The x-ray diffraction study of the composition of the powder materials from the waste steel X13

E.V. Ageeva¹, S.V. Khardikov²*, A. N. Novikov³

¹Southwest state University, Kursk, 305040, Russian Federation.
²Southwest state University, Kursk, 305040, Russian Federation.
³OSU named after I. S. Turgenev, Orel, 302026, Russian Federation.

*hardikov1990@mail.ru

Abstract. To restore and protect the surfaces of parts using a layer of molten metal, various methods of surfacing are used, differing from each other by melting methods and compositions of the welding medium: electric arc, flame, plasma, laser, induction, etc. With this technology, metals of various chemical composition, including copper, bronze, cast iron, as well as nickel, cobalt and chromium alloys can be deposited on the working surfaces of steel structures. There are several ways to obtain such powder materials. The most promising method for obtaining powder materials is the electro-erosion dispersion method (EED), which is distinguished by its ecological purity of the process and relatively low energy expenditure. To study the structure of the particles of the obtained powders, their X-ray structural analysis was performed on a RigakuUltima IV X-ray diffractometer.

1. Introduction

Metal surfacing is used to restore the geometry of worn-out parts of machines and mechanisms, to form reinforcing metal layers on the surface of products and to create bimetallic structures. At its core, surfacing is one of the types of welding technologies, since it is based on the same physical and technological principles as traditional types of welding. To restore and protect the surfaces of parts using a layer of molten metal, various methods of surfacing are used, differing from each other by melting methods and compositions of the welding medium: electric arc, flame, plasma, laser, induction, etc. Using this technology, metals of various chemical composition, including copper, bronze, cast iron, as well as nickel, cobalt and chromium alloys can be deposited on the working planes of steel structures [1–6].

The technology of surfacing allows to achieve not only reliable adhesion of the applied metal to the base, but also to obtain the required physical and chemical characteristics of the deposited layer. The first is achieved by the qualitative preparation of the base product and the exact observance of technological regimes, and the second by the correct selection of welding materials. The essence of the surfacing consists in uniform deposition of narrow strips of molten metal on the surface of the part so that they merge into a continuous metal layer of a given thickness. The properties of the resulting coatings are directly dependent on the quality of the used materials. One of these materials is chromium steel. Powders with a high content of chromium can be used to obtain wear-resistant and heat-resistant coatings.
There are several ways to obtain such powder materials. The most promising method for obtaining powder materials is the electro-erosion dispersion method (EED), which is distinguished by the ecological purity of the process and relatively low energy expenditure [7–10].

For the development of technologies for the practical application of powder, obtained from waste, and evaluating the effectiveness of its use, complex theoretical and experimental studies are required.

2. Materials and techniques

To study the structure of the particles of the obtained powders, their X-ray structural analysis was performed on a Rigaku Ultima IV X-ray diffractometer.

Application area:
- phase analysis of samples;
- quantitative phase analysis of samples;
- determination of areas of coherent scattering and microstrain;
- texture analysis.

Features of the diffractometer series Ultima IV:
- goniometer radius - 185 mm on the output beam;
- slits of variable width, which allow to keep the irradiated surface of the sample unchanged;
- Θ / Θ vertical-type goniometer for all three configurations, adapted to install a wide range of additional optical components;
- high-speed X-ray detector D / teX Ultra, which has a high counting rate, high energy level of resolution and low noise.

Multi-purpose attachment for texture and residual stress analysis with turntables Multi purpose attachment MPA-IV χ (kai) - φ (phi) - Z stage includes:
- automanager of samples (10 ditch);
- software: qualitative and quantitative phase analysis, ICDD PDF-2 diffractogram database, crystallinity analysis, residual stress analysis, construction of direct and reverse pole figures, orientation distribution function.

Specifications radiation source:
- small-sized using a high-frequency converter, maximum power - 3 kW;
- tube voltage - 20–60 kV;
- tube current - 2–60 mA;
- tube anode material – Cu;
- focus size - 0.4 x 12 mm.

There is a vertical type goniometer with a fixed sample. The Rigaku Ultima IV X-ray diffractometer provides independent scanning of each Θs or Θd axis or in scanning mode with associated Θs / Θd axes.

Goniometer settings:
- radius - 185 mm;
- the range of the scan angle in the associated mode of the Θs / Θd axis is from –30 to +1620 (2Θ);
- deviation of the axis from –1.50 to +810, ocmath axis from –950 to +1200;
- scanning step along the Θs or Θd axis - 0.0001–60;
- in the mode of coupled axes - 0.0002–120 (2Θ).

Scan speed in the associated axis Θs / Θd mode is 0.020 ~ 1000 (2Θ), independently of each axis is 0.010 ~ 500.

