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COMPARISON OF CHEMICAL COMPOSITION AND WEAR OF IRON AND NICKEL ALUMINIDE COATINGS APPLIED BY PLASMA SPRAYING

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Given such properties as wear resistance, corrosion resistance, heat resistance, contact and cyclic strength, iron and nickel aluminides can be used as coatings in friction pairs for various purposes. The object of research is the process of obtaining plasma-sprayed coatings from powders based on iron and nickel aluminides. The task of research is the determination and comparison of the structure, chemical composition, and wear resistance of coatings from powders of iron aluminide and nickel aluminide, obtained by the method of atmospheric plasma spraying. To reduce the oxidation of powders during spraying, the conditions for generating a laminar plasma jet were created. Such conditions were provided by the specially developed structure of the electric arc plasma gun and the operating parameters of coating spraying. As a result of the correct choice of spraying modes, completely melted splats were formed during the collision of powder particles with the surface. The resulting coatings had a layered microstructure with microcracks and peeling. The porosity and characteristics of the delamination of the coatings depend on both the modes and the spraying technology. Tribological tests of the coatings were carried out under conditions of dry metal-on-metal sliding friction in a pair with high-speed steel. It is shown that the wear of nickel aluminide coatings under these conditions is 2–2.5 times lower than that of iron aluminide coatings. The coefficient of friction of iron aluminide coatings is slightly lower than that of nickel aluminide coatings. The wear of samples made of 30CrMnSi steel is 3–4 times higher than samples with coatings. The difference in the wear of the coatings is explained by the more intense oxidation of iron aluminide in the friction process. Conclusions were drawn regarding the possibility of using the investigated coatings in various friction pairs of structural elements, in particular in the automotive industry

Keywords: iron aluminide, nickel aluminide, plasma spraying of coatings, dry friction

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1. Introduction

Various methods of gas thermal spraying are used to apply wear-resistant coatings, the most common of which are gas flame spraying (FS) [1, 2], detonation spraying (DS) [3], atmospheric plasma spraying (APS) [4], and high-speed gas flame spraying (HVOF) [5, 6].

Among the indicated methods, APS is the most versatile, technological, and highly productive with the possibility of spraying almost any metals and alloys, in particular refractory ceramics [7]. The use of plasma guns of different designs allows coating to be sprayed on surfaces with complex geometry, both external and internal. Spraying in the laminar plasma jet generation mode makes it possible to apply coatings containing nanodisperse components [8].

Intermetallics based on aluminides such as Fe_3Al, FeAl, Ni_3Al, NiAl, Ti_3Al, TiAl are of particular interest for obtaining wear-resistant coatings. Due to their high tribological and physical-mechanical properties, these materials are widely used in various industries. In particular, in the automotive industry, there is a whole range of parts with sprayed coatings: parts of the transmission, piston group, valve mechanism, shaft necks, brake discs, etc. [9–11].

In this connection, the study of the processes of plasma spraying of coatings based on intermetallics and their tribological properties is relevant.

2. Literature review and problem statement

The characteristic properties and practical application of FeAl, NiAl, and NiTi intermetallics using the HVOF and HVAS methods are given in [12]. It is indicated that FeAl and TiAl coatings are good options from the operational point of view. The authors of the cited paper note that improving wear indicators is one of the main areas of research to better understand the behavior of these coatings at room and high temperatures. Oxidation resistance is also one of the most important challenges that intermetallics must exhibit to be useful for high temperature applications. The study of the wear resistance of FeAl powder coatings under dry friction conditions obtained by the method of cold gas-dynamic spraying (cold-spraying) was carried out in [13]. However, there is no comparison of the wear resistance of FeAl and NiAl intermetallic coatings in the cited work, which does not make it possible to reasonably approach the application of these coatings under specific conditions of use.

In works [14, 15], the chemical composition of coatings of the Fe–Al system obtained by the DS method was investigated and tests for abrasive wear under dry friction conditions were carried out. It is shown that during the formation of these coatings, changes in chemical composition occur with the participation of oxygen with the formation of oxide phases Al_2O_3, Fe_3O_4, FeO, $Fe(Al_2O_4)$ and phases depleted of iron or aluminum. In the zone adjacent to the substrate, there are areas of mixed equiaxed subgrains of FeAl and Fe_3Al phases, small grains of saturated Fe(Al) solid solution, amorphous oxide, micro- and nanopores [15]. However, there is also no comparison of changes in the chemical composition and wear resistance of FeAl and NiAl intermetallic coatings during spraying and dry friction.

Iron aluminides deposited by the DS method, in addition to combining high tribological and mechanical properties with low cost and commercial availability of Fe and Al raw materials, also demonstrate resistance to corrosion and erosion [16], Ni-Al coatings have high erosion resistance by the HVOF technology [17]. In [18], the results of tests of coatings made of Ni-Al alloys with different chemical and phase composition as substitutes for toxic electrolytic chromium coatings are reported. FS and APS technologies were chosen for applying NiAl coatings. The authors of the cited works conducted detailed studies into the properties of coatings based on Fe-Al and Ni-Al systems obtained by various technologies; however, the application of plasma spraying technologies was not given sufficient attention.

