https://doi.org/10.37434/tpwj2020.07.04

COATINGS BASED ON Fe–Al INTERMETALLICS PRODUCED BY THE METHODS OF PLASMA AND SUPERSONIC PLASMA GAS-AIR SPRAYING

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The results of investigations of the structure and phase composition of thermal coatings based on Fe–Al intermetallics are presented. Fe–Al intermetallics were selected as a material of protective coatings due to their high heat and corrosion resistance and cost effectiveness as compared to many modern heat-resistant materials. As spraying materials, the powders of mechanical mixtures of Fe and Al as well as powders produced by the method of mechanochemical synthesis of Fe–Al intermetallics by the treatment of mixtures of powders Fe and Al in a high-energy ball mill were used. The content of powder components corresponds to the intermetallics Fe_3Al, FeAl and Fe_2Al_5. For spraying, the alloyed powders were also used having the composition corresponding to the intermetallic Fe_3Al. To increase the mechanical and physicochemical properties of the intermetallic, as alloying elements Ti, Mg, Cr, Zr, and La were also used. The coating was produced by the methods of plasma and supersonic plasma gas-air spraying. It was found that in plasma coatings with FeAl-powders, in addition to the initial phase (Fe_3Al, FeAl and Fe_2Al_5), Fe and Al oxides are also present, due to which microhardness of the coatings increases by about 1300 MPa relative to the initial powders. The microhardness of the plasma coating of the alloying powder Fe–TiAl, 2 times increases relatively to the initial powder due to the formation of the intermetallic phase FeTi in the coating. During spraying of mechanical mixtures, due to a low probability of contact interaction of Fe and Al particles during flight and rapid cooling of melts particles on the surface of the base, the synthesis of intermetallics does not have a time to develop and in the coatings no intermetallic phases are revealed. In the coatings produced by supersonic plasma gas-air spraying, the main phase is α-Fe(Al)-solid solution, which is the result of a high rate of melt hardening. 14 Ref., 4 Tables, 8 Figures.

Keywords: Fe–Al intermetallic, powders, mechanochemical synthesis, mechanical mixture, plasma spraying, supersonic plasma gas-air spraying, coating, structure, microhardness

Iron aluminides are among the most widely studied intermetallics due to their cheapness, low specific weight, good wear resistance, convenience in mechanical treatment and resistance to oxidation and corrosion [1, 2]. These advantages resulted in determination of areas of their potential application, including heating elements, furnace fittings, heat exchanger pipes, sintered porous «gas–metal» filters, parts of car valve systems, components of installations operating with salt melts [3, 4]. Producing powders of Fe–Al intermetallics by using the method of mechanochemical synthesis (MChS) allows expanding the areas of practical application of these materials by applying a wide range of heat- and corrosion-resistant coatings from intermetallic Fe–Al-alloys produced by thermal spraying (TS) [5]. To provide the process of TS coatings based on Fe–Al intermetallics, MChS technologies were developed, which allow producing composition powders for this purpose Fe_xAl_y [5, 6], as well as composite powders based on Fe–Al intermetallics [7, 8]. Coatings are produced by the methods of plasma [9], detonation [6, 10] and high-velocity oxy-fuel spraying [7, 8]. In the case of spraying powders of mechanical mixtures of iron and aluminium, the formation of intermetallics of the Fe–Al system occurs during heat treatment of coatings at a temperature of ≥650 °C [11].

The aim of the work was to compare the formation of coatings by spraying mechanical mixtures of iron and aluminium powders and Fe-Al aluminides powders produced by the MChS method. On the other hand, the structure and properties of the coatings produced in terms of their application by the methods of plasma (PS) and supersonic plasma gas-air (SPGAS) spraying were compared, which differ in the conditions of heating the sprayed particles, their acceleration and interaction with a plasma jet.

