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Short Communication

X-ray Microanalysis of Sintered Nanocomposite Materials from Tungsten-containing Powders

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Presents the results of microanalysis of the samples sintered from tungsten-containing powders. It is established that the main elements in the composition of the sintered samples from powders of the tungsten are carbon, iron, tungsten and cobalt.

Keywords: The sintered samples, X-ray microanalysis, Electroerosion dispersion, Elemental composition of the powder.

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1. INTRODUCTION

Currently, tungsten-containing alloys are widely used in industry. Solid carbide grade VK 8 is used for processing by cutting materials, processing of stainless steel, high-strength and heat resisting hard- processing steels and alloys, including titanium alloys; in order to equip mining tools, for metal processing without shavings, fast-wearing parts of machines, appliances and accessories; drawing, calibration and pressing bars and tubes from steel non-ferrous metals and their alloys; fast-wearing parts of machines, devices and measuring instruments, operating at low impact loads [1-4].

High speed steel brand R6M5 is used for production of all kinds of cutting tools, used in the processing of carbonaceous alloy structural steel, thread-cutting tools, tool, working with shock loads, powder brand PR 10R6M5-D for shot blasting [5-7].

Due to the wide application of tungsten-containing alloys in industry, the actual problem is the processing of their waste into the industrial used powders. One of the promising methods of processing of any conductive material, including tungsten alloys waste, in the powder is the electroerosion dispersion method. This method differs by low cost of electricity and the lack of environmental pollution (EED) [8-10].

The aim of the research was to conduct X-ray microanalysis of sintered samples from tungsten-containing powders [11].

2. MATERIALLY AND METHODS

Isostatic press EPSI CIP 400-200 * 1000Y was used to obtain the compacted samples.

In the first step of pressing the powder was placed in a flexible rubber mold and pre-compacted manually to a density 3,1847 g/cm^3. Next samples were placed in the working chamber of press at 18 °C, the pressure was pumped to a desired value, and the sample was held at pressure for 2 minutes, after which the pressure was released to atmospheric pressure and compacted samples were removed from the rubber mold.

Isostatic pressing was performed at a pressure of 250 MPa. Compacted samples were baked in an oven Nabertherm VHT 8/22 for 2 hours at a temperature 1250 ºC in argon.

X-ray microanalysis of sintered samples was performed by energy-dispersive X-ray analyzer of firm EDAX, integrated in the scanning electron microscope «QUANTA 200 3D», spectra of the characteristic X-ray radiation were obtained at different points on the sample surface and transverse sections.

Under X-ray microanalysis need to understand the definition of the elemental composition of microscopic objects, according to excitation of characteristic x-rays in they. For the analysis of the characteristic spectrum in the X-ray microanalysis (XRMA) two types of spectrometers are used (no-crystal or with crystal-analyzer), as a base for XRMA electron-optical system of a scanning electron microscope serves.

In the interaction of the electron probe with a sample, one of the excited signals is X-rays, that can be divided into: the characteristic and braking.

The braking X-ray emission arises owing to inhibition of the primary electrons in an electric (Coulomb) field of atoms in an analyzed material. The kinetic energy of the primary electrons in this case partially or totally converts into the energy of X-rays. Accordingly, the emission has a continuous spectrum with energy from zero to the incident electron energy and therefore it is also called the continuous X-rays. In X-ray microanalysis braking radiation is undesirable, as a main contributor to the increasing levels of background and can't be ruled out.

With the penetration of the primary electrons in the sample, they are inhibited not only by the electric field of atoms, but also a direct collision with the electrons of atoms in the material. A result of this the primary electrons can knock electrons from the inner K-, L-, or M-shells, leaving the atom of the sample in energetically excited state. The resulting vacancies are filled by transfer of electrons from a higher energy levels. Atom goes to the ground state, the excess energy is released in the form of X-ray photon. Because the energy of the arising quantum depends only on the energy of electron levels involved in the process, and they are specific to each item, characteristic X-rays arises. So each atom

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has a very definite finite number of levels, between which transitions are possible only to a certain type characteristic X-rays gives a discrete line spectrum.

Analysis of the distribution of elements can be made in a qualitative, semiquantitative and quantitative manner. Qualitative analysis determines the type of elements that are part of the test sample area. If a sample has several phases (sites), the chemical composition of which is unknown, a qualitative analysis is performed for each phase. Qualitative analysis is commonly used to determine the nature of the distribution of elements according to thin section area. After a qualitative analysis a quantitative analysis is often carried out in selected points, according to the received data software allows to determine type of phase, based on its chemical composition. Semiquantitative analysis is realized, if it is required to define distribution of elements along the lines (linear analysis). Linear analysis is carried out by step scanning, ie, by sequential analysis at individual points. Thus, the quantitative determination out of concentrations of the elements with specified accuracy is carried.