In [19], the abrasive wear of Fe_3Al, TiAl, Ti_3Al, Al_3Ti, NiAl, Ni_3Al, and $MoSi_2$ intermetallics, as well as composites based on them, was investigated, compared with the behavior of metals, alloys, and ceramics under wear conditions. The given data indicate the disproportionality of the hardness and wear resistance of these intermetallics. In work [20] it was shown that the wear resistance of Fe_3Al and NiAl coatings under conditions of abrasive friction differs slightly; taking into account the cost of nickel, it is more rational to use iron aluminide coatings.

As a result of our literature review, it was found that intermetallics based on FeAl, NiAl are promising materials for wear-resistant coatings and have a fairly wide range of functional properties and practical applications. But there is no single approach in obtaining and comparing the tribological properties of these coatings, which would be created under the same conditions, by one spraying method.

There is a lack of studies into the chemical composition and wear resistance of plasma-sprayed coatings based on Fe-Al and Ni-Al intermetallics.

All this allows us to state that it is appropriate to conduct a study aimed at the identification and comparison of the chemical composition and tribological properties of plasma-sprayed coatings made of iron and nickel aluminide powders.

3. The aim and objectives of the study

The purpose of this study is to establish the regularities of changes in the chemical composition of intermetallic powders based on iron aluminide and nickel aluminide during plasma spraying, as well as to determine the tribological characteristics of the obtained coatings under dry friction conditions, which will make it possible to reasonably approach the application of these coatings under specific conditions of use.

To achieve the goal, the following tasks were set:

– to reveal the change in the chemical composition of the initial powders of intermetallic iron aluminide and nickel aluminide during plasma spraying;

– to determine the kinetics of wear and the coefficient of friction of the obtained coatings under the conditions of dry sliding friction.

4. The study materials and methods

The object of our research is the process of obtaining plasma-sprayed coatings from powders based on iron and nickel aluminides. Determination of the chemical composition of powders and coatings, as well as electronic images of the topography of the surface of the coatings, was carried out using a scanning electron microscope VEG A3 of the TESCAN company. X-ray diffraction (XRD – X-ray diffractometry) measurements were carried out using an X'Pert, PANalytical diffractometer in Cu-Kα radiation. The composition of the powders determined in this way in the initial state was for the powder based on iron aluminide – 25.27 at.% Al, 4.46 at.% Cr, the rest Fe; for the powder based on nickel aluminide – 44.84 at.% Ni , 55.16 at.% Al. The spectra of the powders are shown in Fig. 1, 2; the chemical composition is given in Tables 1, 2.

Table 1

The chemical composition of iron aluminide powder

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
Iron	26	69059	90.55	80.75	70.27	3.99	4.41
Aluminium	13	151403	15.73	14.03	25.27	0.73	4.65
Chromium	24	15905	5.86	5.22	4.46	0.30	11.77

