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Chemical composition, structure and microhardness of multilayer high-temperature coatings

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ABSTRACT

Introduction. Plasma spraying is one of the modern and effective methods for coatings application for various purposes and compositions. With the help of thermal plasma flows, it is possible to spray almost any powder materials (ceramic, metal, metal-ceramic). Plasma spraying of multilayer protective coatings can be successfully used to increase the durability of pierced mandrels, which are the main tool in the production of hollow billets. The purpose of this work is to study the chemical composition, structure, and microhardness of multilayer high-temperature coatings of two different compositions deposited by plasma spraying, which are supposed to be used to increase the durability of pierced mandrels. **Materials and research methods.** The deposition of multilayer coatings of two compositions was carried out on a plasma-powder spraying unit with contact excitation of an arc discharge *UPN-60KM TSP2017*. Coatings were obtained by sequential deposition of three layers with different powder compositions. After deposition of all three coating layers, oxidative annealing was carried out at a temperature of 900°C to form a dense scale layer of $\text{FeO} + \text{Fe}_2\text{O}_3 + \text{Fe}_3\text{O}_4$ on the surface. The chemical composition of the coatings was studied by X-ray microanalysis using a *TESCAN* scanning electron microscope with an *OXFORD* attachment. The microstructure of the coatings was studied using a *NEOPHOT* metallographic microscope. Phase X-ray diffraction analysis was performed on a *SHIMADZU* diffractometer in $\text{K}\alpha$ -chromium radiation. Microhardness was measured on a *LEICA* hardness tester at a load of 50 g. **Results and discussion.** The nature of the distribution of chemical elements over the thickness of the coating, consisting of four layers, is established: an inner metal layer that provides protection against high-temperature corrosion; a transitional metal layer designed to equalize the thermal properties between the layers; α -Fe metal oxide layer and iron oxides and external thermal barrier oxide layer $\text{FeO} + \text{Fe}_2\text{O}_3 + \text{Fe}_3\text{O}_4$. Coatings are characterized by a non-uniform distribution of structural components and microhardness over its thickness. The microhardness of the inner layer reaches 1,400 HV0.05, the transition layer is 800 HV0.05, and the metal oxide layer is 300 HV0.05.

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Introduction

Plasma spraying is one of the advanced and effective methods for applying coatings of various purposes and compositions. By means of thermal plasma flows, it is possible to spray almost any powder materials (metallic, ceramic, cermet, etc.) [1–3]. Plasma spraying for applying self-fluxing powders is the most widely used technology [4, 5]. The self-fluxing process consists in spontaneous removal of oxides from the surface of particles during its fusion. For this to happen, oxides must have low melting temperature,

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low density, and high fluidity. Nickel-based *Ni-Cr-B-Si* alloys are most often used for this purpose; during plasma spraying of the alloys, B_2O_5 - Cr_2O_3 - SiO_2 oxides form a fusible slag, which, when fusing, comes to the surface in the form of a thin glassy coating [6–8].

Coatings applied by plasma spraying have some disadvantages. First of all, its porosity ranges between 8 % and 15% [3–5]. In order to reduce porosity, special plasma surfacing techniques were developed, such as high-speed plasma surfacing, applying multilayer coatings, nanostructuring of sprayed coatings, rare-earth doping, repeated laser surface melting, and a combination of the techniques [9–11]. However, the presence of pores in a coating can be functionally advantageous in some cases. At high operating temperatures, oxygen easily penetrates through the pores deep into the coating, thus causing massive oxidation throughout its thickness, with the formation of a protective film of Cr_2O_3 or Al_2O_3 oxides [12–14]. A combination of several differently applied protective layers is successfully used to reduce the disadvantages of coatings [15–19]. When a single application method is used, the layers may differ in the chemical and phase composition and perform different protective functions. In fact, functional gradient coatings are formed.