Materials and procedures of investigations. To select the compositions of coatings from FeAl-alloys, a materials science analysis of the phase equilibrium diagrams of the systems with the participation of Fe and Al was performed [12]. The analysis was made in

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Table 1 Characteristics of powders of Fe−Al system used for plasma and supersonic plasma gas-air spraying

Powder	Method of producing	Phase composition	Microhardness H_{μ}, MPa
86Fe + 14Al (wt.%)	Mechanical mixing	Fe, Al	Fe–1500±230; Al–330±50
Fe_3Al	MChS	Fe_3Al	3590±1010
67Fe + 33Al (wt.%)	Mechanical mixing	Fe, Al	Fe–1500±230; Al–330±50
FeAl	MChS	FeAl, $FeAl_2$	2790±820
45Fe + 55Al (wt.%)	Mechanical mixing	Fe, Al	Fe–1500±230; Al–330±50
Fe_2Al_5	MChS	Al, Fe, Fe_2Al_5	3890±840
86Fe + 14(Al1,5Cr1Zr) (wt.%)	Mechanical mixing	Fe, solid solution of Cr and Zr in Al	Fe – 1500±230 AlCrZr – 355±50
	MChS	Solid solution of Cr and Zr in Fe_3Al	3840±800
86Fe + 14(Al5Mg) (wt.%)	Mechanical mixing	Fe, solid solution of Mg in Al	Fe–1500±230; AlMg–490±80
	MChS	Solid solution of Mg in Fe_3Al	4630±950
86Fe + 14(Al5MgLa) (wt.%)	Mechanical mixing	Fe, solid solution of Mg and La in Al	Fe–1500±230; AlMgLa−580±120
	MChS	Solid solution of Mg and La in Fe_3Al	5580±840
61Fe + 39(62,5Ti37,5Al) (wt.%)	Mechanical mixing	of Fe, TiAl	Fe–1500±230; TiAl–440±140
	MChS	Solid solution of Al in FeTi ($Fe_{1-x}TiAl_x$)	3400±1290

order to select the alloying elements that improve the properties of the intermetallics of the Fe–Al system. According to the results of this analysis, to investigate the structure, microhardness and phase composition of thermal coatings, intermetallic powders were selected (Fe_3Al, FeAl and Fe_2Al_5) and powders of Fe_3Al intermetallics, alloyed with lanthanum, magnesium, chromium, zirconium and titanium, which were produced by the method of MChS [13], as well as mechanical mixtures of powders intended to produce intermetallics of the chosen composition. As the basis to produce alloyed powders, intermetallic Fe_3Al was chosen, since at such a ratio of components it is possible to produce a single-phase product in the MChS process without additional heat treatment [14].

Mechanical mixing of powders was performed in a ball mill for 15 h, the MChS process was performed in a planetary mill «Activator 2SL» during 5 h [13]. Table 1 presents the characteristics of the produced powders, which were used for spraying. The fraction of the sprayed powders was 40–80 μm.

Plasma spraying was performed in the installation UPU-8M, supersonic plasma gas-air spraying was carried out in the installation Kyiv-S. A set of characteristics of the conditions of atmospheric plasma spraying in the subsonic and supersonic mode of plasma jet leakage, used for coating, is the following:

- subsonic: plasma-forming gas — Ar/N_2, $T_{pl} \sim 10000$ K, $W_{pl} \sim 600$ m/s, $W_h \sim 100−130$ m/s, $\tau_h \sim 1.5$ ms;
- supersonic: plasma-forming gas-air, $T_{pl} \sim 6000$ K, $W_{pl} \sim 2500$ m/s, $W_h \sim 300−350$ m/s, $\tau_h \sim 0.5$ ms.

The operating parameters of the spraying processes are given in Table 2.

X-ray diffraction phase analysis (XRD) of the coatings was performed by using the diffractometer DRON-3 in CuK_{α} radiation with a graphite monochromator at a step displacement of 0.1 deg. and an exposure time at each point of 4 s, followed by a computer processing of the obtained digital data.

The microstructure of the coatings was examined in an optical microscope Neophot 32, and the microhardness of the coatings was determined in a microhardness tester PMT-3.