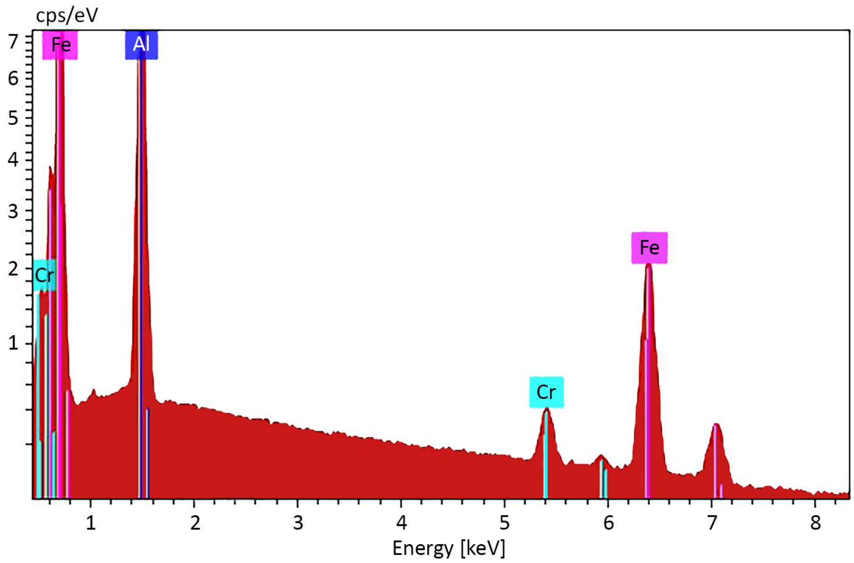

Fig. 1. Spectrum of starting iron aluminide powder

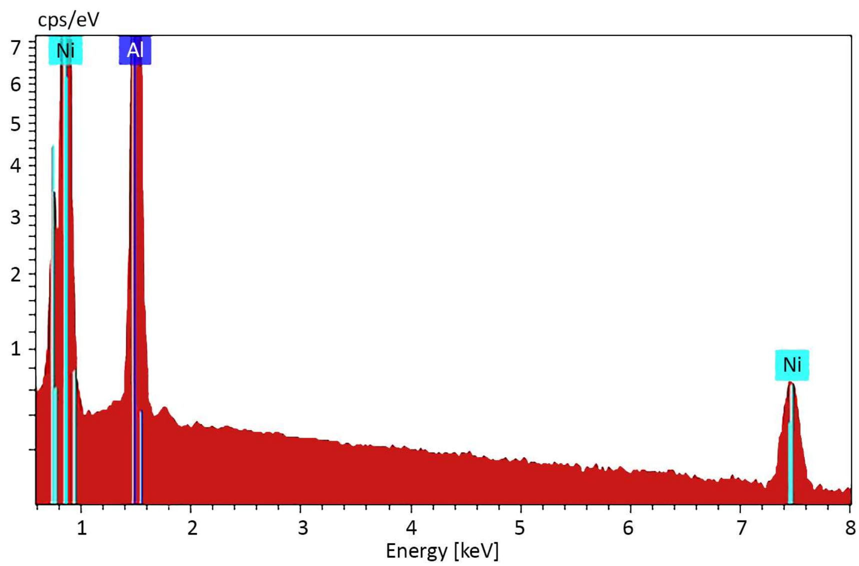

Fig. 2. The spectrum of the initial powder of aluminide nickel

Table 2

The chemical composition of nickel aluminide powder

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
Nickel	28	166947	50.28	63.72	44.84	5.61	12.10
Aluminum	13	110432	28.62	36.28	55.16	1.31	4.59

Powder spraying was carried out by the APS method under the laminar plasma jet generation mode, which contributed to reducing the oxidation of powder particles during flight. The laminar flow of the plasma jet was provided by the specially developed design of the electric arc plasmatron and the spraying technological modes. In this plasmatron, as a result of a separately made anode unit, the arc was not compressed, and the plasma jet was formed by blowing with concentric streams of crimping and shielding argon gas. The arc current was set in the range of 80–90 A, at a voltage of 30–35 V, the powder consumption was 4 kg/h with a total consumption of plasma-forming, transporting, and protective gas (argon) of 5 l/min. Owing to such design features and technological modes, the nature of the plasma jet flow was close to laminar with a Reynolds number Re≈300, which provided protection of the powder components from oxidation and favorable conditions for the formation of the coating.

Samples for spraying were made of medium-carbon low-alloy steel 30CrMnSi and had dimensions of 20×10×5 mm^3. The samples were subjected to tempering at a temperature of (840±10) °C with cooling in oil and subsequent tempering at a temperature of (560±10) °C with cooling in air.

Before spraying, the samples were subjected to abrasive jet treatment using 12A corundum at an angle of 60–90° from a distance of 90–150 mm under an air pressure of 0.5–0.7 MPa. The samples were heated by a plasmatron immediately before spraying to a temperature of about 150–200 °C. The distance between the sample and the plasmatron nozzle was kept constant at 250 mm, the thickness of the coatings was from 250 to 300 μm.

Tribological studies were carried out under conditions of dry sliding friction, where the surface of the coating was in contact with a flat counterbody, and the sample executed a reciprocating movement due to a crank-slider mechanism. The load on the sample was 100 Pa. For the counterbody, high-speed steel was used. The hardness of coatings was determined by Rockwell method in a stationary hardness tester NOVOTEST.

5. Research results

5. 1. Determination of chemical composition of coatings made of aluminide, iron and nickel aluminide

The chemical composition of the coatings obtained by APS can differ greatly in chemical and phase composition from the original powders due to the high heating of particles in the plasma jet, rapid cooling on the surface of the substrate, as well as interaction with the atmosphere. The spectra of powder coatings based on iron aluminide and nickel aluminide are shown in Fig. 3, 4; the chemical composition of coatings is given in Tables 3, 4.

Coatings obtained as a result of APS of intermetallic iron aluminide and nickel aluminide powders have a typical layered microstructure characteristic of thermally sprayed coatings. The porosity of the coatings can reach up to 10 % depending on the spraying modes, there are also microcracks arising due to residual stresses, mainly in oxides due to high brittleness and low fracture toughness. In addition, the images of the coating-substrate cross-sections show partial peeling of the coating from the surface of the substrate. Porosity and delamination of the coating are determined mainly by modes and technology of spraying.