Plasma spraying of multilayer protective coatings can be successfully used to increase the durability of piercing mandrels, which are the main tools used to produce hollow billets [20–22]. During operation, piercing mandrels are affected by cyclic heating (20 to 1100 °C) and by the pressure of the metal being deformed (up to 170 MPa) as it moves relative to the mandrel at a speed of up to 1 m/s [20, 23]. The durability of mandrels can be effectively increased by oxidizing the nose surface and the spherical surface in order to form an oxidized layer, which prevents the billet metal from sticking to the mandrel and is a heat-insulating layer [21–23]. The formation of an oxide layer on the surface increases the wear resistance of mandrels by a factor ranging from 1.5 to 2.0 [23]. The oxide layer provides additional thermal resistance, the value of which depends on the thickness of the scale layer, its thermophysical and mechanical properties [24–26]. As the temperature increases, the scale softens, and very soon it starts to act as a lubricant during contact. As is known [27, 28], under high-temperature oxidation (900–1000 °C), a layer consisting of FeO (wustite), Fe_2O_3 (hematite), and Fe_3O_4 (magnetite) is formed on a metal surface. The FeO film is an inner layer, and it peels off easily; therefore, when forming an oxide layer, one should reduce the amount of wustite as much as possible and make it turn into magnetite (Fe_3O_4). To increase the service life of piercing mandrels, protective coatings containing iron oxides can be successfully used. The development of various combinations of differently functioning layers is of interest.

The aim of this paper is to study the chemical composition, structure, and microhardness of plasma-sprayed multilayer high-temperature coatings of two different compositions, which are supposed to be used to increase the service life of piercing mandrels.

Materials and research methods

The coatings were applied by means of a *UPN-60KM TSP2017* plasma powder spraying unit with contact initiation of an arc discharge (*NPP TSP LLC*, Ekaterinburg). All the layers of the multilayer coatings were sprayed onto *20CrNi4V* (3310) chromium-nickel steel specimens under the same following conditions: a current of 310 A and a voltage of 57...60 V, with argon as the main plasma-forming gas and hydrogen as a high-enthalpy gas. Multilayer coatings with two different compositions were studied. The coatings were produced by successive overlaying of three layers. The first layer is meant for protection against high-temperature oxidizing and wear, it is produced by spraying self-fluxing powders of compositions 1 and 2 (see Table 1). This layer is required to prevent rapid fracture of mandrels in the case of the wear of the upper layers. It will enable one to remove the mandrel from operation in due time and to reapply the fractured outer layers, thus restoring the protective properties of the mandrel. The second layer is transitional, and it is produced by spraying a 50:50 mixture of high-temperature powders of compositions 1 or 2 with *Fe* powder. This layer is designed for a smooth variation of properties from the outer layer to the inner one, and it delivers iron to form an oxide film at high operating temperatures. The third metal-oxide layer is produced by spraying *Fe* powder in an oxidizing environment. It serves to restore the outer oxidized layer at the operating temperatures of piercing mandrels. The morphology of the particles of the sprayed powders is shown in Fig. 1.

Table 1

Composition of powders being sprayed

Powder composition 1, wt. %						
<i>C</i>	<i>Cr</i>	<i>Si</i>	<i>B</i>	<i>Mn</i>	<i>V</i>	<i>Fe</i>
1.2	3.8	2.3	3.6	1.0	1.0	Bal
Powder composition 2, wt. %						
<i>C</i>	<i>Ni</i>	<i>Si</i>	<i>B</i>	<i>Mn</i>	<i>Fe</i>	
0.5	9.0	1.2	2.7	4.0	Bal	