Results of investigations. As a result of metallographic analysis of plasma coatings (Figure 1) from a mechanical mixture of powders 86Fe + 14Al, 67Fe + 33A1, 45Fe + 55Al and from MChS-powders Fe_3A1, FeAl and Fe_2A1_5, it was found that in

Table 2 Parameters of PS and SPGAS processes

Method of spraying	Parameters of spraying process					Heat input	
	Arc current, A	Arc voltage, V	Consumption of plasma-forming gas, m^3/h	Spraying distance, mm	Powder consumption, kg/h	To plasma, kW·h/m^3	To powder, kW·h/kg
PS	500	40	25	120	3	13.3	6.7
SPGAS	280	380	450	120	6	3.9	17.7

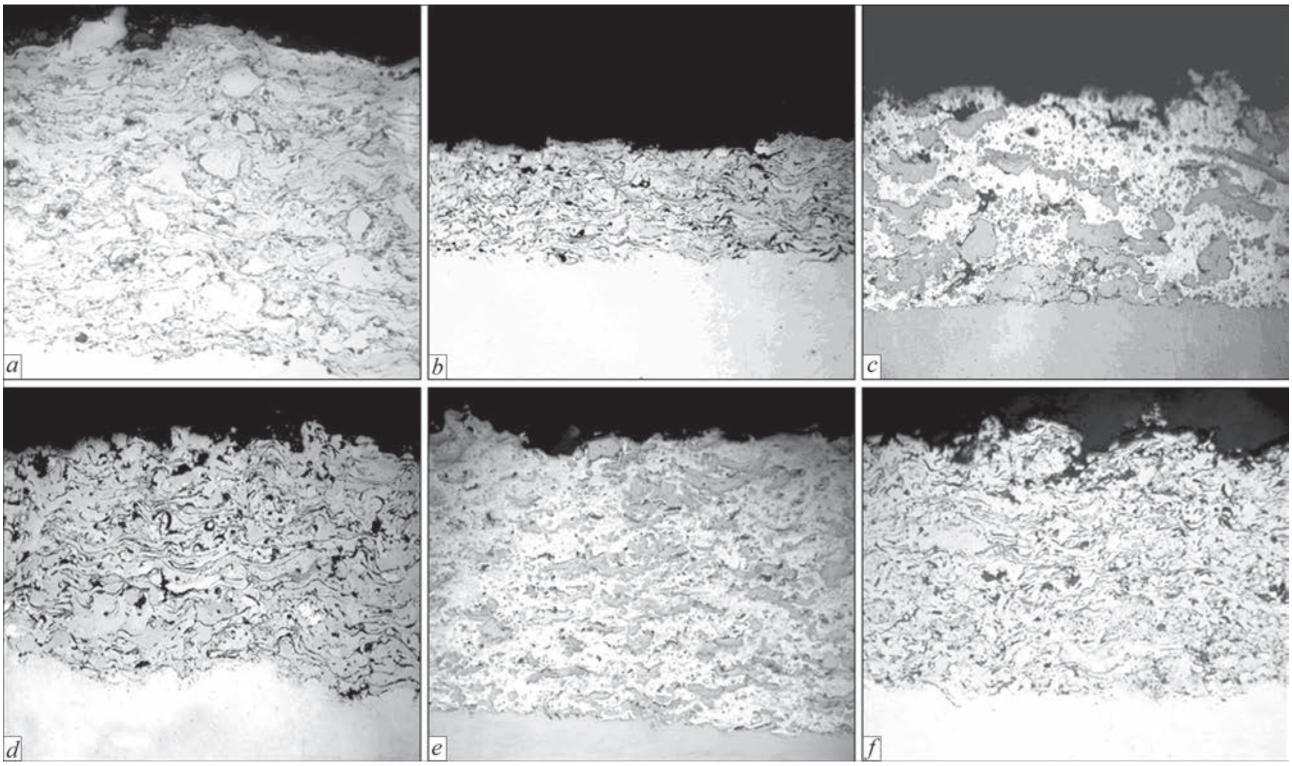


Figure 1. Microstructure (×200) of plasma coatings: from mechanical mixtures 86Fe + 14Al (*a*), 67Fe + 33Al (*c*), 45Fe + 55Al (*e*); of Fe_3Al intermetallic powders (*b*), FeAl (*d*) and Fe_2A1_5 (*f*)

the case of spraying coatings from a mechanical mixture, the structure of the coatings is coarse-grained, in which it is easy to distinguish iron and aluminium both in the form of separate particles (Figure 1, *a*) and in the form of iron inclusions in aluminium matrix (Figure 1, *c*, *d*). When using intermetallic powders, in all cases a dense lamellar structure is formed (Figure 1, *b*, *d*, *f*).

