Methodical complex for investigation dependencies between micro structure and macro properties of materials and operating characteristics of products

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Abstract. The work presents a methodical complex of studies of base metal and its coating composition, that allows to study the change in structural characteristics of material at micro-level, mezzo-level and macro-level. The effect of changes in structural state on the properties of coatings and base materials during treatment of cast iron parts working surfaces by air plasma spraying (APS) was established. Physico-Mechanical properties of coated samples and the features of their structure formation has been evaluated during experiments. An optimal composition of a powder mixture containing self-fluxing solid alloys and wear-resistant fillers, which provides enhanced performance in conditions of hydroabrasive wear is proposed. It is established that plasma spraying of the coating (65% PR-NH17SR4 + 35% KHNp-30) with subsequent heat treatment and reflowing leads to an increase in wear resistance up to 1.5-2 times.

1. Introduction
One of the priority areas of materials science and surface engineering is the establishment of dependencies between changes in structure of surface and near-surface layers of the material and its performance properties when applying various types of functional coatings.

Effective development of this area of research is possible only with obtaining synergetic effect by use of complex research methods and subsequent application of integrated results in applied tasks of high-tech industries, for example, in the process of extraction and processing of mineral raw materials using hydrottransport lines.

Reliability of hydraulic equipment has recently received considerable attention due to the need for increased productivity and service life of pumps produced in Russia and used in the mining and processing industry. The main factor influencing resource of soil pumps required at mining and processing plants is the hydroabrasive wear of parts, as a result, most of a broken parts that needs replacement are impellers, throatbushes, back liners, armored washers and casings (shells) [1, 2].

In practice, replacement of critical components that have served recommended service life, due to the scarcity of spare parts for soil pumps and relatively high labor input for their replacement, is usually not performed. In this regard, some pumps are running with reduced performance, which damages production efficiency.

There are known studies [3, 4 and 5] that suggested various options to increase a life of soil pumps running under conditions of hydroabrasive wear. The proposed solutions included application of various types of spraying, abrasive compounds and surfacing to a pump housing and impeller.
However, this increases roughness of a surface and unevenness of wear. As a result, imbalance of a pump impeller and decreased effectiveness can occur. At the same time, studies of wear-resistant coating, reinforced with composite materials in order to increase wear resistance of cast iron parts are rare.

In connection with the foregoing, introduction of effective ways and means to reduce the amount of hydroabrasive wear and to maximize wear and impact resistance is an important topic in the industry, since this means can significantly improve durability and reliability of machinery. Urgency of this problem confirms the need for comprehensive studies on materials of wear-resistant and impact-resistant coatings formed on wearable parts of pumps.

2. Problem statement
Modern classification of the hierarchy of material structures divides them into the following groups:
– macro-level, i.e. grain sizes, grain orientation, various large inclusions, fracture type;
– mezzo-level, which includes a structure inside grains, dislocations and their ensembles, disclinations, polygons;
– micro-level (otherwise x-ray structure), i.e. point defects, type of crystal lattice, micro-deformations, etc.

In this regard, for a comprehensive study of the effect that structural characteristics have on properties of parts with coatings, it is necessary to conduct a sound research. Methods that are proposed in this work are considered for components made of chromium-nickel wear-resistant cast iron grade WRCIH28N2, which is currently used for the manufacture of fast wearing parts of pumps GrAT85/40. In hydroabrasive wear conditions, wear resistance of surfaces will be higher when more carbides are contained in an alloy. To increase wear resistance of white cast iron, in addition to presence of carbides, it is necessary to provide high strength of the metal substrate [6, 7]. It is known that the wear rate of surfaces is maximal in samples with pearlitic matrix and minimal in samples that have undergone bainite heat treatment [8, 9]. These results made it possible to make an assumptions about potential improvement in the characteristics of cast iron WRCIH28N2 through use of heat treatment technologies after air-plasma spraying.

This work focuses on presentation and implementation of a set of research methods, including structure and properties change analysis at micro-level, mezzo-level and macro-level, tested on materials and products in demand by industry. Processes of coatings formation and altered surface layer on WRCIH28N2 samples; composition, structure and properties of coatings; physical and mechanical properties of WRCIH28N2 before hardening and after hardening as well as comparison of efficiency of properties of the changed surface layer after plasma spraying treatment of cast iron samples with subsequent reflow of coating during heat treatment process were subject of research.

To achieve this goal, it is necessary to solve following tasks:
– carry out a comprehensive study of coatings and near-surface layers at various hierarchical structural levels formed by plasma spraying followed by heat treatment;
– justify optimal composition of powder mixture for an air-plasma application of wear-resistant coating, which can be subsequently melted;
– testing effectiveness of the coating composition based on self-fluxing solid alloys under conditions of shock loads and hydroabrasive wear.

