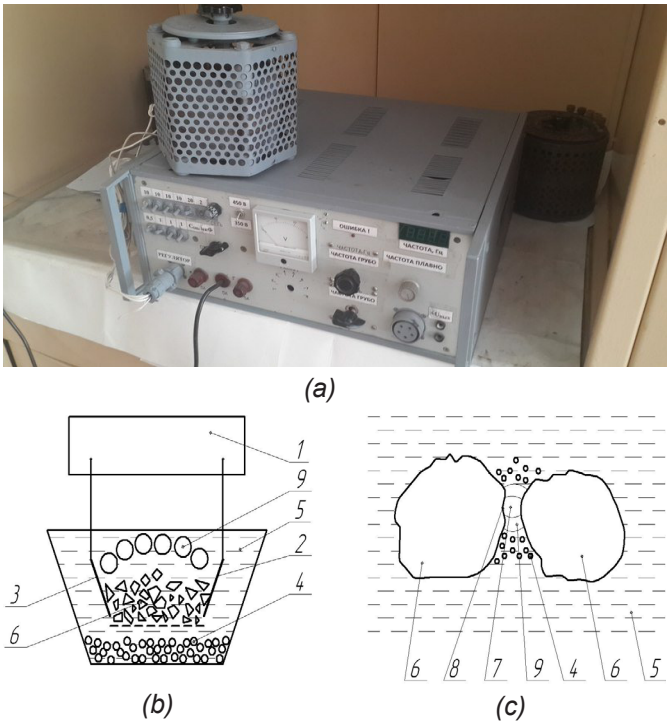

Figure 1: Process of EED of KHMS alloy: a) photo of the installation; b) installation diagram; c) process diagram

study of the microstructure and morphology of the powders was carried out on a QUANTA 200 3D scanning electron microscope (SEM) (manufactured by FEI (Holland)). The studied powders were placed on conductive graphite tape glued to the microscope stage to provide the necessary grounding. Images of the sample surface were obtained using a wide-field secondary electron detector at an accelerating voltage of 20 kV in a low vacuum mode. Surfaces of powder samples As a result of energy-dispersive analysis of X-ray radiation of the surface of powder samples, using a QUANTA 200 3D electron microscope and an EDAX analyzer, their spectra were obtained.

X-ray spectral microanalysis (X-ray microanalysis) was performed using an EDAX energy-dispersive X-ray analyzer built into a Nova NanoSEM 450 scanning electron microscope.

The study of the dispersion of powders was carried out using a laser particle size analyzer "Analysette 22 NanoTec" following the research methodology FR 1.27.2009.06762 "Procedure for measuring particle size in suspensions, emulsions, and aerosols in the nanometer and colloidal ranges using the effect of dynamic light scattering."

Table 1: Technological modes of obtaining samples of cobalt-chromium powders

Sample number powder	1	2	3	4	5	6	7	8
Dispersing working environment	distilled water				butyl alcohol			
Electrode voltage, V	140–150	80–90	120–130	120–130	95–105	95–105	120–130	150–160
Capacity of discharge capacitors, Hz	65,5	55,5	45,5	35,5	48	48	45,5	48
Pulse repetition rate, μF	160–170	150–160	120–130	140–150	95–105	75–85	160–170	75–85

Of the obtained powders was certified by X-ray diffraction using an Ultima IV Rigaku diffractometer. The phase composition was certified using an Ultima IV Rigaku by X-ray diffraction according to the standard procedure in the PDXL program using the PDF-2 powder X-ray standards database (JCPDS ICDD) (2008).

RESULTS AND DISCUSSION

Microanalysis and microscopy of powders obtained in distilled water and butyl alcohol led to the results shown in Figure 2 and Tables 2 and 3.

The experiment found that the composition of the powder in various media mainly consists of the following elements: Co, Cr, Mo, Ni, Fe, C and O.