X-ray diffraction phase analysis (Figure 2) showed that during spraying of mechanical mixtures due to a low probability of contact interaction of iron and aluminium particles during flight and a rapid cooling of melt particles on the base surface during the formation of the coating layer, the synthesis of intermetallics does not have a time to develop in the coatings also or intermetallic phases are not detected at all (as, for example, in a mixture intended to produce Fe_3A1, Figure 1, *a,* Table 3), or they appear in the form of traces (as in the case of spraying mixtures intended to produce FeAl and Fe_2A1_5, Figure 2, *c*, *d*, Table 3), and

Table 3. Characteristics of PS and SPGAS-coatings from powders produced by mechanical mixing and MChS method

Powder		Coating		
Composition	Method of producing	Method of spraying	H_μ, MPa	XRD
86Fe + 14Al (wt.%)	Mechanical mixing	PS	Based on Fe–2800±810 Based on Al–540±150	Fe, Al, traces of Al_2O_3 (Figure 2, *a*)
Fe_3Al	MChS	PS	3630±1240	Fe_3Al, FeAl, traces of Al_2O_3 (Figure 2, *b*)
		SPGAS	5090±620	Solid solution of Al in Fe, FeAl, $Fe_xAl_2O_4$ (Figure 4, *a*)
67Fe + 33Al (wt.%)	Mechanical mixing	PS	Based on Fe–2470±640 Based on Al–460±90	Fe, Al, traces of FeAl (Figure 2, *c*)
FeAl	MChS	PS	4150±900	Fe, FeAl, Fe_3O_4, Fe_2O_3, $Al_3Fe_5O_{12}$ (Figure 2, *d*)
		SPGAS	4330±1040	Solid solution of Al in Fe, FeAl, FeO, Al_2O_3, $Fe_xAl_2O_4$ (Figure 4, *b*)
45Fe + 55Al (wt.%)	Mechanical mixing	PS	Based on Fe–2450±800 Based on Al–580±100	Al, Fe, traces of FeAl (Figure 2, *e*)
Fe_2Al_5	MChS	PS	5200±1250	FeAl, Fe, $FeAl_2O_4$, Fe_2Al_5, Al (traces) (Figure 2, *f*)
		SPGAS	5360±850	Solid solution of Al in α-Fe, FeAl, Fe_3Al, $Fe_xAl_2O_4$, Al_2O_3 (Figure 4, *c*)