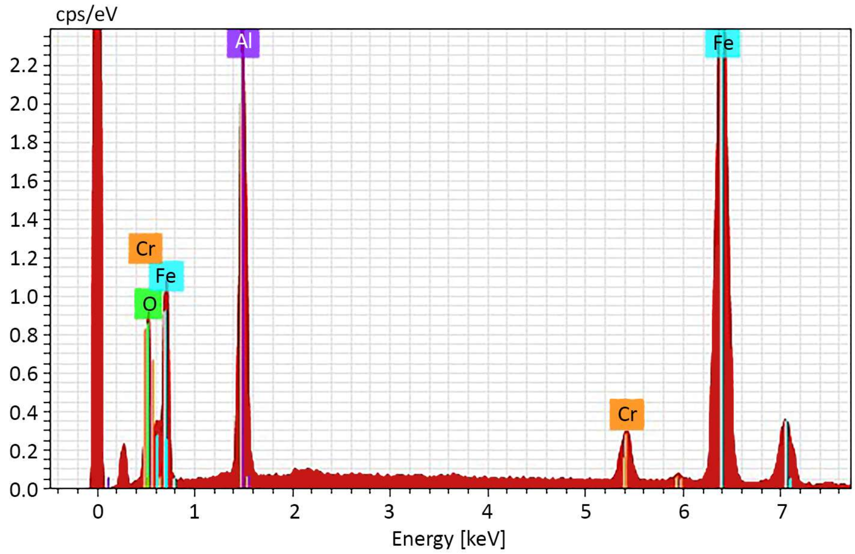

Fig. 3. APS spectrum of iron aluminide coating

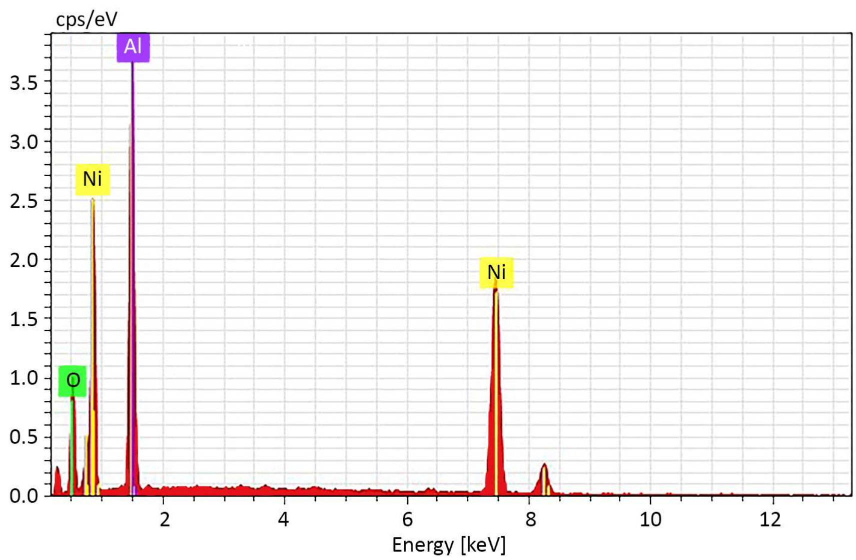

Fig. 4. APS spectrum of nickel aluminide coating

For a more detailed analysis of the structure and chemical composition of coatings, research was conducted on iron aluminide coating, the microstructure of which is shown in Fig. 5, 6; chemical analysis data are given in Table 5.

The lightest structural component, marked with number 1 (Fig. 5), consists mostly of iron with a small amount of aluminum and chromium, which, according to the Fe-Al-Cr ternary diagram [21], may correspond to a solid solution of α-Fe with capacity-centered cubic lattice. Structural components of a gray shade represent various oxides formed as a result of the interaction of elements of powder particles with the atmosphere in the process of plasma spraying. Thus, the area marked with number 2 consists mostly of $FeAl_2O_4$ oxide with an additional chromium content of 10.14 %. The darkest structural component, marked on the microstructure with number 3, consists mainly of aluminum oxide γ-Al_2O_3 with a content of 6.91 % iron and 1.71 % chromium. The area marked with number 4 is based on a solid solution of α-Fe with inclusions of iron oxide. This area has a characteristic shape, which may correspond to a drop that was formed as a result of the splashing of an overheated powder particle at the moment of impact on the surface. The coating area marked with number 5 is based on iron oxides FeO and Fe_3O_4 with additional content of 1.32 % aluminum and 0.86 % chromium.

It should be noted that, with the exception of the mixed α-Fe+FeO phase (section number 4), chromium is always present in concentrations from 0.26 to 10.14 at.%. In addition, to a much lesser extent, areas of the coating with a composition corresponding to the original powder were detected. A similar situation occurs in the case of spraying nickel aluminide powder.

Table 3

Chemical composition of APS iron aluminide coating

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
O	8	3619	11.57	10.72	25.15	2.03	17.52
Al	13	16458	22.03	20.43	28.40	1.12	5.08
Cr	24	3250	3.87	3.58	2.59	0.16	4.22
Fe	26	35492	70.42	65.27	43.86	1.93	2.74

Table 4

Chemical composition of APS nickel aluminide coating

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
O	8	2650	15.71	16.65	33.68	2.87	18.27
Al	13	11495	29.60	31.36	37.64	1.52	5.12
Ni	28	12354	49.06	51.99	28.68	1.43	2.91

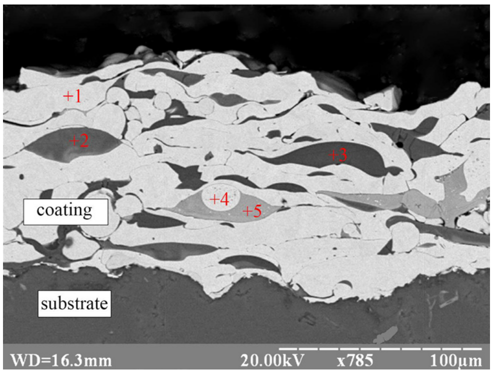

Fig. 5. The microstructure of the iron aluminide coating obtained by APS with markings to determine the chemical composition in individual areas