3. Materials and methods
As a sample material were used parts made of chromium-nickel wear-resistant cast iron WRCIH28N2, cut from a plate part of worn-out pump (GrAT85/40) throatbush. Development of optimal application process and composition of composite wear-resistant coatings was carried out on the following powder mixtures:
– 65% PR-NH17SR4+35% KHNp-30;
– 50% PR-NH7SR3+50% KHNp-30;
– 75% PR-NH17SR4+25% FBH6-2;
– 50% PR-NH7SR3+50% FBH6-2.
These ratios of self-fluxing solid alloys (SFSA) and wear-resistant fillers provide high-quality self-fluxing properties of the compositions. Powder mixtures selected for research were sprayed onto the samples of basic material using modernized Kiev-7 unit with parameters given in Table 1. Process of coating formation in plasma spraying is carried out by treating surface with ions during condensation, deposition of high-energy ions, as well as atoms and molecules involving plasma chemical processes [5, 10].

Table 1. Sputtering parameters for samples at the «Kiev-7» unit.

| Mode number | Parameter                          | Value          |
|------------|------------------------------------|----------------|
| 1          | Plasma arc current, A              | 200-220        |
| 2          | Voltage, V                         | 200-210        |
| 3          | Gas consumption, m³/hour           | Air 4-4.2, propane-butane gas 0.2-0.3 |
| 4          | Powder consumption, kg/hour        | 3.5-4          |
| 5          | Sputtering distance, mm            | 180-200        |

In connection with the decision to combine process of coating reflow caused by air-plasma spraying of self-fluxing mixtures with standard heat treatment of cast iron WRCIH28N2 (Table 2), a chromium carbide powder clad with nickel was chosen as a wear-resistant filler. The choice is due to the fact that tungsten carbide, oxidized in air while heated to 540°C, burns out during tempering: T = 540-560°C, t = 3 hours, while chromium carbide is stable at temperatures up to 870°C, and the cladding sheath of Ni protects it from oxidation and during air-plasma spraying.

Table 2. Cast iron WRCIH28N2 heat treatment modes.

| Heat treatment type | Heating temperature, °C | Heating rate, °C/hour | Holding time, hours | Cooling medium | Hardness, HRC |
|---------------------|-------------------------|------------------------|---------------------|----------------|--------------|
| Hardening           | 1060-1100               | 50-100                 | 1-2                 | Air            | 52-54        |
| Tempering           | 540-560                 | 50-100                 | 3-4                 | Air            | 58-60        |

Implementation of research was carried out using the following methods and techniques. After polishing the samples and making cross microsections, macro hardness and micro hardness was determined on a Rockwell instrument and PMT-1M micro hardness tester respectively. Metallographic study of coating structure and base metal (WRCIH28N2) were carried out on a LaboMet-3 optical microscope. Diffraction patterns were recorded on a D8 Advance X-ray diffractometer in Cu-Kα radiation with the following parameters: scan step 0.05°, signal accumulation time 2 sec/point, voltage and filament current of 40 kV and 40 mA, respectively. Decoding of the obtained diffractograms was carried out using the 2006 ICDD PDF-2 powder diffraction database. The lattice parameters were refined in the program TOPAS 3.0 (Bruker). Test method for wear resistance in shock abrasive wear with loose abrasive particles corresponds to GOST 23.207-79, which most closely matches conditions of the tasks set. Tests were carried out for 21 hours (3 working shifts) in conditions close to operational ones. Abrasive material was granite crushed stone granulated up to 10 mm and hardness 7-8 units according to Mohs scale. For faster tests, its concentration in water was 40-50% (volumetric). Weighing of samples before and after the tests (ΔG) was carried out on the electronic scales MK-15.2-A21 up to second decimal place, and measurement of the wear depth (Δh) was performed by a special micrometer.

4. Results and discussion
Surface structure of cast iron samples after etching with nital are shown in figures 1-4.
It is established that the macro hardness of base material (cast iron WRCIH28N2) after heat treatment is increased by 5-8 HRC in comparison with the cast state due to the diffusion hardening of residual austenite of metal substrate in bainitic-troostite transformation that occurs during tempering. This is evident when comparing microstructures presented in fig. 1 and 2, and is also confirmed by results of X-ray diffraction analysis, which revealed by a marked decrease in the percentage of gamma-phase and an increase in the distorted (with increased lattice parameters) alpha phase of iron (see fig. 5). In addition, there are a significant amount of carbides (Cr, Fe)\textsubscript{7}C\textsubscript{3} and individual chromium carbides Cr\textsubscript{7}C\textsubscript{3} (see fig. 6).

