X-RAY STRUCTURAL ANALYSIS OF SINTERED SAMPLES FROM ELECTROEROZIVE COBALT-CHROME POWDERS

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Abstract. Based on the results of studies aimed at performing X-ray structural analysis of sintered samples from cobalt-chromium powders obtained for additive technologies by electroerosive dispersion, it was established that the main phases in the samples are Co, CoFe, and FeSi.
Key words: cobalt-chromium alloys, electroerosive dispersion, powder, spark plasma sintering, X-ray diffraction analysis.

1. Introduction
The main requirement for powders for additive 3d-technologies is the spherical shape of the particles. Such particles must 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) [1-10].

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 [11, 12].

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 allows one to vary the granulometric composition of the resulting powder due to changes in electrical parameters.

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

2. 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-chromium powders, a unit for EED conductive materials was used. Dispersion parameters are:
- sample No. 1 (voltage 100 V, capacity 48 $\mu$F, pulse repetition frequency 120 Hz);
- sample No. 1 (voltage 140 V, capacity 48 $\mu$F, pulse repetition frequency 80 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. The 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.

Figure 1. Consolidation of powders by the method of spark plasma sintering (scheme)
Figure 2. Technology of spark plasma sintering: a) schematic diagram of SPS synthesis; b) general scheme of heating by the method of SPS
Advantages of the technology are as follows: uniform distribution of heat over the sample; high density or controlled porosity; bonding materials are not required; uniform sintering of homogeneous and dissimilar materials; short cycle time; the production of the part immediately in the final form and the obtaining of a profile close to the given one.

3. Results and its discussion

The phase composition of the samples was studied by X-ray diffraction on a Rigaku Ultima IV diffractometer in Cu-Kα radiation (wavelength λ = 0.154178 nm) using Soller slits. The diffraction spectrum for the phase analysis is sampled according to the θ-2θ scanning scheme with Breguot-Brentano focusing in the angular interval of 5 ... 100 deg. 2θ. The shooting is carried out in a point-by-point mode with a scanning step of Δ(2θ) = 0.02 deg, speed - 0.6 deg / min, working voltage - 45 kV, current - 200 mA. To refine the profile of the experimental radiographs, the software package PDXL RIGAKU was used. Subtraction of the background was carried out by the Sonneveld-Visser method, the smoothing of the experimental profile - by the Savitsky-Naked method, and the separation of components kα1 and kα2 - by the Racinger method. To describe the diffraction maxima, a superposition of the Gaussian function and the Lorentz function was used. The approximation of each of the reflections in the diffractograms of the samples studied by the pseudo-Voigt function made it possible to accurately determine the position of the reflections, taking into account the displacement caused by the overlap of the reflexes, half the intensity maximum and the intensity itself. The phase composition of the coatings was determined using the ICCD PDF-2 database (2014).

The X-ray patterns of the samples are shown in Figure 3.
Figure 3. Diffractogram: a) sample No. 1; b) sample No. 2

The position and interplanar distances of all reflections are shown in Table 1.

Table 1. Position and interplanar distances of all reflections

| Sample | Periods of gratings, Å |
|--------|------------------------|
| № 1    |                        |
| Cobalt (Co) 225:Fm-3m | Nickel (Ni) 194:P63/mmc |
| Cubic crystal lattice | Hexagonal crystal lattice |
| a = b = c = 3,561079 Å | a = b = 2,652590 Å, c = 4,380519 Å |
| Chromium Nickel Silicon (Cr₃Ni₅Si₂) 198:P213 | Fersilicite (FeSi) 198:P213 |
| Cubic crystal lattice | Cubic crystal lattice |
| a = b = c = 6,111743 Å | a = b = c = 4,397473 Å |
| Nº 2    |                        |
| Chromium Nickel (Cr₀.8Ni₀.2) 229:1m-3m | Cobalt (Co) 225:Fm-3m |
| Cubic crystal lattice | Cubic crystal lattice |
| a = b = c = 2,869322 Å | a = b = c = 3,564040 Å |
| Cobalt Iron (CoFe) 229:1m-3m | Molybdenum (Mo) 229:1m-3m |
| Cubic crystal lattice | Cubic crystal lattice |
| a = b = c = 2,846754 Å | a = b = c = 3,211225 Å |
| Cobalt Silicate (Co₂(SiO₄)) 62:Pbnm |                        |
| Orthorhombic crystal lattice |                        |
| a = 4,711625, b = 10,322408 Å, c = 6,043066 Å |

4. Conclusion

Thus, based on the results of studies aimed at X-ray diffraction analysis of sintered samples from cobalt-chromium powders obtained for additive technologies by electroerosive dispersion, it was established that the main phases in the samples are Co, CoFe, and FeSi.
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