In case of contact of the electron beam on the sample some electrons can escape from the surface of the sample, as a result of their interaction with the crystal lattice of the sample. Backscattered electrons on the fluorescent screen create a picture called a picture of backscattered electrons or Kikuchi lines. The diffraction of back-scattered electrons allows to receive information about the texture and orientation of the crystal samples grains, to conduct mapping of the crystal lattice orientations (i.e., the distribution of orientations on the sample). The diffraction of backscattered electrons also allows to carry out an analysis of the microstructure defects, allows to carry analysis of phases that make up a solid body, to allocate grains and determine their boundaries, to analyze the homogeneity of matter to carry out microstrain and microstrain analysis. If necessary, such analysis is possible in comparison with the images of secondary and backscattered electrons, in the characteristic X-ray (composition of elements of interest) of the same areas of sample.

3. EXPERIMENTAL RESULTS AND DISCATIONS

The points in Figure 1 correspond to the spectrum of the characteristic X-rays. Each chemical element in the spectrum corresponds to the peak of a certain height.

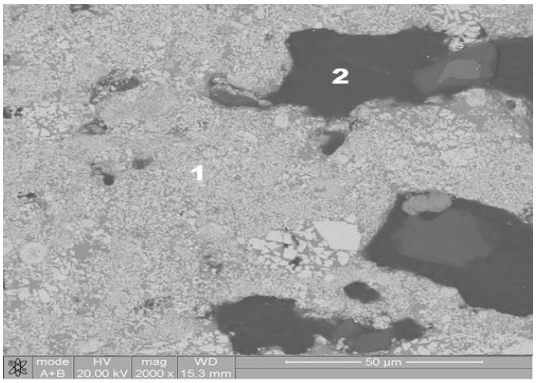

Fig. 1 – The points of conducting X-ray microanalysis of sintered samples from a tungsten powders

The elemental composition and microstructure of sintered samples from tungsten powder are shown in Figures 2, 3 and in Tables 1 and 2.

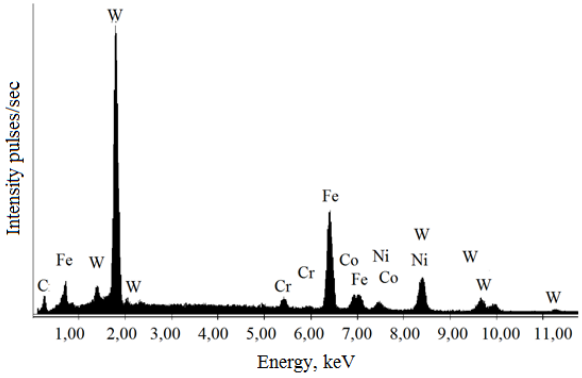

Fig. 2 – The elemental composition at the point 1

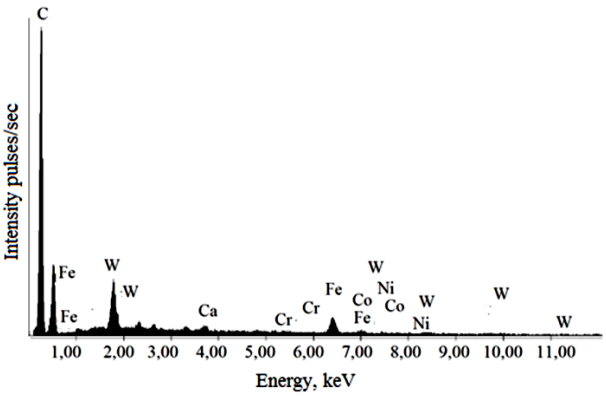

Fig. 3 – The elemental composition at point 2

Table 1 – The elemental composition of sintered samples from tungsten powder at point 1

Element	Mass fraction,%	The atomic fraction,%
C	7,02	38,07
Cr	1,95	2,44
Fe	27,02	31,51
Co	4,00	4,42
Ni	3,06	3,39
W	56,95	20,17
Total	100,00	100,00

Table 2 – The elemental composition of sintered samples from tungsten powder at the point 2

Element	Mass fraction,%	The atomic fraction,%
C	80,81	96,62
Ca	0,96	0,34
Cr	0,51	0,14
Fe	6,71	1,72
Co	1,08	0,26
Ni	0,82	0,20
Total	100,00	100,00

Thus, X-ray microanalysis allows to determine elemental composition of sintered samples from tungsten powders. It was established experimentally that the

basic elements in the composition of sintered samples from tungsten powder are carbon, iron, tungsten and cobalt.

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