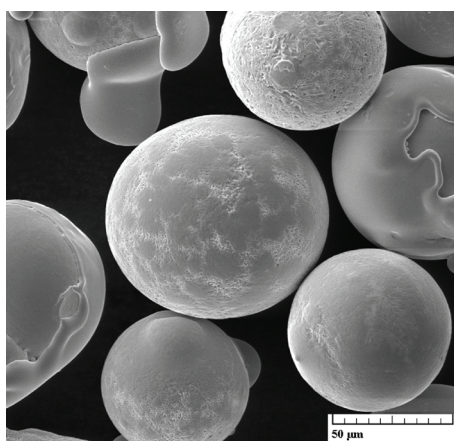
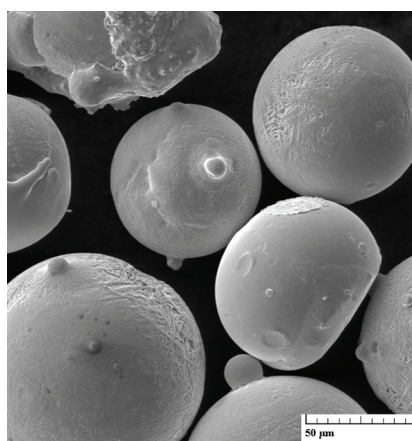
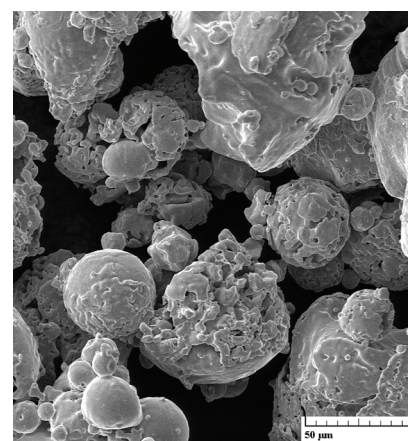
*a**b**c*

Fig. 1. Morphology of powder particles for multilayer coating spraying:

a – powder of composition 1; *b* – powder of composition 2; *c* – *Fe* powder

The structural features and thickness of the obtained coatings were studied on the cross cuts by means of a *Tescan Vega II XMU* scanning electron microscope, equipped with an *Oxford HKL Nordlys F+* energy dispersive attachment, and a *Neophot-21* microscope at 100- to 1,000-fold magnification. By means of a *Shimadzu* X-ray diffractometer using $K\alpha$ *Cr* radiation, phase X-ray diffraction analysis of each layer was performed on the specimen surface immediately after layer application. Microhardness was measured in a *Leica* hardness tester with a load of 50 g.

After spraying of all the three coating layers, oxidizing annealing was performed at 900 °C in order to create a dense layer of $FeO+Fe_2O_3+Fe_3O_4$ scale on the surface. It is this oxide layer that will act as a lubricant in the operation of piercing mandrels and as a thermal barrier decreasing the surface temperature. The average size of the powder particles ranged between 60 and 100 μm for the coating of composition 1 and between 70 and 90 μm for the coating of composition 2; the size of the *Fe* powder particles was 40 to 60 μm.

Results and discussion

As was supposed, the sprayed coatings consist of four layers (Fig. 2). The thicknesses of each layer are presented in Table 2. According to the results of the phase X-ray diffraction analysis, the surface oxide layers on both studied coatings are identical and consist of a mixture of the oxides FeO , Fe_2O_3 , and Fe_3O_4 (Fig. 3). The surface layer is denoted by 4 in Fig. 2; it should play the role of a lubricant, which decreases the friction coefficient and prevents the base metal from overheating.

Obviously, the presence of diffraction peaks from the *Fe*-based α -solid solution is due to the fact that the metal oxide layer (3 in Fig. 2) got into the analysis of the specimen surface. The structure and element distribution of this layer are shown in Fig. 4.