![Figure 1. Micro structure of cast iron WRCIH28N2 in the state of delivery, x200](image1)

![Figure 2. Micro structure of cast iron WRCIH28N2 after quenching and annealing, x200](image2)

![Figure 3. Microstructure of coating 65% PR-NH17SR4+35% KHNp-30 after melting, x200](image3)

![Figure 4. Microstructure of coating 65% PR-NH17SR4+35% KHNp-30 after melting (KHNp-30 part), x200](image4)

The hardness of composite coatings containing KHNp-30 is somewhat less than the hardness of initial SFSA, since in the firing process, coagulation (coarsening) of carborides takes place due to a diffusion yield from lattice solid solution and the eutectic of chromium and boron. As a result, the hardness of metal substrate decreases, although micro hardness of the carboride phase remains high (HV 1300-1400), and individual Cr\textsubscript{7}C\textsubscript{3} chromium carbides reach HV2000 (fig. 3, 4). Mixture with FBH6-2 do not contain such solid phases, but the hardness of their lattice solution, while decreasing during tempering, remains quite high (HV 900-1000).

In the structural-phase analysis of base material and coatings, phase composition of the initial cast iron was investigated at the first stage. It is established that in the cast state the structure of investigated
cast iron mainly consists of carbide of approximate composition $(\text{CrFe})_7\text{C}_3$. In addition, there is a phase of ferrite ($\alpha\text{-Fe, bcc lattice}$), residual austenite ($\gamma\text{-Fe, fcc lattice}$) and chromium carbide phase of $\text{Cr}_7\text{C}_3$.

Further, the change in the structure of initial cast iron after heat treatment was investigated. A detailed study of X-ray diffraction patterns in the region of the 111 $\gamma$-Fe (austenite) and 110 $\alpha$-Fe (fig.5) peaks indicated that integrated intensity ($I$) of the peak 110 for samples 3 and 4 increased in comparison with the original sample 1. Apparently, this is due to an increase in the content of the ferrite phase in the samples after heat treatment.

![Graphical representation of X-ray diffraction patterns](image)

**Figure 5.** Detailed study of the X-ray diffraction patterns of cast iron samples: 1 – initial, 2 – original with back coating (65% PR-NH17SR4+35% KHNp-30); 3 – sample 2 after heat treatment; 4 – initial sample with back coating and after heat treatment (50% PR-NH7SR3+50% KHNp-30)

According to the diffraction data, the parameter of the ferrite lattice was determined (table 3).

| Mode number | $a$, Å |
|-------------|--------|
| Cast. data  | 2.867  |
| 1           | 2.872 ± 0.001 |
| 2           | 2.872 ± 0.001 |
| 3           | 2.874 ± 0.001 |
| 4           | 2.875 ± 0.001 |

Table 3. The lattice parameter $\alpha\text{-Fe}$ (P.G. 229, Im3m)
After a temperature treatment, a slight increase in the parameter of the ferrite lattice is observed, which may be caused by a slight composition change due to carbon atoms entering into the structure of ferrite formed in the bainitic-troostite transformation of residual austenite ($\gamma$-Fe).

During the analysis of phase composition of samples 2, 3 and 4 with coating (fig. 6) was established a presence of phases: Ni, Cr$_7$C$_3$ and Cr$_3$C$_2$. For sample 4, a slight increase in the intensities ($I$) of Cr$_3$C$_2$ carbide peaks (2$\theta$: 45-47 °) was observed, which may be due to an increase in the amount of peaks in the initial coating.

According to X-ray diffraction data, lattice parameter of the main phase of the coating, Ni, was calculated (table 4). It is established that the Ni lattice parameters increases after heat treatment of sample 4 with a coating 50% PR-NH7SR3+50% KHNp-30. With a coating composition of 65% PR-NH7SR4+35% KHNp-30, such a change is not observed.

**Table 4.** Ni lattice parameters (P.G. 225, Fm3m)

| Mode number | a, Å       |
|-------------|-----------|
| Cast. data  | 3.535     |
| 1           | -         |
| 2           | 3.538 ± 0.001 |
| 3           | 3.548 ± 0.001 |
| 4           | 3.538 ± 0.001 |

Tribological tests were performed on six types of samples of basic material and coating, the numbering and composition of which are given in table 5. Also in the table are values of hardness, weight $\Delta G$ and linear $\Delta h$ wear of samples, coefficients of weight ($K_1$) and linear ($K_2$) wear resistance, defined as ratio of reference sample wear amount (main material in the delivery state) to wear value of the test sample:

$$K_1 = \frac{\Delta G_{\text{ref}}}{\Delta G_{\text{tes}}}; \quad K_2 = \frac{\Delta h_{\text{ref}}}{\Delta h_{\text{tes}}}.$$
Table 5. Test results of base material and wear-resistant coating samples under conditions of hydroabrasive wear.