In the study of the morphology of the powders, it was found that the powder can consist of particles of regular spherical shape, elliptical, and conglomerates. Powder particles in the form of a sphere or ellipse are explained by rapid crystallization and quenching during EED and ejection into the working fluid. At a significant temperature range of colliding particles, they stick together with the formation of fragile boundaries. This is due to the

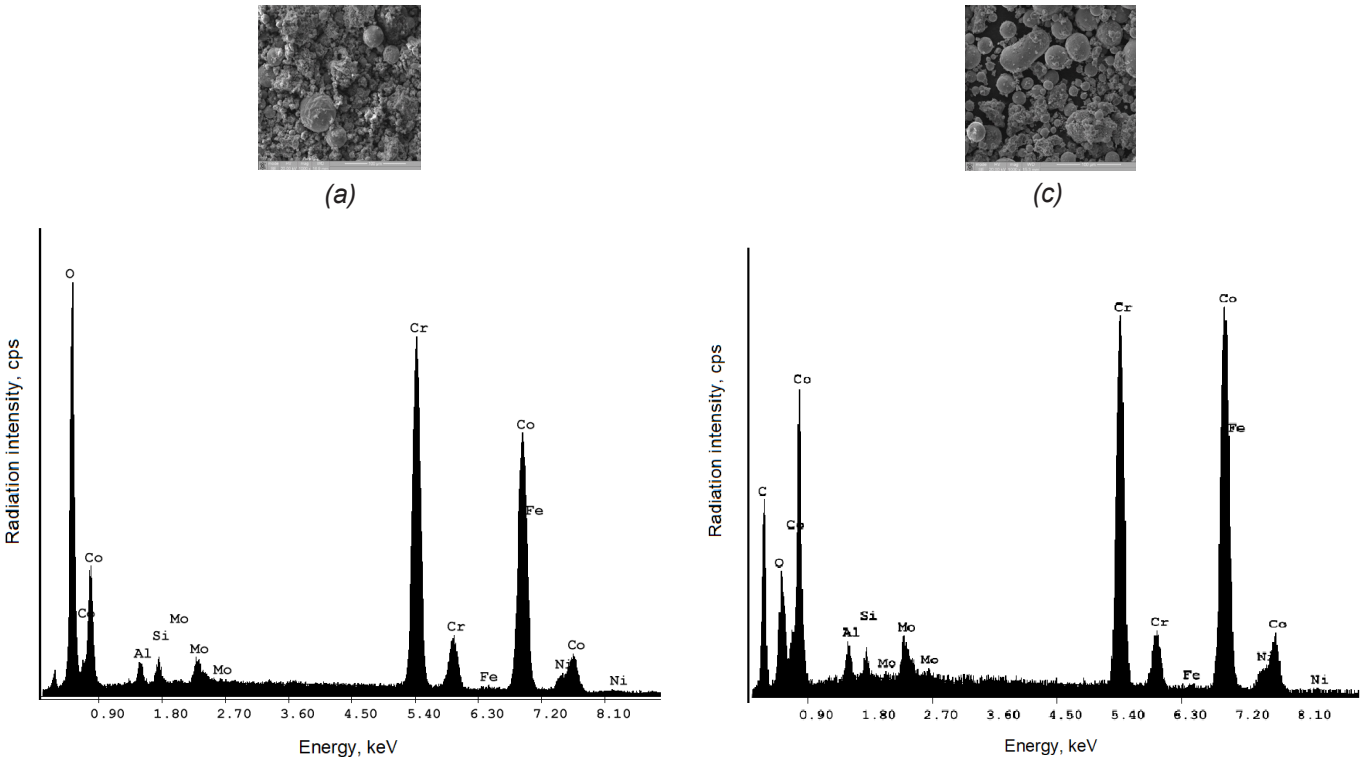

Figure 2: Electron microscopic image and elemental composition of the powder obtained: a) and b) in distilled water; c) and d) in butyl alcohol, respectively

Table 2: Elemental composition of the powder

Element	Dispersion environment			
	Water		Alcohol	
	Mass fraction,%	Atomic fraction,%	Mass fraction,%	Atomic fraction,%
C	_	_	25,37	57,42
O	16,75	40,97	16,75	40,97
Al	1,04	1,50	1,04	1,50
Si	0,98	1,36	0,98	1,36
Mo	2,00	0,82	2,00	0,82
Cr	30,65	23,07	30,65	23,07
Fe	0,53	0,37	0,53	0,37
Co	44,78	29,73	44,78	29,73
Ni	3,27	2,18	3,27	2,18
Total	100,00	100,00	100,00	100,00

collision of particles of different sizes formed from the liquid and vapor phases. Particles, if the difference in their temperatures is insignificant, can form conglomerates of indefinite shape upon collision.