Two standard sets of Soller slits for working in focusing geometry and pseudo-parallel beam geometry. The device has fully automatic adjustment for goniometer, amplitude discriminator, counter, optical nodes and additional devices. The detector is a scintillation counter with linearity of 700,000 pulses (standard), a uniaxial semiconductor detector D / teX Ultra with a sensitivity exceeding the sensitivity of the scintillation counter by two orders of magnitude.

The powder material of the test sample is poured into a cuvette with a diameter of 20 mm and a depth of 0.5 mm and pressed so that the sample surface is parallel to the edges of the cuvette. The
cuvette is placed in the holder, as a result of which, on an adjusted goniometer, the sample surface is aligned with the focusing plane.

Survey parameters:
- range 10–95 degrees 2Θ;
- step - 0.020 degrees;
- speed - 1 degrees / min;
- operating voltage - 40 kV;
- current - 40 mA.

Diffraction pattern processing:
- PDXL program;
- smoothing - Savitsky-Golay method;
- background calculation - Sonneveld-Visser method;
- peak search - peak top method.

3. Conduct X-ray analysis

The properties of powder particles, on which their area of application depends, are largely determined by their structure.

The results of X-ray diffraction analysis of powders, obtained by the method of EED from chrome-containing wastes, are presented in Figure 1 and in Table 1.

On the experimental radiograph there are strong reflexes corresponding to:
- phase Fe₃O₄;
- phase Cr;
- phase Fe₃C.

![Figure 1. The powder XRD pattern.](image)
Table 1. The x-ray maxima of the powder in lighting kerosene

| 2θ, (degree) | Height (Hz) | Intensity W (degree) | Size (angstroms) |
|--------------|-------------|---------------------|------------------|
| 19.67(3)     | 113(31)     | 0.10(8)             | 4.509(8)         |
| 29.22(3)     | 188(40)     | 0.18(8)             | 3.054(3)         |
| 35.50(4)     | 343(54)     | 0.55(12)            | 2.527(3)         |
| 43.09(7)     | 146(35)     | 1.0(4)              | 2.098(3)         |
| 44.523(16)   | 773(81)     | 0.42(7)             | 2.0333(7)        |
| 57.387(16)   | 118(32)     | 0.36(17)            | 1.6044(4)        |
| 62.8(2)      | 73(25)      | 0.8(5)              | 1.479(5)         |
| 64.929(10)   | 181(39)     | 0.17(6)             | 1.4350(2)        |
| 82.37(13)    | 118(32)     | 0.7(4)              | 1.1698(15)       |

4. Conclusion
The phase composition of the powder, obtained by the method of EED, is determined by the nature of the WF: the presence of carbon, the dielectric constant and boiling point. Dispersion of waste alloys of ball-bearing steel in lighting kerosene led to carbon enrichment and the formation of the Fe₃C phase.

Thus, the phase composition of powders, obtained by the method of EED, is influenced by the complex of thermochemical properties of working fluids. This can determine the choice of WF to obtain a given composition of powders.

5. References
[1] Lazarenko B R and Lazarenko N I A. p. 70000 USSR, B 22f, 09/00 A method for producing powders and a device for its implementation.- No. 1371/321510; declared 04/01/1943; publ. 09/23/1964, Bull. 22, 2
[2] Karlsson J, Snis A, Engqvist H and Lausmaa J 2013 J. of Mat. Proc. Tech. 213 2109-18
[3] Janitor M I2006 Development of physico-chemical and technological fundamentals of the processing of tungsten-cobalt hard alloy by electroerosive dispersion: diss. ... cand. tech. Sciences, Dvornik Maxim Ivanovich (Khabarovsk) p 116
[4] Ageev E V 2011 Hard. tech. and coat. 6 8-14
[5] Latypov R A, Latypova G R, Ageev E V and Burak P I2013 IS/5 80-86
[6] Ageev E V2011 Electromet. 10 24–27
[7] Ageev E V, Kirichek A V, Altuhov AYuand AgeevaEV2014J. Nano- Electron. Phys. 6 3, 03001.
[8] AgeevaEV, AgeevEV, HoryakovaNMandMalukhovVS2014J. Nano- Electron. Phys. 6 3, 03011.
[9] AgeevaEVAgeevEV KarpenkoVVyandOsmininaAS2014J. Nano- Electron. Phys. 6 3, 03049.
[10] AgeevEV The patent 2449859, the Russian Federation, C2, B22F9/14. No 2010104316/02; appl. 08.02.2010; publ. 10.05.2012. - 4 p.

Acknowledgments
The work was supported by a scholarship of the President of the Russian Federation to young scientists and graduate students (SP-945.2019.1).