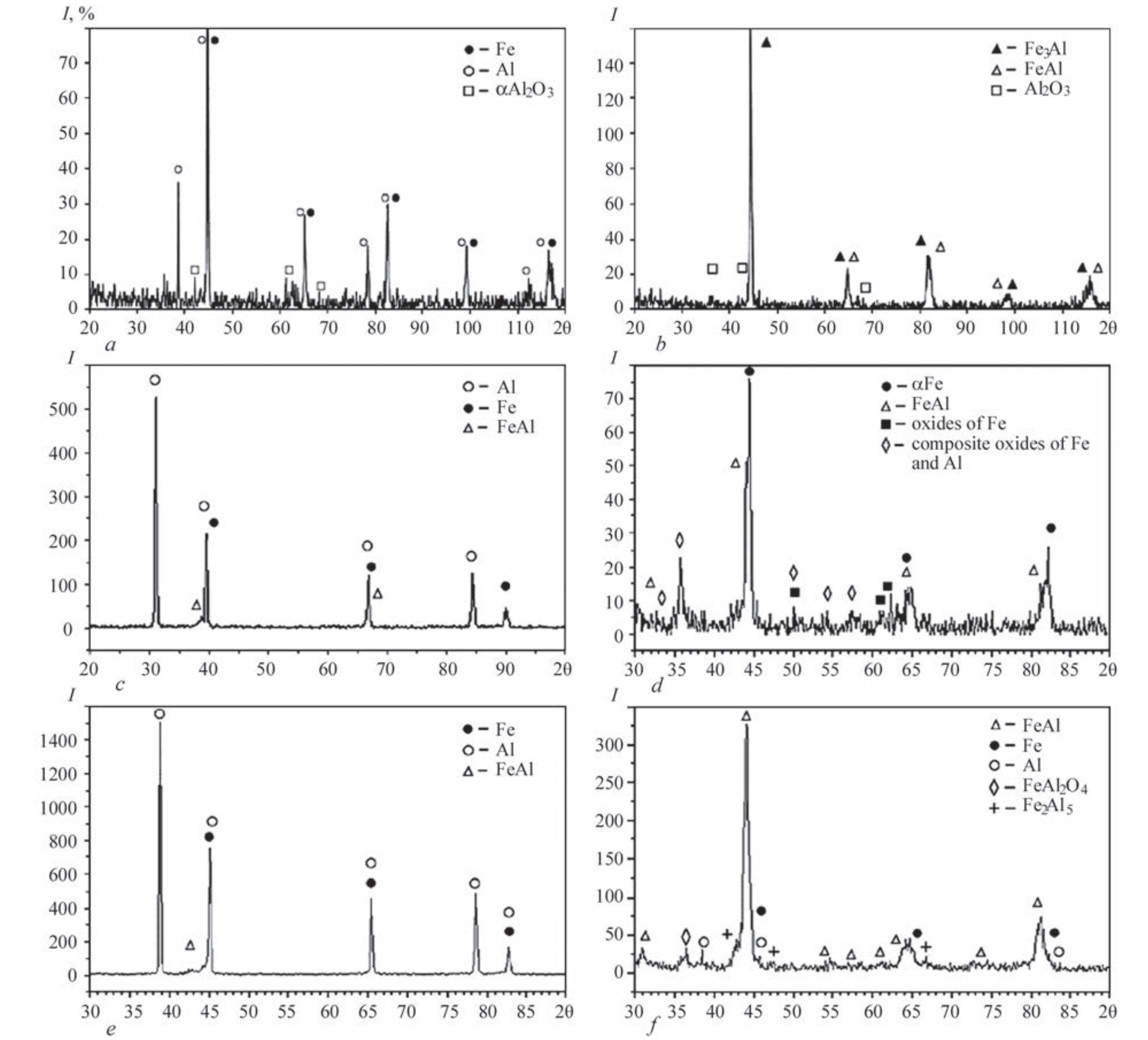


Figure 2 X-ray patterns of plasma coatings: from mechanical mixtures 86Fe + 14Al (*a*), 67Fe + 33Al (*c*), 45Fe + 55Al (*e*); from intermetallic Fe_3A1 powders (*b*), FeAl (*d*) and Fe_2A1_5 (*f*)

phases that do not meet those expected according to the calculation.

During deposition of coatings of intermetallic powders, their phase composition, as a rule, does not completely coincide with the composition of the initial powders, which is associated with the active development of the process of particles oxidation during their flight. Oxides are present in all coatings. In the coating Fe_3A1, $A1_2O_3$ aluminide is present; in the FeAl coating, iron oxides Fe_3O_4, Fe_2O_3 and a composite oxide $A1_3Fe_5O_{12}$; in the coating Fe_2A1_5, oxide $FeA1_2O_4$ are present.