The established technological features of structure formation subsequently made it possible to determine the optimal dispersion modes for the cobalt-chromium alloy, which would provide a spherical or elliptical shape of powder particles.

X-ray diffraction patterns of the samples under study are shown in Figures 3 and 4.

The position and interplanar distances of all reflections are presented in Table 3.

The study of the phase composition of powders, based on the results, experimentally established that, depending on the technological modes of production and the composition of working fluids, the following main phases can occur: Cobalt (Co) with a cubic crystal lattice, a=b=c=3.561079 Å; Chromium (Cr) with a hexagonal crystal lattice a=b=2.738459 Å, c=4.55078 Å; Nickel (Ni) with a hexagonal crystal lattice, a=b=2.652590 Å, c=4.380519 Å; sigma-Cr7Co3 (Cr_7Co_3 with a tetragonal crystal lattice, a=b=8.656172 Å, c=4.484030 Å; Cobalt Iron (CoFe), with a cubic crystal lattice, a=b=c=2.846754 Å; Chromium Carbide (Cr_3C_2) with an orthorhombic crystal lattice: a=2.821Å, b=5.53Å and c=11.47Å; Iron (Fe) with a cubic crystal lattice, a=b=c=3.604293 Å; Cobalt Carbide (Co_3C), with an orthorhombic crystal lattice, a=b=4.455931 Å, c=6.86598 Å; Cobalt Oxide (CoO) with a cubic crystal lattice a=b=c=4.563279 Å; Magnetite (Fe_3O_4) with a cubic crystal lattice a=b=c=8.4774342 Å.

The determination of the size distribution of microparticles of powder samples, presented in Figures 5-12 and Tables 3 and 4, was carried out.

It was found that the average particle size is 24.97 microns, the arithmetic value is 24.97 microns. The determination of the size distribution of microparticles of the sample "No. 2", obtained in distilled water (Figure 6).

It was found that the average particle size is 26.58 microns, the arithmetic value is 26.576 microns.

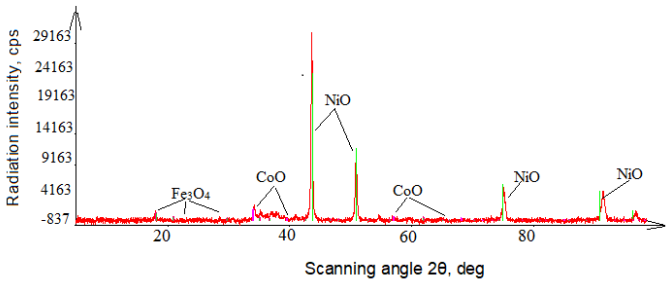

Figure 3: Diffraction pattern of a powder sample obtained in distilled water

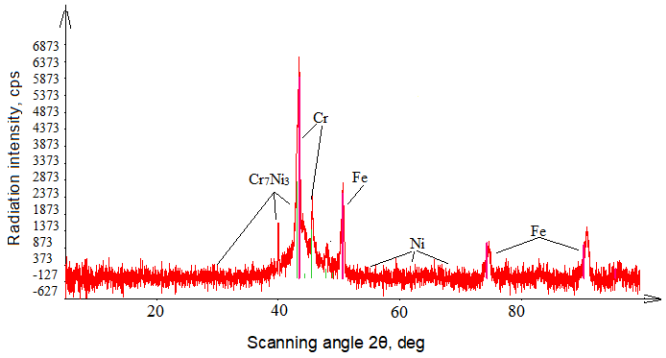

Figure 4: Diffraction pattern of a powder sample obtained in butyl alcohol

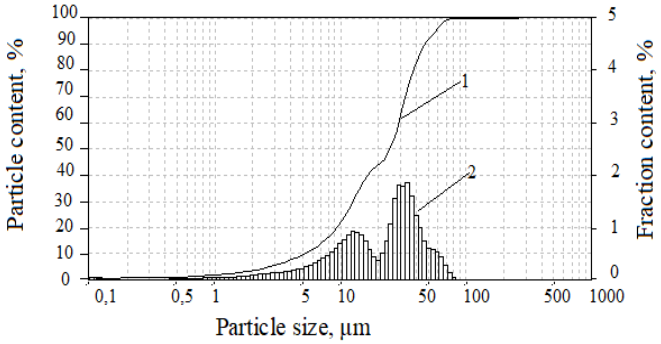

Figure 5: Size distribution of microparticles of sample "No. 1": 1 - integral curve, 2 - histogram