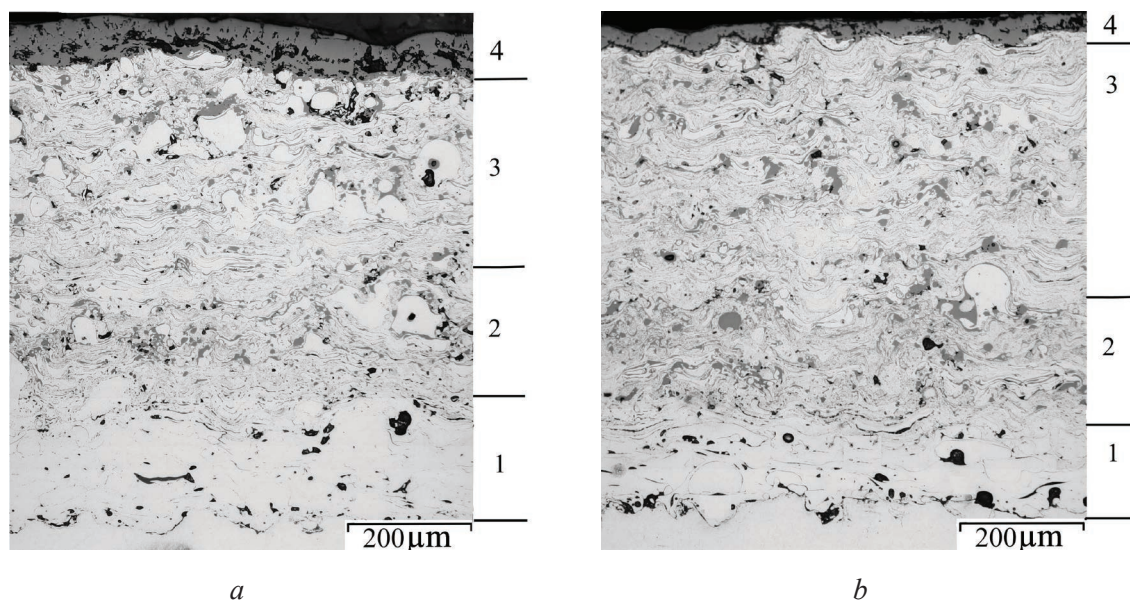
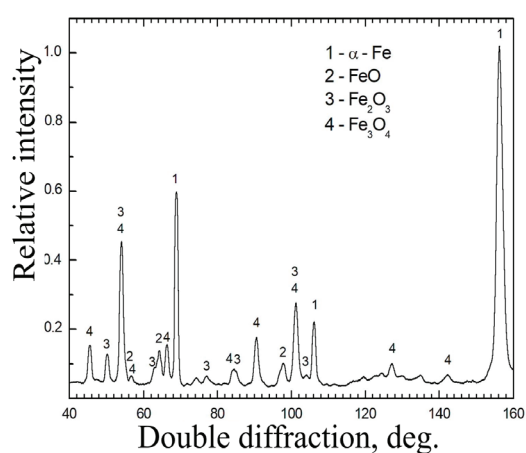


Fig. 2. The microstructure of the multilayer high-temperature coating:
a – the coating of composition 1; *b* – the coating of composition 2

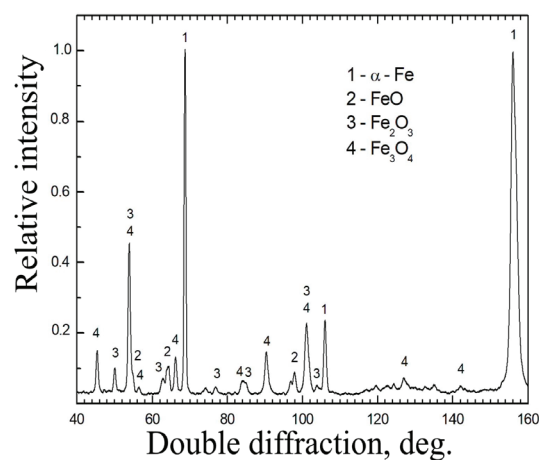
Table 2

Thickness of coating layers

Coating	Composition 1, μm	Composition 2, μm
1 st layer	200	150
2 ^d layer	250	250
3 ^d layer	350	370
4 th layer	50	30
Total thickness	850	800



a



b

Fig. 3. A fragment of the diffraction pattern of the outer oxide layer:
a – coating of composition 1; *b* – coating of composition 2