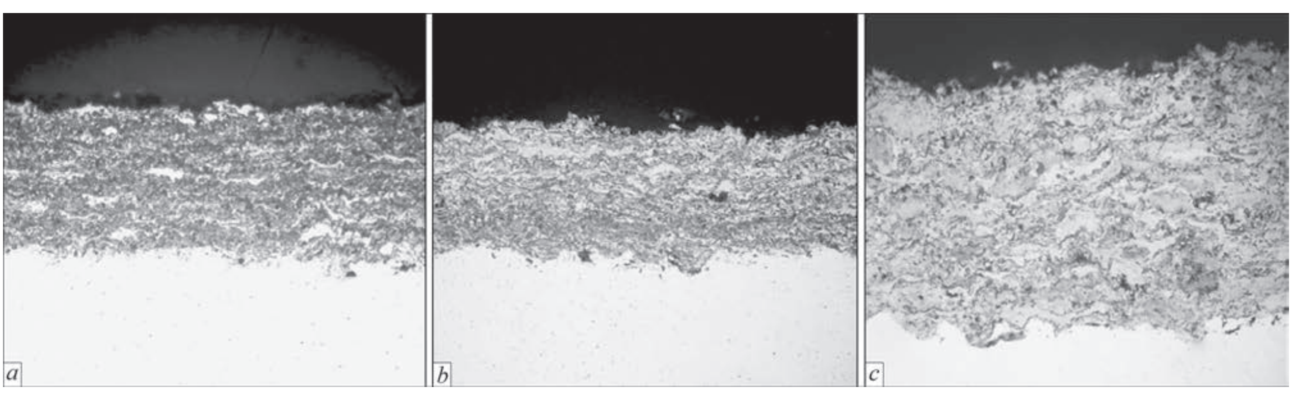
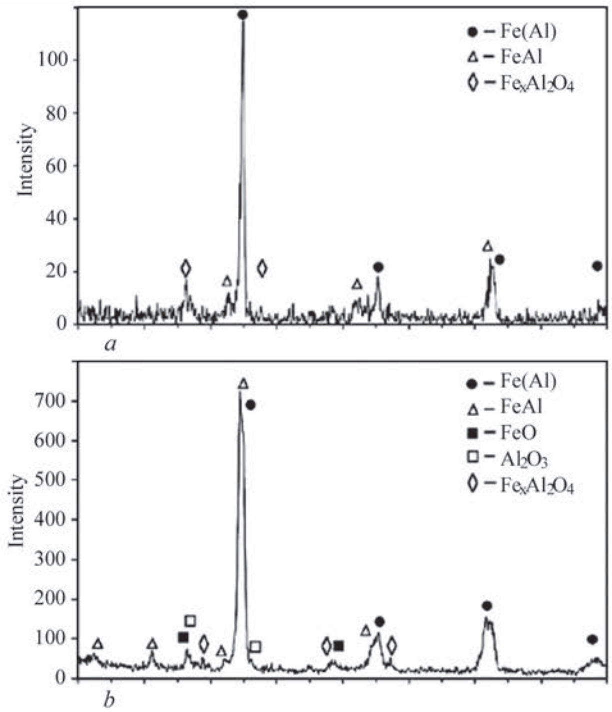


Figure 3 Microstructure (×200) of SPGAS-coatings produced with the use of MChS powders: *a* — Fe_3A1; *b* — FeAl; *c* — Fe_2A1_5