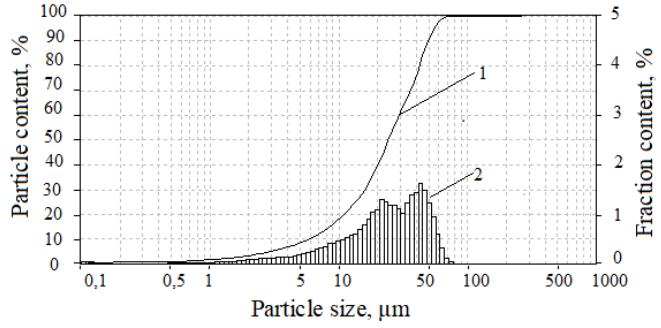

Figure 6: Size distribution of microparticles of sample "No. 2": 1 - integral curve; 2 - histogram

Table 3: Lattice periods

Sample No.	Периоды решеток, Å		
1	Iron Nickel (Fe0.64 Ni0.36) 225:Fm-3m Cubic crystal lattice a=b=c=3.586800 Å	Cobalt Oxide (CoO) 216:F-43m Cubic crystal lattice a=b=c=4.563279 Å	Magnetite (Fe_3O_4) 216:F-43m Cubic crystal lattice a=b=c=8.4774342 Å
5	Iron (Fe) 63:Cmcm 225:Fm-3m Cubic crystal lattice a=b=c=3.604293 Å	sigma-Cr_7Ni_3 (Cr_7Ni_3) 136:P42/mnm Tetragonal crystal lattice a=b=8.656172Å, c=4.484030 Å	-

The determination of the size distribution of microparticles of the sample "No. 3", obtained in distilled water (Figure 7).

It was found that the average particle size is 27.93 microns, the arithmetic value is 27.927 microns.

Determination of the size distribution of microparticles of the sample "No. 4", obtained in distilled water (Figure 8).

It was found that the average particle size is 30.68 microns, the arithmetic value is 30.682 microns.

The determination of the size distribution of microparticles of the sample "No. 5", obtained in butyl alcohol (Figure 9).

It was found that the average particle size is 31.59 microns, the arithmetic value is 31.585 microns.

The determination of the size distribution of microparticles of the sample "No. 6", obtained in butyl alcohol (Figure 10).

It was found that the average particle size is 28.95 microns, the arithmetic value is 28.945 microns.

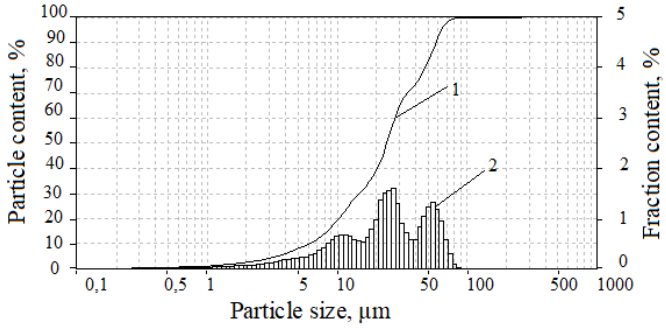

Figure 7: Size distribution of microparticles of sample "No. 3": 1 - integral curve; 2 - histogram

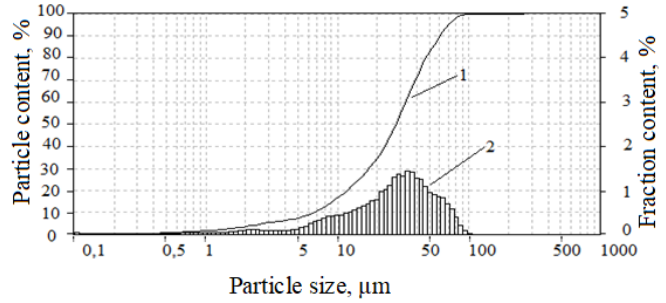

Figure 8: Size distribution of microparticles of sample "No. 4": 1 - integral curve; 2 - histogram

