

## Short Communication

### Investigation of the Phase Composition of Samples Sintered from Tungsten-containing Composite Micro - and Nanopowders

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Presents the results of research of phase composition of samples sintered from tungsten-containing composite micro- and nano-powders obtained by mixing a powder VK8 (70 %) and PRS powder (30 %) obtained in kerosene lighting. It is established that the main phase of the sintered sample are the WC and Fe<sub>3</sub>C.

**Keywords:** Electroerosion powders, X-ray crystallography, Kerosene lighting.

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## 1. INTRODUCTION

Sintered hard alloys have a number of very valuable properties that make them effectively used in many fields of technology. The basic of these properties is the high hardness (86 ... 92 HRA), combined with high resistance to frictional wear as at metal and at non-metallic materials (rock, glass, wood, plastics, etc.) [1-3].

The ability of alloys to preserve in a large extent these properties at elevated temperatures is also an extremely important characteristic.

One of the main problems of using hard alloys is currently recycling of their wastes and reuse. Numerous attempts to withdraw tungsten from the composition of hard alloys, because of its high cost, did not end successfully, as none of the refractory compounds provides so high strength characteristics. Therefore the problem of recycling wastes of hard alloys are currently very relevant [4, 5].

Powders of high-speed steels are used for the manufacture of cutting and forming tools by hot compaction. The most effective use of high-speed steels powder for the manufacture of large sizes tools, small tools, tool, working in modular machines and machining centers, bimetallic tool.

One of the promising methods for producing of micro-powders and nano-metric fractions from almost any conductive material, including a hard alloy, characterized by a relatively low energy consumption, safety and environmental cleanliness of the process, is the method of dispersing the EDM (EED) [6-9].

The aim of this work was to conduct X-ray diffraction (XRD) of sintered patterns from powder, obtained by electroerosion dispersing of tungsten wastes in lighting kerosene.

## 2. MATERIALLY AND METHODS

For obtaining of powder from tungsten wastes by the electroerosion dispersion in a lighting kerosene setting for EED of conductive materials was used, developed by the authors [6]. When receiving of the powder from waste of brand VK8 the following setup switches were used: capacity of discharge capacitors 65 uF, voltage 150 V, the pulse frequency of 175 Hz.

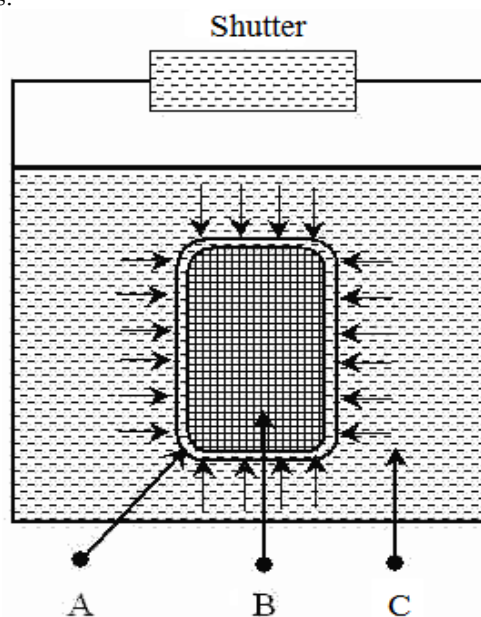
When receiving of the powder from the high-speed steel waste (HSS) of brand R6M5 the following setup switches were used: capacity discharge capacitors of 55 uF, voltage 200 V, pulse frequency of 100 Hz. As a result of local exposure of short-term electrical discharges between electrodes destruction of waste material has happened with forming of a dispersed powder particles. The resulting powder was mixed in a ratio VK8 – 70 %, HSS – 30 %.

Isostatic press EPSI CIP 400-200 \* 1000Y was used for obtaining the compacted materials.

Isostatic pressing process is shown schematically in Figure 1.

The advantages of the cold isostatic pressing method include:

- uniformity of distribution of pressure and density in the preform by the full (isostatic) compression;
- absence of friction losses and the need for plasticizers;
- absence of warpage during sintering;
- arbitrary ratio of height and cross-section of pre-forms.



**Fig. 1** – Isostatic pressing: A – rubber form, B – compactability preform, C – the working fluid (water)

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<sup>3</sup>. 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 °C in argon.

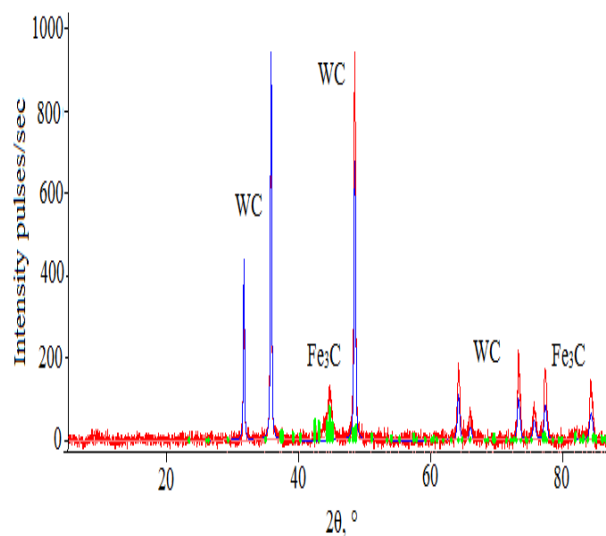
The research of the phase composition of the powders was carried out by X-ray diffraction on the diffractometer Rigaku Ultima IV. Field of application X-ray diffractometer Rigaku Ultima IV: phase analysis of samples, quantitative phase analysis of samples, identification of areas of coherent scattering and microstrain, texture analysis. Features of diffractometer series Ultima IV: radius of the goniometer on the output beam 185 mm, slits of variable width. It allows you to keep constant the irradiated surface of the sample,  $\Theta/\Theta$  goniometer of vertical type for all three configurations, adapted for the installation of a wide range of additional optical components. The new model of high-speed X-ray detector D/teX Ultra, which allows to carry out in 100 times more high-speed measurements, than previous detectors of this company. This detector has a high count of rate, a high energy level of resolution and low noise. Multifunctional console to analyze the textures and residual stresses with rotary tables / Multi purpose attachment MPA-IV  $\chi$  (kai) –  $\varphi$  (phi) - Z stage. Auto-changer samples (10 cuvettes). Software: qualitative and quantitative phase analysis, the diffraction patterns database ICDD PDF-2, crystallinity analysis, residual stress analysis, construction of direct and inverse pole figures, the function of the distribution of orientations.