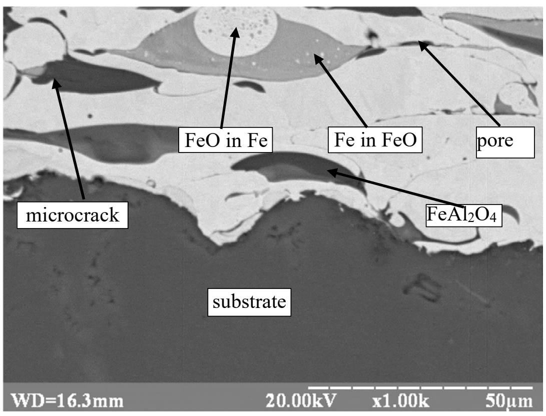

Fig. 6. Microstructure of the coating of aluminide iron obtained by APS

The results of the XRD analysis of the initial powder and plasma coating are shown in Fig. 7.

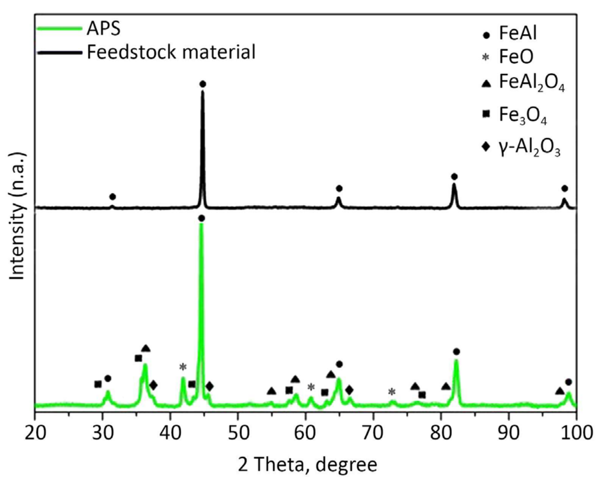

Fig. 7. XRD diffractogram of the original iron aluminide powder and coating of sprayed by APS method [22]

As can be seen from Fig. 7, the coating contains different iron and aluminum oxides. The formation of oxides is formed in the APS process by two mechanisms. On the one hand, the sprayed powder particles, melted at plasma temperatures that can reach 10000 °C, absorb oxygen from the surrounding atmosphere during flight. On the other hand, particles can oxidize on the substrate after impact and in the process of coating formation during cooling. If the particles are oxidized during flight, they are relatively homogeneously distributed in the coating, as in the case of the tested samples. If they are oxidized only after impact, only thin oxide structures can be detected between individual lamellae [23].

Table 5

Chemical composition of APS coating of iron aluminide in individual areas

The content of elements, wt%	Analysis area				
	1	2	3	4	5
Al	0.09	36.67	91.38	0	1.32
Cr	0.26	10.14	1.71	0.02	0.86
Mn	0.1	0	0	0	0
Fe	99.55	53.19	6.91	99.98	97.82
Total	100	100	100	100	100

5. 2. Wear of coatings of iron aluminide and nickel aluminide

The surface morphology of the coatings before and after wear under dry friction conditions is shown in Fig. 8, 9. In Fig. 8, 9 show splats of individual particles. This form of splats corresponds to the state of a completely melted powder particle at the moment of impact on the surface, which confirms the correct choice of spraying modes.

In the case of spraying of iron aluminide powder, individual particles may enter the coating in a state of incomplete melting (Fig. 8, *a*), but this does not significantly affect the wear resistance of the coatings.

The spectrum (Fig. 10, 11) and chemical composition (Tables 6, 7) of the friction surface of coatings based on iron aluminide and nickel aluminide indicate a change in the chemical composition of the surface of the coating in the friction zone. As a result of friction on the surface of the coating based on iron aluminide, the amount of aluminum decreased from 28.40 to 15.54 at.%. The amount of chromium practically did not change and remained at the level of 2.59–2.55 wt.%; the amount of iron did not significantly increase from 43.86 to 51.36 at.%. These changes may be related to the redistribution of elements in the coating, or the transfer of iron from the counterbody to the pores of the coating in the process of friction. An increase in the amount of oxygen from 25.15 to 30.54 at.% is also observed, which indicates additional oxidation of aluminum during friction, the oxides of which can break out from the surface of the coating and be carried out together with the products of friction.

Table 6

Chemical composition of the friction surface of iron aluminide coating

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
0	8	6443	11.72	12.49	30.54	1.83	15.63
Al	13	11002	10.05	10.73	15.54	0.53	5.30
Cr	24	4627	3.18	3.40	2.56	0.13	4.17
Fe	26	56993	68.79	73.38	51.36	1.86	2.71

As a result of friction on the surface of the coating based on nickel aluminide, the amount of aluminum decreased from 37.64 to 26.43 at.%. The amount of nickel did not change significantly (from 28.68 to 30.86 at.%). This fluctuation may be due to the uneven composition of the coating on the surface, as well as measurement error. In the composition of the surface of the coating, a small amount of iron is observed at the level of 1.7 at.%, which is also evidence of its transfer from the counterbody to the pores of the coating during friction. The amount of oxygen increased from 33.68 to 41.01 at.%, which confirms the additional oxidation of aluminum in the friction process, as in the case of friction of a coating based on iron aluminide.