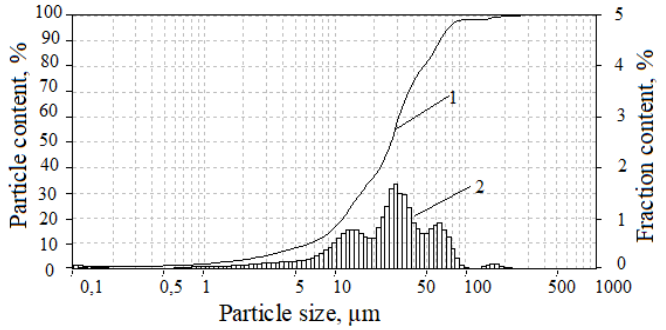

Figure 9: Size distribution of microparticles of sample "No. 5": 1 - integral curve; 2 – histogram

Determination of the size distribution of microparticles of the sample "No. 7" obtained in butyl alcohol (Figure 11).

It was found that the average particle size is 27.09 microns, the arithmetic value is 27.088 microns.

The determination of the size distribution of microparticles of the sample "No. 7", obtained in butyl alcohol (Figure 12).

It was found that the average particle size is 33.61 microns, the arithmetic value is 33.61 microns.

The conducted studies of the influence of technological modes of electroerosive dispersion (voltage was changed from 20V to 220V, capacitance of capacitors from 10μF to 100μF, pulse frequency from 50Hz to 400Hz) on the properties of metal powders. The following main results can be distinguished.

Based on the results of the study of the dispersion of powders obtained under various technological modes

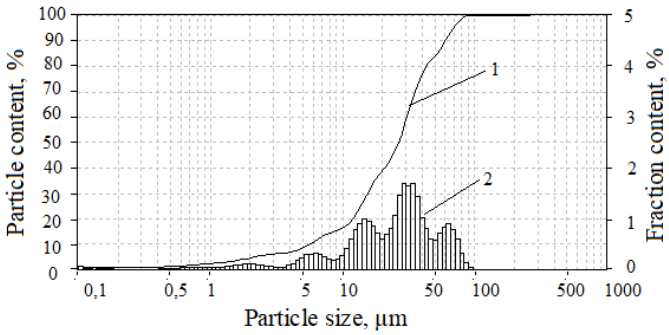

Figure 10: Size distribution of microparticles of sample "No. 6": 1 - integral curve; 2 - histogram

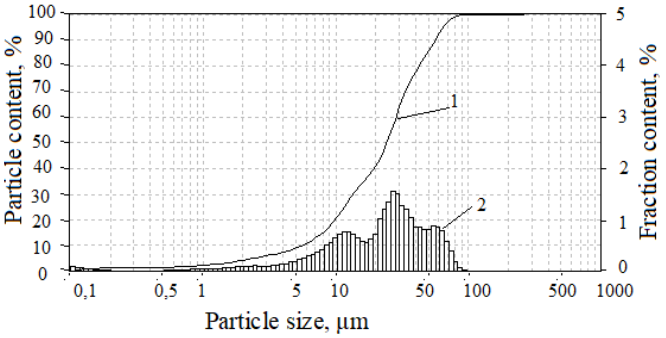

Figure 11: Size distribution of microparticles of sample "No. 7": 1 - integral curve; 2 - histogram

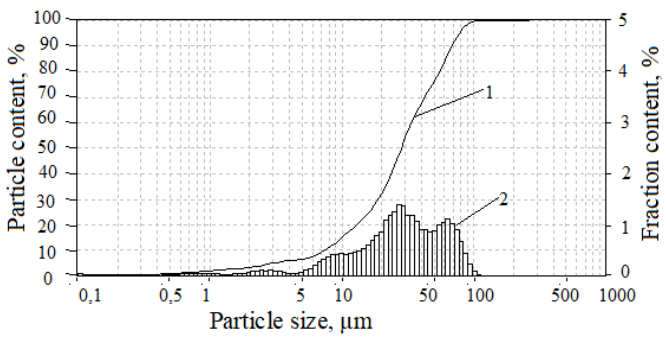

Figure 12: Size distribution of microparticles of sample "No. 8": 1 - integral curve; 2 - histogram

