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X-RAY SPECTRAL MICROANALYSIS OF SINTERED SAMPLES FROM ELECTROEROSIVE COBALT-CHROMIUM POWDERS

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To develop technologies for the reuse of alloy powders obtained from cobalt-chrome wastes and to evaluate the effectiveness of their use, complex theoretical and experimental studies are required. The main advantage of the proposed technology is the use of waste as raw materials, which is much cheaper than the pure components used in traditional technologies. The purpose of this work was to perform X-ray spectral microanalysis of sintered samples from cobalt-chrome powders obtained for additive technologies by electroerosive dispersion. For the implementation of the planned studies, wastes of the cobalt-chrome alloy of the brand KHMS "CELLIT" were chosen. For the production of cobalt-chrome powders, a device for electro-erosive dispersion of conductive materials was used. As a working fluid, butyl alcohol was used. The powders are consolidated by the method of spark plasma sintering using the system of spark plasma sintering SPS 25-10. Using the energy-dispersive X-ray analyzer from EDAX, built into the scanning electron microscope QUANTA 200 3D, spectra of characteristic X-ray radiation were obtained at various points on the surface of the sample. It has been experimentally established that Co and Cr are mainly contained in cobalt-chrome powder-alloys on the surface.

Key words: Cobalt-chromium alloys, Electroerosive dispersion, Powder, Spark plasma sintering, X-ray spectral analysis

INTRODUCTION

Additive technologies (AT) production of products from materials based on metals and alloys is one of the most promising and actively developing areas of production. Research and application work on the development and improvement of both ATs themselves and the technologies for the production of precursor powders for them are actively conducted in all developed countries of the world.

At present, Russian organizations that have purchased expensive equipment for AT are dependent on a foreign supplier and are forced to purchase imported powder materials. Different companies-manufacturers of AT-machines prescribe work with a certain list of materials, usually supplied by this company. At the same time, granules of limited compositions that exclude high-temperature application in the interests of rocket and space technology (RST) are delivered to Russia.

The main requirement for powders for additive 3d-technologies is the spherical shape of the particles. Such particles most compactly fit into a certain volume and ensure the "fluidity" of the powder composition in the supply systems of the material with minimal resistance. In addition, the powder should contain a minimum amount of dissolved gas. The microstructure of the powder must be uniform and finely dispersed (with a uniform distribution of phase constituents) [01-05].

Proceeding from the peculiarities of the methods for obtaining spherical powders with the aim of obtaining spherical granules of regulated granularity, the electroerosive dispersion technology is proposed, which is characterized by relatively low energy costs and ecological purity of the process [06-09].

The main advantage of the proposed technology is the use of waste as raw materials, which is much cheaper than the pure components used in traditional technologies. In addition, this technology is powdered, which allows powder-alloys.

The wide use of the EED method for the processing of metal waste into powders for the purpose of their reuse and application in additive technologies is hampered by the lack in the scientific and technical literature of full-fledged information on the effect of the initial composition, regimes and media on the properties of powders and technologies of practical application. Therefore, in order to develop technologies for the reuse of alloy powders obtained from nichrome waste and to evaluate the effectiveness of their use, complex theoretical and experimental studies are required.

The purpose of this work was to perform X-ray diffraction analysis of sintered samples from cobalt-chromium powders obtained for additive technologies by electroerosive dispersion.

MATERIALS AND METHODS

For the implementation of the planned studies, the wastes of the cobalt-chromium alloy of the brand KHMS "CELLIT" were chosen. Isobutyl alcohol was also used as the working fluid. For the production of cobalt-chrome

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powders, a unit for EED of conductive materials was used. Dispersion parameters: voltage 100 V, capacity 48 μF, repetition rate 120 Hz.

The powders are consolidated by the method of spark plasma sintering using the spark plasma sintering system SPS 25-10 (Thermal Technology, USA).

The starting material was placed in a matrix of graphite placed under a press in a vacuum chamber. Electrodes integrated into the mechanical part of the press feed electric current to the matrix and create spark discharges between the sintered particles of the material, providing intensive interaction.

The process of powder consolidation is schematically shown in Figure 1.

The microstructure of sintered samples was studied by scanning electron microscopy.