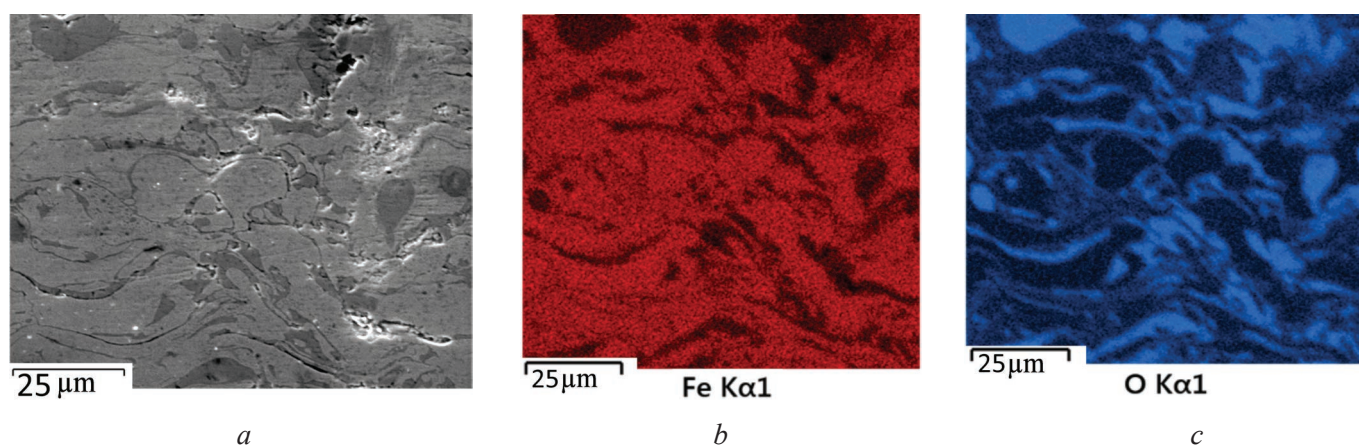


Fig. 4. Distribution of elements in the metal-oxide layer (3 in Fig. 2):

a – image in secondary-electron mode; *b* – in the characteristic X-ray radiation of iron; *c* – in the characteristic X-ray radiation of oxygen

The inner layer (1 in Fig. 2) has a complex phase composition. Areas formed from molten undeformed and plastically deformed particles of the initial powder, as well as from deformed particles that have not had time to melt, are clearly distinguishable in its structure (Table 3). The particles of the first kind have managed to solidify before impact on the substrate; therefore, it has a practically initial round shape and a pronounced dendritic structure (1 in Fig. 5). The second-type particles have solidified directly on the substrate under deformation, and it has a polygonal structure without dendrites (3 in Fig. 5). The third grain type in the coatings studied (2 in Fig. 5) is represented by unmolten initial powder particles heated and deformed during collision with the substrate surface. The phase X-ray diffraction analysis has shown that the matrix in the coatings is constituted by solid solutions, namely, chromium ferrite for the coating of composition **1** (Fig. 6a) and ferrite + austenite for the coating of composition **2** (Fig. 6b). Fine carbides, silicides, and borides are the strengthening phases (Fig. 6).

The second layer consists of the same phases as the first one, but with a higher content of ferrite formed during *Fe* powder spraying. In these ferrite grains there are no strengthening phases; therefore, it plays a role of dampers relaxing the arising thermal stresses. This layer provides smooth variation of the properties from layer to layer in the coatings. In this case, the porosity of the second layer is a positive factor since, during operation, under conditions of friction forces, the outer oxide layer penetrates into the pores of the

Table 3

The chemical composition of the grains of the high-temperature coating marked in Figs. 5, at. %

