Phase composition of electrospark coatings based on electroerozive powders of micro and nanofractions

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Abstract. The results of the investigation of the phase composition of electrospark coatings, obtained by the method of electrospark alloying at the UR-121 plant, are presented. It is established that the main phases of the electrospark coatings are Fe, W₂C and WC.

1. Introduction
At present, one of the promising methods for obtaining multifunctional coatings on metallic surfaces is the method of electrospark alloying (ESA). The main electrode materials are mainly sintered hard alloys, the cost of which, because of the presence of expensive tungsten, is relatively high. To solve this problem, as an electrode material, it is proposed to use powder of high-speed steels of grade R6M5 mixed with powder of hard alloy VK-8 in various proportions [1-9].

One of the progressive and industrially not used methods of producing micro powder and nanofraction from any conductive material that is characterized by low power consumption and no pollution of the environment is the method of electroerozision dispersion (EED) [10, 11].

The purpose of this work was to study the phase composition of the electrospark coatings obtained with the electrode material from EED powders of high-speed steels of grade R6M5 mixed with hard alloy of grade VK-8 powder in various proportions.

2. Materials and methods
To obtain the micro powder and nanofraction from solid carbide and high-speed steel wastes, a device for EED of conductive materials was used.

In the first pressing step, the powder was placed in a flexible rubber mold and preliminarily manually compacted to a density of 3,1847 g/cm³. The samples were then placed in a working chamber of the press at a temperature of 18 °C; the pressure was pumped to the required value, at this pressure the sample was held for 2 minutes, after which the pressure was dropped to atmospheric and the compacted samples were removed from the rubber mold. The following isostatic pressures of 250 MPa were used. The compacted samples in a furnace “Nabertherm VHT 8/22” were sintered for 2 hours at a temperature of 1250 °C in argon. The first electrode was obtained by mixing the powders in the ratio of VK-8 (90%) + R6M5 (10%), and the second electrode - by mixing powders in the ratio of VK-8 (70%) + R6M5 (30%).

Electro spark coatings formed by such electrodes were obtained on samples of steel 30KhGSA on the UR-121 (manufactured by PELM, Podolsk).
The phase composition of the samples was studied by X-ray diffraction on a Rigaku Ultima IV diffractometer in Cu-Kα radiation (wavelength $\lambda = 0.154178$ nm) using Soller slits. The diffraction spectrum for the phase analysis is sampled according to the 0-2Θ scanning scheme with Breguot-Brentano focusing in the angular interval of 5 ... 100 deg. 2Θ. The shooting is carried out in the point-by-point mode with a scanning step of $\Delta (2\theta) = 0.02$ 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 the 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, the intensity and half the maximum intensity (FWHM). The phase composition of the coatings was determined using the ICCD PDF-2 database (2008).

The field of application of the Rigaku Ultima IV X-ray diffractometer is: phase analysis of samples, quantitative phase analysis of samples, determination of regions of coherent scattering and microstrains, textural analysis.

The peculiarity of the Ultima IV series diffractometer is: the radius of the goniometer is 185 mm on the output beam, the slots are of variable width. It allows one to keep the irradiated surface of the sample unchanged. The Θ/Θ vertical type goniometer for all three configurations, adapted for mounting a wide range of additional optical components.

A new model of high-speed X-ray detector D / teX Ultra, which allows one to carry out measurements 100 times faster compared to previous detectors of this company. It is a detector of high counting speed, high energy resolution level and low noise level. This model includes:

- The multifunctional device, used for analysis of textures and residual stresses with rotary tables (multi purpose attachment MPA-IV χ (kai) - φ (phi) - Z stage).
- Auto-changer samples (10 cuvettes).
- Software: qualitative and quantitative phase analysis, ICDD PDF-2 diffraction chart database, crystallinity analysis, residual stress analysis, construction of forward and backward pole figures, orientation distribution function.

Specifications of the model are as follows:

**Light source:**
- small-sized using a high-frequency converter;
- the maximum power is 3 kW;
- the voltage on the tube is 20-60 kV;
- the tube current is 2-60 mA;
- the tube anode material is Cu;
- the focus size is 0.4x12 mm.

**Goniometer:**
- Θ / Θ vertical type, the sample is stationary;
- the scanning method - independent scanning of each axis Θs or Θd; the scan mode with associated axes Θs/Θd;
- the radius of the goniometer is 185 mm;
- the range of scanning angles in coupled axes mode Θs / Θd from -30 to +1620 (2Θ);
- axes Θs from -1,50 to +810, axes Θd from -950 to +1200;
- scan step for axis Θs or Θd 0,0001 - 60;
- in the coupled axes mode 0,0002 - 120 (2Θ);
- scanning speed in coupled axes mode Θs / Θd 0,020 ~ 1000 (2Θ), independently of each axis 0,010 ~ 500;
- positioning speed 5000/min (2Θ).
Slots:
- with a controlled width for the output and diffracted beam;
- two standard sets of Soller slots for working in focusing geometry and the geometry of a pseudo-parallel beam.

Adjustment:
- fully automatic for goniometer, amplitude discriminator, counter, optical nodes and additional consoles.

Detector:
- Scintillation counter with a linearity of 700,000 imp. (standard);
- one-dimensional semiconductor detector D/teX Ultra with a sensitivity that exceeds the sensitivity of the scintillation counter by two orders of magnitude.

3. The study of the phase composition of electro-spark coatings
The X-ray patterns of the test samples are shown in Fig. 1-3 and in Table 1, 2.

![X-ray pattern of VK90 + HSS10 sample](image)

**Figure 1.** X-ray pattern of VK90 + HSS10 sample

| Name                | Chemical formula | Type of crystal lattice |
|---------------------|------------------|-------------------------|
| Iron                | Fe               | cubic                   |
| Wolfram carbide     | W₂C              | hexagonal               |
| Wolfram carbide     | WC               | hexagonal               |

**Table 1.** Phase composition of the surface of sample VK90 + HSS10

Investigations of the phase composition of the electrospark coating, which was obtained using the first electrode from a sintered EED powder mixed in the ratio of VK-8 (90%) + R6M5 (10%), showed that the main phases in the coating are Fe, W₂C and WC.
Table 2. Phase composition of the surface of sample VK70 + HSS30

| Name                        | Chemical formula | Type of crystal lattice |
|-----------------------------|------------------|-------------------------|
| Iron                        | Fe               | cubic                   |
| Wolfram carbide             | W2C              | hexagonal               |
| Wolfram carbide             | WC               | hexagonal               |

Investigations of the phase composition of the electrospark coating, which was obtained using the second electrode from a sintered EED powder mixed in the ratio of VK-8 (70%) + R6M5 (30%), showed that the main phases in the coating are Fe, W2C and WC.

Figure 2. X-ray pattern of sample VK70 + HSS30

Figure 3. Overlapping of diffractograms of samples VK90+HSS10 and VK70+HSS30.
4. Conclusion
Based on the performed X-ray diffraction microanalysis of electrospark coatings obtained with the electrode material from electroerosive powders of micro- and nanofraction of high-speed steel grade R6M5 mixed with VK-8 carbide powder in various proportions, it was established that the main phases of the coatings are Fe, W2C and WC.

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