Using the energy-dispersive X-ray analyzer from EDAX, built into the scanning electron microscope QUANTA 200 3D, spectra of characteristic X-ray radiation were obtained at various points on the surface of the sample.

By X-ray spectral microanalysis is meant the determination of the elemental composition of microobjects by the characteristic X-ray radiation excited in them. For the analysis of the characteristic spectrum in X-ray spectral analysis, two types of spectrometers are used (uncrystalline or with a crystal analyzer), the base for X-ray spectral analysis is the electron-optical system of a scanning electron microscope.

RESULTS AND ITS DISCUSSION

The results of microscopy and microanalysis of the powders are shown in Figures 2, 3 and Table 1.

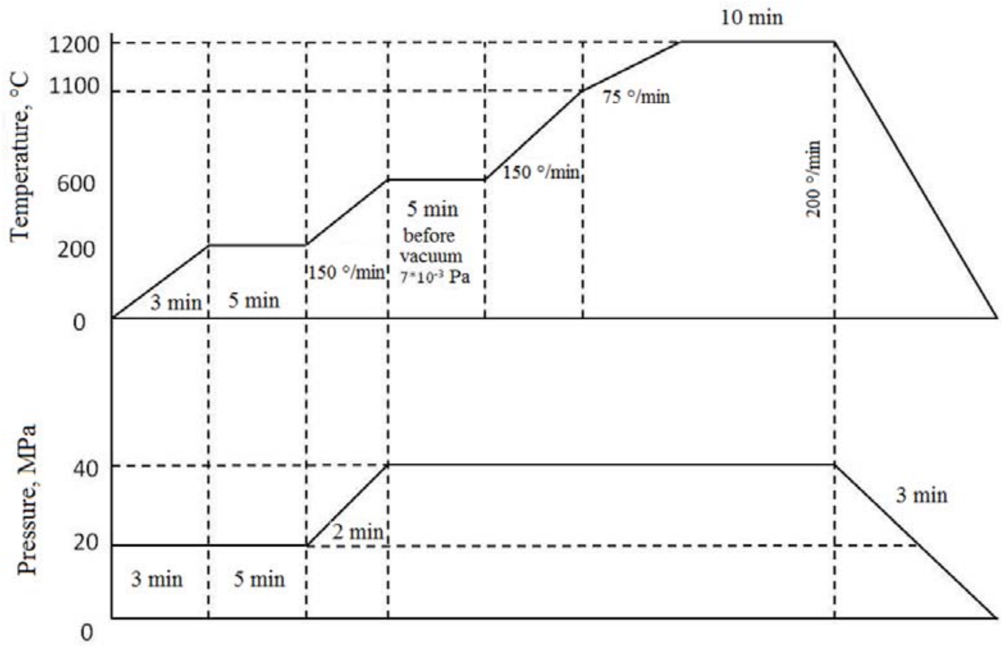

Figure 1: Consolidation of powders by the method of spark plasma sintering (scheme)

Table 1: Element composition of the sintered sample

Element	Wt %	At %
S i K	1.11	2.27
M o L	5.39	3.22
C r K	28.89	28.59
F e K	0.72	0.74
C o K	63.19	61.56
N i K	3.69	3.61
Total	100.00	100.00