The wear of coatings made of iron aluminide and nickel aluminide intermetallic powders in comparison with 30CrMnSi steel is shown in Fig. 12.

Table 7

Chemical composition of friction surface of nickel aluminide coating

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
O	8	4538	18.33	20.03	41.01	2.99	16.30
Al	13	10718	19.92	21.77	26.43	1.03	5.18
Fe	26	1215	2.66	2.90	1.70	0.15	5.54
Ni	28	17041	50.62	55.30	30.86	1.44	2.85

The histogram of the ratio of wear and hardness of these coatings is shown in Fig. 13.

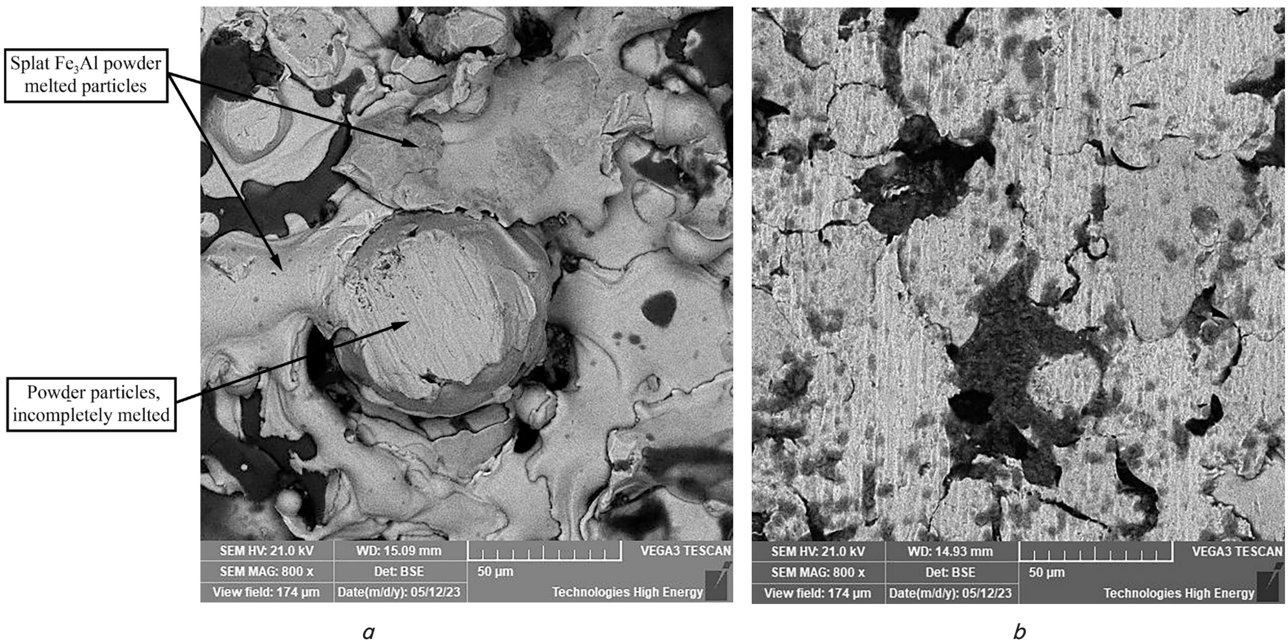

Fig. 8. Morphology of the APS surface of iron aluminide powder coating: *a* – in the original state; *b* – after friction and wear