Coating of composition 1 (Fig. 5, <i>a</i>)							
Grain No.	<i>B</i>	<i>C</i>	<i>Si</i>	<i>V</i>	<i>Cr</i>	<i>Mn</i>	<i>Fe</i>
1	13.5	2.0	4.5	1.1	4.5	2.6	71.7
2	9.9	0.9	3.1	1.4	6.5	3.5	74.3
3	14.9	1.3	7.4	0.8	14.2	1.5	59.7
Coating of composition 2 (Fig. 5, <i>b</i>)							
Grain No.	<i>B</i>	<i>C</i>	<i>Si</i>	<i>V</i>	<i>Mn</i>	<i>Fe</i>	<i>Ni</i>
1	13.5	2.2	3.6	0.3	3.6	69.1	7.5
2	6.8	1.3	4.5	0.3	4.1	75.2	7.5
3	7.3	0.6	5.2	0.3	3.3	75.6	7.6

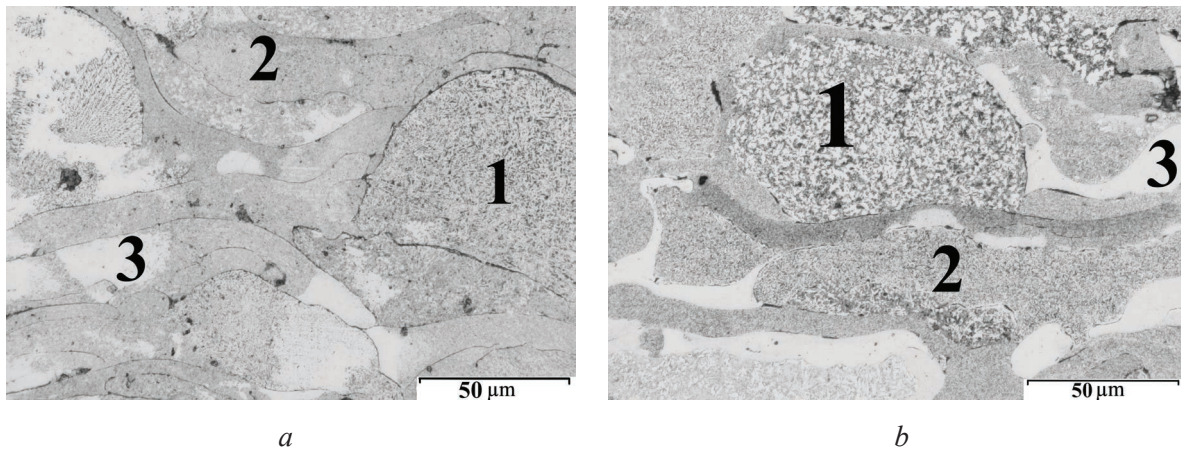


Fig. 5. The microstructure of the inner layer of the coatings (1 in Fig. 2):

a – composition 1; *b* – composition 2

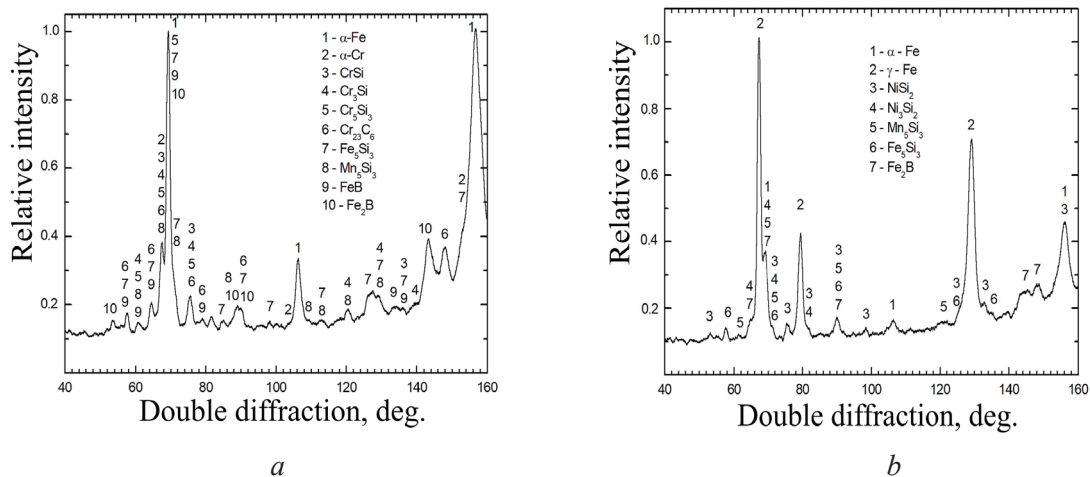


Fig. 6. Fragments of diffraction patterns of the inner layer of the coatings under study:

a – composition 1; *b* – composition 2

second layer and even the third one. Under severe operating conditions, which are typical for piercing mandrels, the outer oxide layer will gradually fail. At high temperatures, the recovery of the oxide layer due to iron oxidation in the third (metal-oxide) layer of the coatings is to be expected. The oxide layer can be restored by additional heat treatment, e.g., by surface heating with a gas or plasma torch. The porosity of the third layer will intensify oxygen penetration into the coating and increase the surface participating in the oxidation reaction.

As the outer layers wear, nickel and chromium contained in the second and first inner layers become involved in the oxidation process. A decrease in the coating wear rate with transition to the inner layers is to be expected. This will be owing to the strengthening phases (carbides, silicides, and borides) having high hardness and increasing the hardness of the first inner layer (Fig. 7).

The dendritic structure of the molten initial powder particles that have solidified before colliding with the substrate surface (Fig. 8) should help to slow down the wear of piercing mandrels during operation. In the coating of composi-

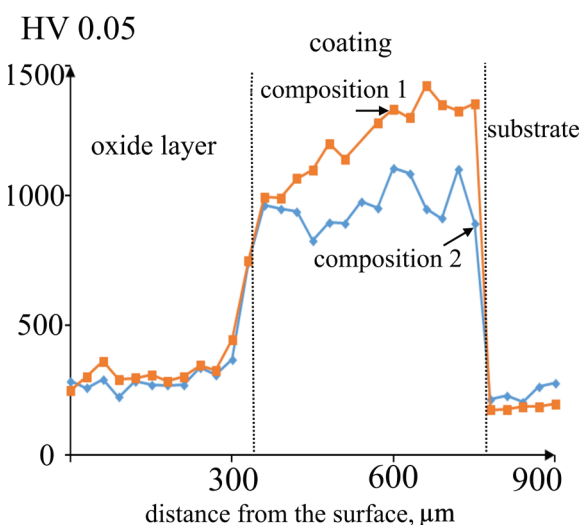


Fig. 7. Microhardness distribution through the coating thickness

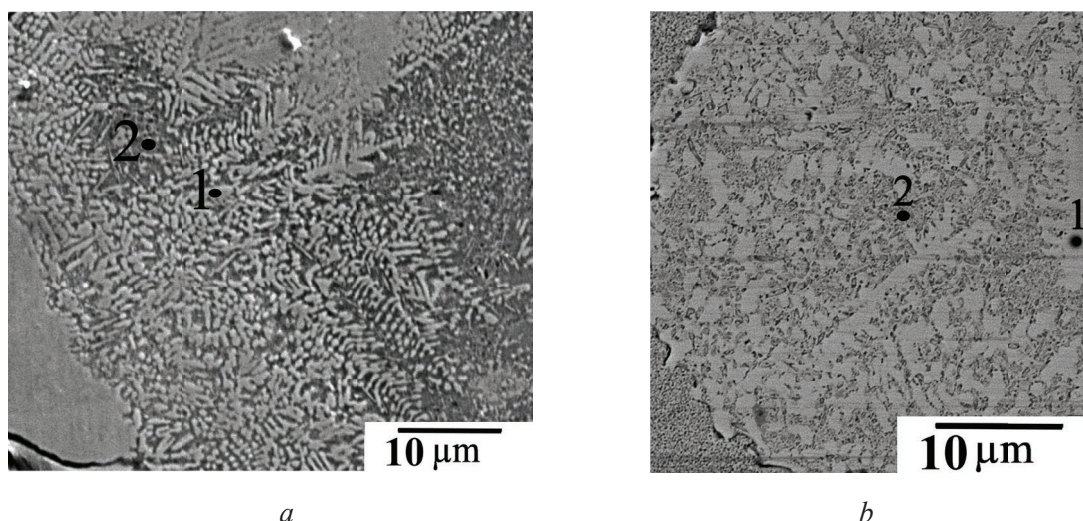


Fig. 8. Microstructure and chemical composition of non-deformable particles of type 1:
a – coating of composition 1; b – coating of composition 2