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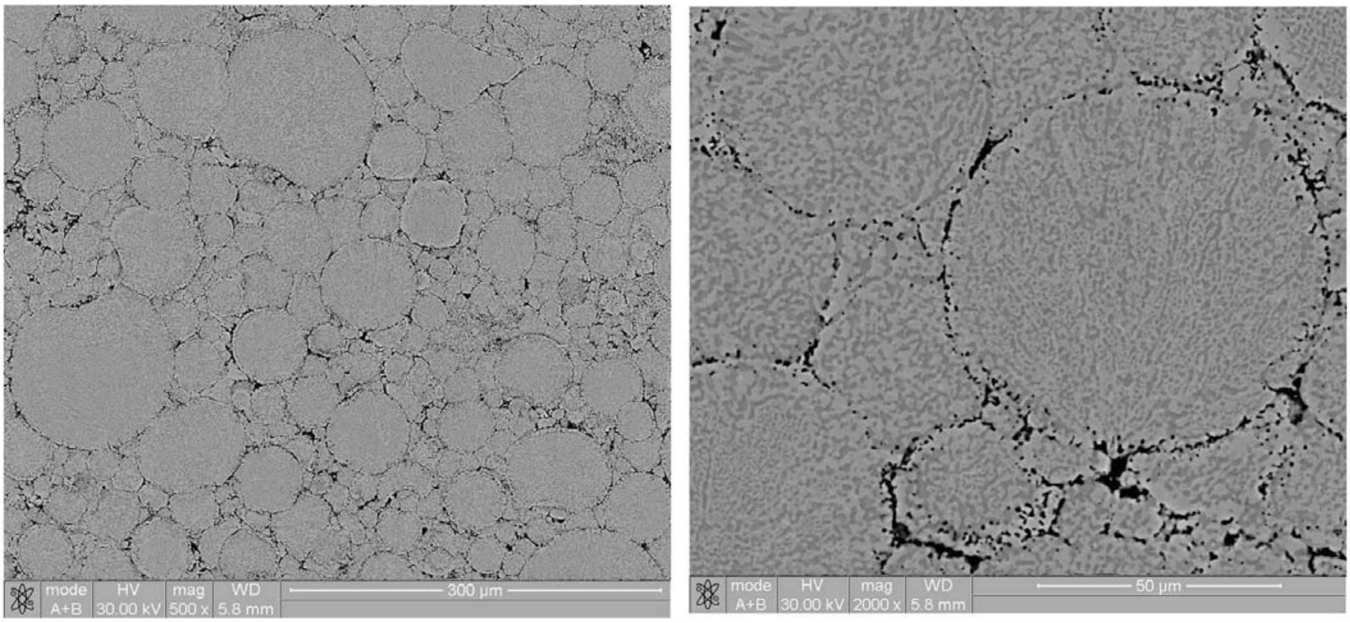

a) b)

Figure 2: Electron microscopic image of a sintered sample with an increase in scale: a) x500; b) x2000

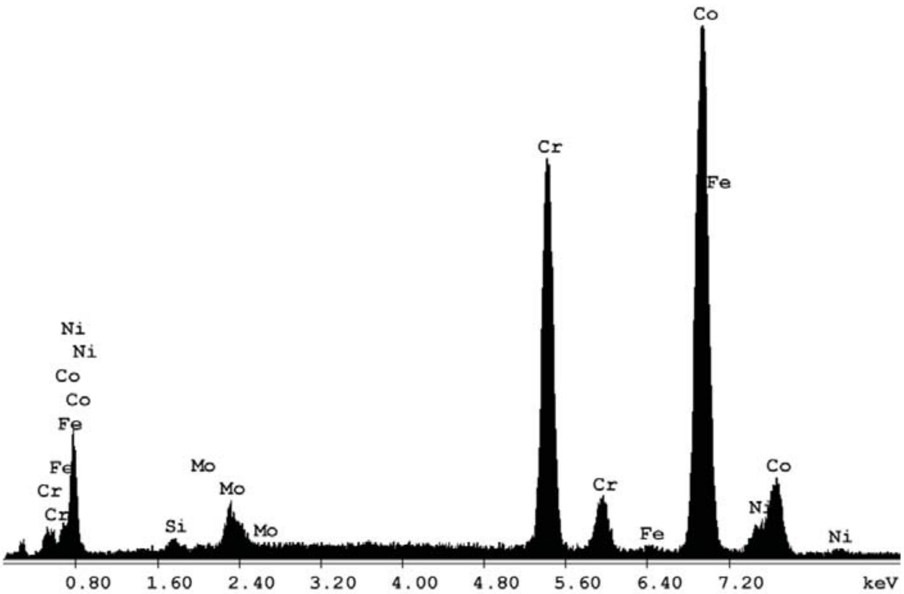

Figure 3: Element composition of the sintered sample

CONCLUSIONS

Thus, based on the conducted experimental studies aimed at conducting X-ray spectral microanalysis of sintered samples from cobalt-chromium powder-alloys obtained for additive technologies by electro-erosive dispersion of metal wastes of KHMS alloy in isobutyl alcohol, the surface contains mainly Co and Cr.

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