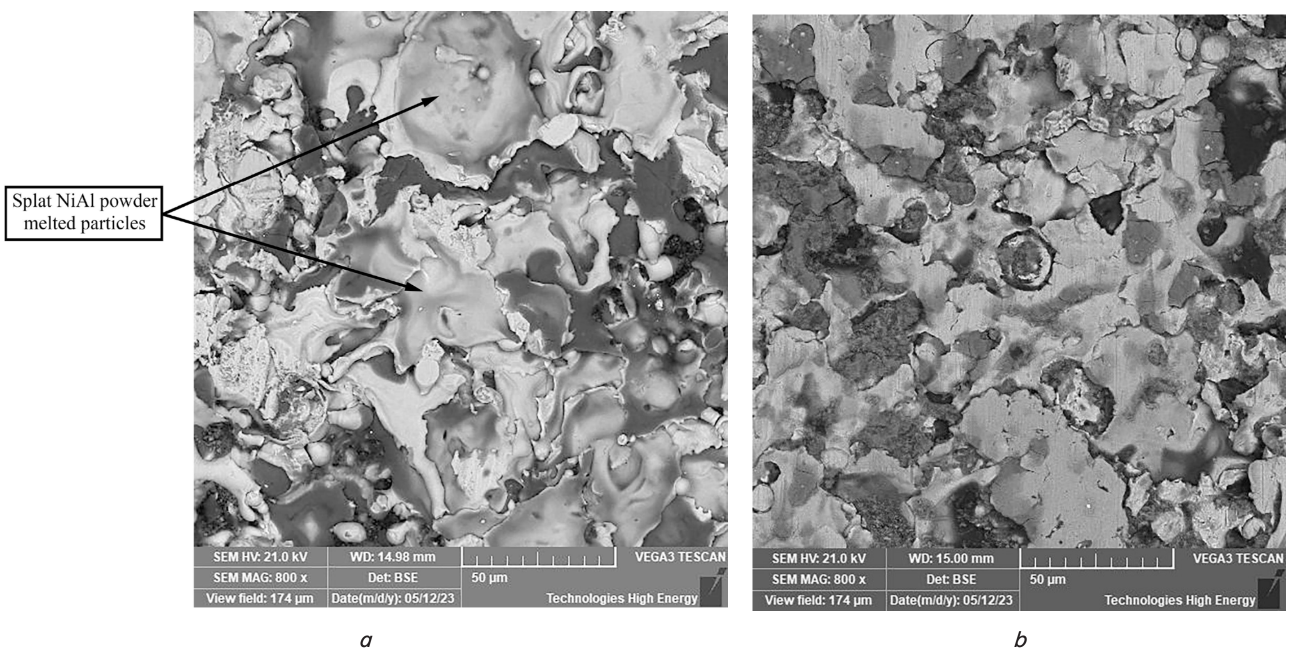

Fig. 9. Morphology of the APS surface of the nickel aluminide powder coating: *a* – in the initial state; *b* – after friction and wear

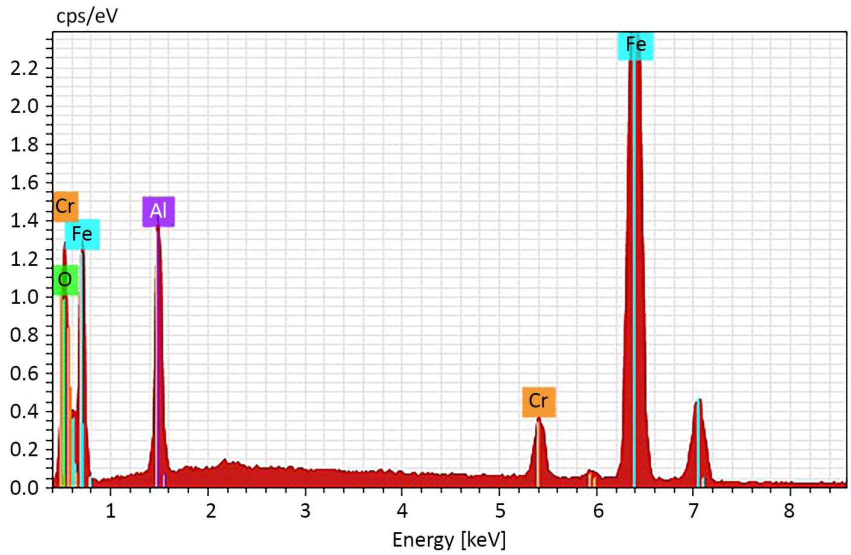

Fig. 10. Friction surface spectrum of iron aluminide coating

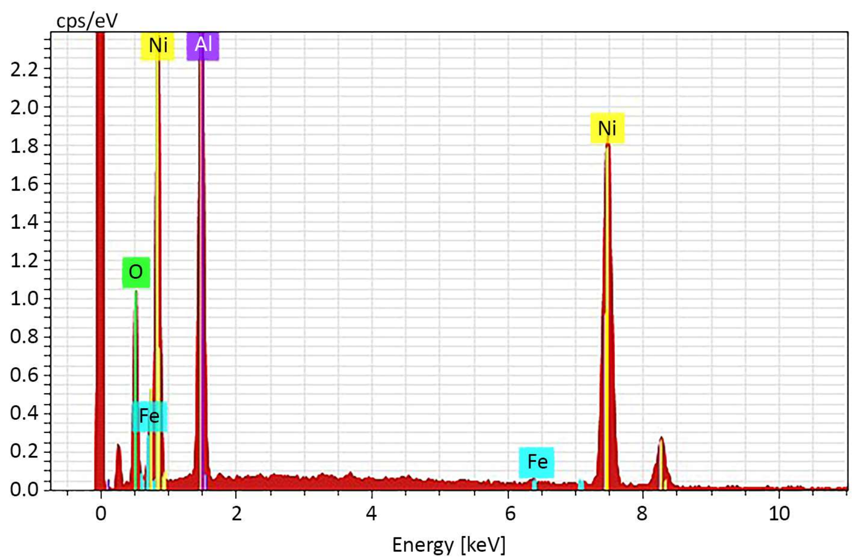

Fig. 11. Friction surface spectrum of nickel aluminide coating

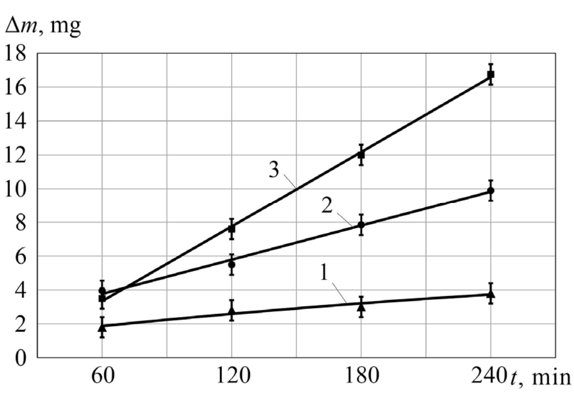

Fig. 12. Dependence of coating wear on time:
1 — from nickel aluminide powders, 2 — iron aluminide,
3 — 30CrMnSi steel

The data in Fig. 12 show that the wear of nickel aluminide coatings after 240 min of friction is 2–2.5 times lower than that of iron aluminide coatings. Such a decrease may be associated with more intense oxidation of iron intermetallic coatings in the friction process. The wear of samples made of 30CrMnSi steel is 3–4 times higher than samples with coatings.