tion 1, dendrites have a fine structure with closely spaced secondary branches, and this testifies to high solidification rates (Fig. 8, a). The dendritic branches are enriched with silicon and carbon, and the interdendritic spaces are enriched with boron (Table 4 and Fig. 8, a). Most likely, the dendritic branches consist of chromium ferrite with dispersed particles of $Cr_{23}C_6$ carbides, $CrSi$, Cr_3Si , and $(Cr,Fe,Mn)_5Si_3$ silicides, and the interdendritic spaces consist of chromium ferrite with disperse FeB and Fe_2B boride particles. In the coating of composition 2, the particles that have solidified before collision with the substrate are characterized by a coarser dendritic structure (Fig. 8, b).

Table 4

The results of local chemical analysis of the areas, marked in Fig. 8, at. %

Coating composition 1 (Fig. 8, a)							
Area No.	B	C	Si	V	Cr	Mn	Fe
1	3.39	0.23	2.40	1.28	5.70	3.70	83.30
2	7.52	0.00	1.10	1.47	6.12	3.53	80.26
Coating composition 2 (Fig. 8, b)							
Area No.	B	C	Si	V	Mn	Fe	Ni
1	8.55	2.11	3.91	0.30	4.58	73.65	6.90
2	17.16	2.42	3.59	0.24	3.63	66.17	6.80

In austenite, in the dendritic branches, a larger number of disperse nickel, manganese and iron silicide particles are formed, with Fe_2B iron borides concentrated in the interdendritic spaces (Fig. 8, b and Table 4). The presence of silicon and chromium in the inner layer will ensure the formation of a $Cr_2O_3 + SiO_2$ protective film during oxidation, which is known to be a reliable protection from high-temperature oxidation.

Conclusion

Compositions of layers in multilayer high-temperature coatings protecting the surface of piercing mandrels are proposed.

The first layer, 150 to 200 μm thick, is produced by plasma spraying of self-fluxing $Fe-Cr-Mn-V-Si-C-B$ and $Fe-Ni-Mn-Si-C-B$ powders and characterized by high hardness (up to 1400 HV 0.05) due to the

strengthening phases of carbides, silicides, and borides chaotically located in the ferritic (for coating 1) and austenitic (for coating 2) matrices. The aim of this layer is to provide high wear resistance and thermal oxidation resistance due to the formation of protective SiO_2 and Cr_2O_3 oxide films.

The second layer is produced by plasma flux spraying of a mixture of heat-resistant self-fluxing powders and an iron powder in the 1:1 ratio for providing a smooth transition of properties from the oxygen-containing to metallic layers. The thickness of this layer is 250 μm , and its microhardness is 800 HV 0.05. It consists of the same phases as the first layer, but with an additional number of ferrite grains free from strengthening phases.

The third (metal-oxide) layer formed by plasma spraying of iron powder in an oxidizing environment is meant for the recovery of the surface oxide layer after wear at operating temperatures of piercing mandrels. It has a thickness of 350 μm and a microhardness of 300 HV 0.05. The layer is a mechanical mixture of ferrite grains and iron oxides.

The fourth (surface) layer of the $FeO+Fe_2O_3+Fe_3O_4$ oxide mixture is produced by oxidizing the surface of the metal oxide third layer by means of a plasma torch. The aim of this layer is to reduce the friction coefficient of piercing mandrels during operation and to decrease their surface temperature.

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Conflicts of Interest

The authors declare no conflict of interest.