| Material                  | Coating thickness, mm | ΔG  | κ₁  | Δh  | κ₂  | Hardness HRC |
|---------------------------|-----------------------|-----|-----|-----|-----|--------------|
| WRCI H28N2, casting       | –                     | 6.1 | 1.00| 2.2 | 1.00| 48-52        |
| WRCI H28N2, heat treatment| –                     | 4.5 | 1.35| 1.6 | 1.35| 56-60        |
| 65% PR-NH17SR4+35%KHNp-30| 1.5                   | 3.6 | 1.70| 1.1 | 2.00| 54-56        |
| 75% PR-NH17SR4+25%FBH6-2  | 1.5                   | 4.3 | 1.40| 1.3 | 1.70| 51-53        |
| 50% PR-NH7SR3+50%KHNp-30  | 1.5                   | 5.0 | 1.20| 1.65| 1.34| 47-49        |
| 50% PR-NH7SR3+50%FBH6-2   | 1.5                   | 5.7 | 1.08| 1.7 | 1.30| 42-54        |

The results of the tests showed that coating 65% PR-NH17SR4+35% KHNp-30 has the highest wear resistance, second highest result has coating 75% PR-NH17SR4+25% FBH6-2, and the third one is basic material WRCI H28N2 after heat treatment. Thus, 65% PR-NH17SR4+35% KHNp-30 together with hardened cast iron WRCI H28N2 even under the hardest allowed wear conditions can increase service life of components by 1.5 and more times.

5. Conclusion

1. Obtained results allow to speak about the effectiveness of proposed set of methods for studying structure and properties of materials and a possibility of obtaining due to it not only new knowledge about the structure and properties of materials but also new technological solutions for materials science in the manufacturing process of a products.

2. A technology for applying wear-resistant coatings has been developed and comparative studies of the physical and mechanical properties of base material and wear-resistant coatings samples have been carried out. Introduction of the presented technology in practice allows to increase the life of parts and components up to 1.5-2 times.

3. An optimal in terms of their characteristics set of wear-resistant coatings have been choosen. They are arranged in the following order: the main material WRCI H28N2 after heat treatment, the coating 75% PR-NH17SR4+25% FBH6-2, the coating 65% PR-NH17SR4+35% KHNp-30.

References

[1] Penkin N S, Kapralov E P and Malyaroy P V 1992 *Improvement of wear resistance of mining and processing equipment* (Moscow: Nedra) p 264

[2] Povetkin V V, Tatybaev M K, Alpeisov A T, Bektibay B J and Tkachenko D E 2015 Main properties of ground pump operation and wear of working parts *Collection of scientific papers on the results of the international scientific and practical conference "Actual issues of technical sciences in modern conditions”* pp 113–119

[3] Lebedev V A and Mozok V M 2013 Increasing of life cycle of wear parts due to heterogeneous surfacing (Review of developed and implemented technological solutions) *Heavy Engineering* 11 pp 10-14

[4] Brusova O M 2014 To the issue of increasing service life of ground pumps *Vestnik of the Perm National Research Polytechnic University. Geology. Oil and gas, mining* 13(10) pp 98–106

[5] Fauchais P and Vardell A 2011 Innovative and emerging processes in plasma spraying: from micro-to nano-structured coatings *J. Phys. D: Appl. Phys.* 44 pp 194011

[6] Petrochenko E V, Molochkova O S and Alehina O N 2017 Effect of boron on the structural-phase state of complex-alloyed white cast irons *Theory and technology of metallurgical production* 4(23) pp 37–40
[7] Kolokoltsev V M, Petrochenko E V and Molochkova O S 2016 Influence of complex V, Cu, Ti and B alloying on structural and phase state, mechanical properties and wear resistance of white cast iron *Cis iron and steel revive* 11 pp 23–29

[8] Emelyushin A N, Petrochenko E V and Nefed’ev S P 2013 Investigation of the structure and impact-abrasive wear resistance of coatings of the Fe-Cr-Mn-Si system, additionally alloyed with nitrogen *Welding international* 2 pp 150–153

[9] Ri Hosen, Ri E H and Teih V A 2000 Influence of alloying elements on crystallization, structure formation and physical and mechanical properties of white cast iron *Foundry* 10 pp 15–17

[10] Zverev E A, Skeeema V Yu, Skeeema P Yu and Khlebova I V 2017 Defining efficient modes range for plasma spraying coatings *IOP Conf. Ser.: Earth Environ. Sci.* 87 pp 082061

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