Specifications. Radiation source: compact with the use of high-frequency inverter, maximum power – 3 kW, voltage in the tube – 20 ... 60 kV, tube current – 2 ... 60 mA, the material of the anode tube – Cu, focus size – 0,4 × 12 mm. Goniometer:  $\Theta/\Theta$  of vertical type, the sample is stationary. Scanning method - independent scanning of each axle  $\Theta_s$  or  $\Theta_d$ ; Scan mode with associated axes  $\Theta_s/\Theta_d$ . Goniometer radius – 185 mm; range of scanning angles in a mode of associated axes  $\Theta_s/\Theta_d$  from – 30 to + 1620 (2 $\Theta$ );  $\Theta_s$  axis from – 1.50 to + 810,  $\Theta_d$  axis from – 950 to + 1200; step of scans to  $\Theta_s$  axis or  $\Theta_d$  0.0001 – 60; in the mode of associated axes 0.0002 – 120 (2 $\Theta$ ). Scanning speed in the mode of associated axes  $\Theta_s/\Theta_d$  0,020 ~ 1000 (2 $\Theta$ ), independently of each axis 0,010 ~ 500; positioning speed 5000/min (2 $\Theta$ ). Slits: with controlled width on the output and the diffracted beam. Two standard sets of Soller slits for work in the focusing geometry and the geometry of the pseudo-parallel beam. Adjustment: fully automatic for goniometer, amplitude discriminator, counter, optical components and other consoles. Detector: scintillation counter with linearity of 700,000 pulses (Standard), a one-coordinate semiconductor detector D/teX Ultra with a sensitivity, exceeding the sensitivity of the scintillation counter in two orders.

The research of the phase composition of the powders was carried out by X-ray diffraction on the diffractometer Rigaku Ultima IV in Cu-K $\alpha$  radiation (wavelength  $\lambda = 0,154178$  nm) with Soller slits. Shooting of the diffraction spectrum for phase analysis carried out by the scheme  $\theta$ -2 $\Theta$  scanning with focusing according to Bregu-Brentano in the angle range 5 ... 100 deg. 2 $\Theta$ . Shooting was in the single spot mode with a step of scanning  $\Delta(2\theta) = 0,02$  deg, a rate of 0,6 deg/min, the operating voltage of 45 kV, 200 mA. To clarify the profile of the experimental radiographs the software package PDXL RIGAKU was used. Subtraction of background was performed by the method of Sonneveld - Visser, smoothing of the experimental profile by the method of Savitsky-Golay, and the separation of components  $ka_1$  and  $ka_2$  by the method of Rachinger. For a description of the diffraction peaks superposition of the Gaussian function and the Lorentz function was used. Approximation of each of the reflexes in the diffractograms of investigated samples by the pseudo - Voigt function allowed to determine the position of reflections exactly, based on the displacement caused by the overlapping of reflections at half maximum of intensity (FWHM) and intensity. The phase composition of the coatings was determined by BD ICDD PDF-2 (2008).

### 3. EXPERIMENTAL RESULTS AND DISCUSSIONS

The results of X-ray diffraction are shown in Figure 2 and Table 1.



**Fig. 2** – The XRD pattern of sintered samples from the powder, obtained by electroerosion dispersing of tungsten waste in a lighting kerosene

**Table 1** – The phase composition of the sintered powder

No	2 $\theta$ , (degree)	Diame-ter, (ang-stroms)	Height, (Hz)	Inten-sity, W (de-gree)	Asymm-etry factor
1	31,563 (7)	2,8322 (6)	328 (18)	0,221 (17)	1,8 (4)
2	35,646 (6)	2,5166 (4)	654 (26)	0,278 (14)	0,78 (12)

3	43,6 (3)	2,072 (11)	7 (3)	1 (2)	1 (4)
4	44,58 (4)	2,0309 (17)	78 (9)	1,1 (3)	1,2 (5)
5	48,329 (6)	1,8817 (2)	751 (27)	0,324 (15)	0,91 (11)
6	64,08 (3)	1,4520 (6)	136 (12)	0,51 (6)	1,1 (4)
7	65,86 (4)	1,4170 (7)	50 (7)	0,51 (12)	1,2 (7)
8	73,19 (2)	1,2921 (3)	151 (12)	0,44 (5)	1,2 (3)
9	75,55 (6)	1,2575 (8)	59 (8)	0,50 (12)	1,0 (5)

10	77,187 (19)	1,2349 (2)	143 (12)	0,42 (5)	0,63 (17)
11	84,14 (3)	1,1496 (3)	106 (10)	0,56 (8)	0,9 (2)

According to results of research, it was found, that the main phases of the sintered samples from powder, obtained by the electroerosion dispersing of tungsten waste in a lighting kerosene, which was mixed at a ratio VK8 – 70 % and the HSS – 30 %, are Fe<sub>3</sub>C and WC.

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