(the range was determined earlier, the voltage was from 80 to 150V, the capacitance of the capacitors was from 30μF to 65μF, the pulse frequency was from 100Hz to 200Hz), mathematical dependences were obtained that make it possible to describe the influence of dispersion technological modes on the particle size distribution of metal-powder compositions. To obtain powders of a given size, it is most expedient to change the capacitance of the discharge capacitors or the voltage at the electrodes of the reactor, which directly depends on the supply voltage of the EED unit (pulse generator), and leave the remaining parameters constant.

On the basis of theoretical studies, the following working fluids used for the process of electroerosive dispersion were selected: water (oxygen-containing medium) and butyl alcohol (carbon- and oxygen-containing liquid) and the effect of their composition on the elemental and phase compositions of metal-powder compositions was investigated.

It has been established that the properties of the working dispersion medium affect the elemental and phase compositions of the powder material. So, for example, when using distilled water, the mass fraction of oxygen in the powder, depending on the technological modes of production, is 4-11% and oxides are formed, for example, Cobalt Oxide (Co O), Magnetite (Fe_3O_4), etc.

When using butyl alcohol, oxygen compounds in the powder are insignificant (from 0.1% to 2%, depending on the technological modes of production), but carbon compounds are present in the form of chromium carbides Cr_3C_2 and cobalt carbides Co_3C, the presence of which can subsequently increase the hardness and wear resistance of parts obtained by laser sintering on additive machines.

CONCLUSIONS

The main results of the work carried out aimed at the certification of powders obtained from the KHMS "Cellite" alloy (Co-63%, Cr-27%, Mo-5%, Ni-2%, Fe-2%) by electroerosive dispersion under various technological conditions are as follows:

1. The average particle size of the obtained cobalt-chromium powders is, depending on the technological modes of production, from 20μm to 110μm. It was also found that the resulting powders can have a fairly wide range of particle size distribution - from several nanometers to hundreds of microns.
2. Depending on the technological modes of production, various mechanisms of the formation of powder particles can occur. Flake particles ranging in size from a few nanometers to (usually) 1 micron are obtained by crystallization of the material vapor. They usually form agglomerates or stick to larger particles. Spherical and elliptical particles with a diameter from tens of nanometers to hundreds of microns (depending on the production mode) are formed by crystallization of the molten material.
3. As a result of the study of the phase composition of the powders, it was experimentally established that, depending on the technological modes of production and the composition of the working fluids, the following main phases can occur: Cobalt (Co) with a cubic crystal lattice, a=b=c=3.561079 Å; Chromium (Cr) with a hexagonal crystal lattice a=b=2.738459 Å, c=4.55078 Å; Nickel (Ni) with a hexagonal crystal lattice, a=b=2.652590 Å, c=4.380519 Å; sigma-Cr7Co3 (Cr7Co3 with a tetragonal crystal lattice, a=b=8.656172 Å, c=4.484030 Å; Cobalt Iron (CoFe), with a cubic crystal lattice, a=b=c=2.846754 Å; Chromium Carbide (Cr_3C_2) with an orthorhombic crystal lattice: a=2.821Å, b=5.53 Å and c=11.47 Å; Iron (Fe) with a cubic crystal lattice, a=b=c=3.604293 Å; Cobalt Carbide (Co_3C), with an orthorhombic crystal lattice, a=b= 4.455931 Å, c=6.86598 Å; Cobalt Oxide (CoO) with a cubic crystal lattice a=b=c=4.563279 Å; Magnetite (Fe_3O_4) with a cubic crystal lattice a=b=c=8.4774342 Å.

The dependences of the properties of cobalt-chromium metal powders (microstructure, elemental composition, morphology, dispersity, phase composition) on the composition and properties of the starting material, technological regimes and properties of the working dispersion medium obtained on the basis of experimental studies will make it possible to effectively control the process of obtaining metal-powder compositions based on Co-Cr alloys, suitable for additive technologies, and will be used in modeling this process.

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