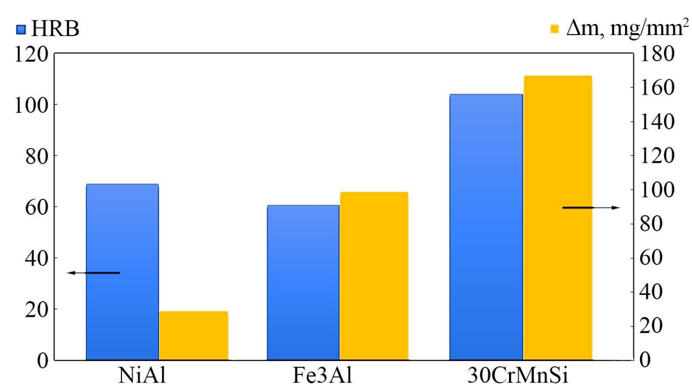

Fig. 13. Histogram of the ratio of hardness and wear of coatings made of nickel aluminide powders, iron aluminide, and 30CrMnSi steel

6. Discussion of results of determining the chemical composition and wear of coatings

In the process of spraying powders based on iron aluminide and nickel aluminide, significant oxidation of powder particles occurs with the formation of various oxides in the coating, which is confirmed by the results of XRD diffractograms (Fig. 7). According to the results of the chemical composition before and after friction, additional oxidation of the coating was determined, while mainly aluminum is oxidized (Fig. 3, 4, 10, 11). Reduction of oxidation processes can be due to the use of a protective environment or spraying in a vacuum, and the properties of such coatings are the results of further research.

Under the conditions of dry sliding friction without lubrication, nickel aluminide-based powder coatings showed the best wear resistance than iron aluminide-based coatings, although the hardness of these coatings is approximately the same (Fig. 12).

Thus, the hardness of coatings based on nickel intermetallics and iron intermetallics is 68.9 HRB and 60.6 HRB, respectively, that is, it differs by 1.1 times. In turn, the wear resistance of coatings based on nickel aluminide is 2 times higher than that of coatings based on iron aluminide, which correlates with the data reported in [20]. The hardness of 30KhGSA steel is 104 HRB, i.e., 1.5–1.7 times higher than that of coatings, while the wear resistance is more than 3 times worse. Such a discrepancy may also be associated with the oxidation of iron during friction, the formation of abrasive particles of iron oxides and thus creating conditions for more intense abrasive wear of steel samples.

The analysis of the surfaces of coatings based on iron aluminide and nickel aluminide before and after the friction test allows us to assert that under the given friction conditions, the surface of the coating is smoothed without deep burrs and areas of adhesion. At the same time, the roughness of the working surfaces of the coating decreases. To compare the results of wear, the coefficient of friction was determined, which was 0.19 for nickel intermetallic coatings and 0.17 for iron intermetallic coatings. This result can be explained by the increased surface roughness of the powder coatings based on nickel intermetalide. In the initial state, the nickel intermetallide powder had a flake shape, unlike the iron intermetallide powder, which had a spherical shape, which is related to the powder manufacturing technology.

Based on our research, the use of powders based on iron and nickel aluminides makes it possible to obtain wear-resistant coatings to increase the resource of friction units in the automotive industry and other industries.

To the limitations of the practical application of the resulting coatings, we can add that in high-temperature friction units, the presence of plastic deformation leads to the destruction of oxide films of iron and nickel aluminide coatings. This phenomenon leads to adhesion of surface materials in tri-pairs, which is unacceptable. Plastic deformation of coatings at high temperatures can be reduced by additional modification.

In further works, it is planned to investigate resistance to corrosion and erosion, adhesive and cohesive properties of modified coatings based on intermetallics.

7. Conclusions

1. Based on the results of investigating the chemical composition of intermetallic powders based on iron aluminide and aluminum aluminide after spraying, oxidation of the constituent powders with the formation of simple and complex oxides FeO, Fe_3O_4, $FeAl_2O_4$, Al_2O_3 was established, which is characteristic of atmospheric plasma spraying.

2. The kinetics of wear and the coefficient of friction of coatings under conditions of dry sliding friction were determined. It is shown that the wear of nickel aluminide coatings under these conditions is lower than that of iron aluminide coatings by 2–2.5 times and 3–4 times compared to 30CrMnSi steel. These dependencees are related to the processes of oxidation of coating elements in the process of friction and wear.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

All data are available in the main text of the manuscript.

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