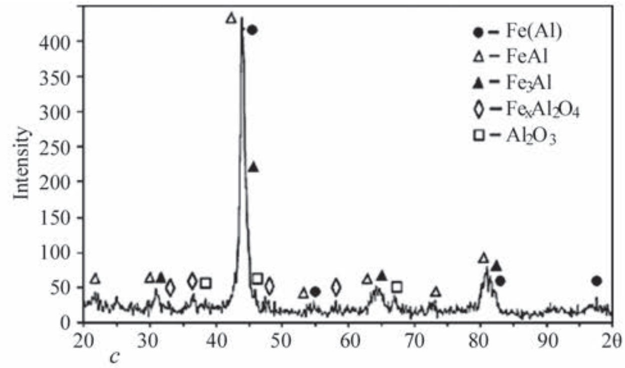
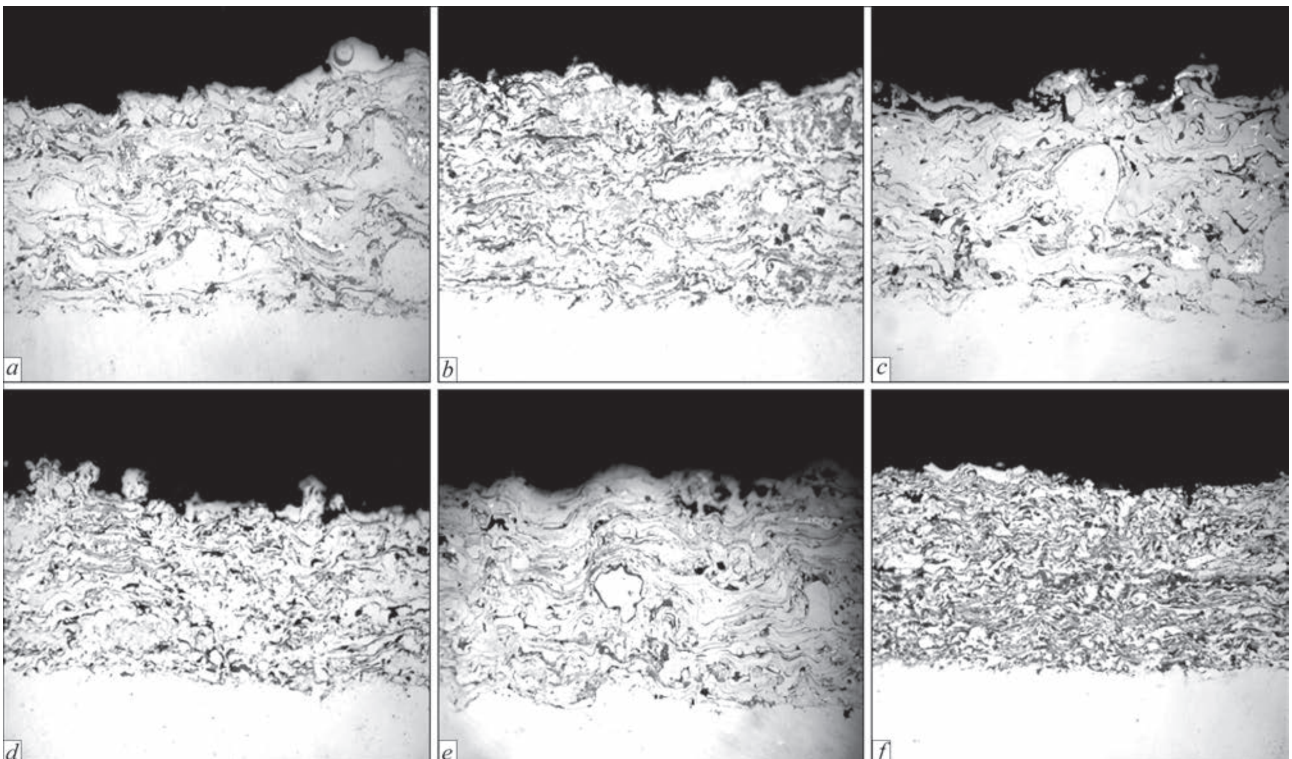


Figure 4. X-ray patterns of SPGAS-coatings produced with the use of MChS-powders: *a* — Fe_3Al; *b* — FeAl; *c* — Fe_2Al_5



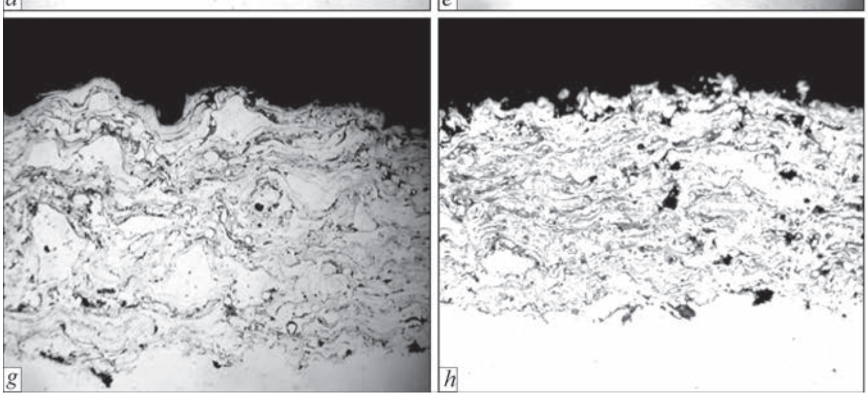


Figure 5. Microstructure (×200) of plasma coatings: from mechanical mixtures Fe + AlCrZr (*a*), Fe + AlMg (*c*), Fe + AlMgLa (*e*), Fe + TiAl (*g*); from MChS-powders Fe–AlCrZr (*b*), Fe–AlMg (*d*), Fe–AlMg-La (*f*), Fe–TiAl (*h*)

As a result of metallographic analysis of SPGAS-coatings (Figure 3) it was found that during spraying of MChS-powders Fe_3A1, FeAl and Fe_2A1_5 a dense structure is formed with the presence of oxide lamellae (content of oxide component in the coatings Fe_3Al, FeAl and Fe_2A1_5 amounts to 50, 25 and 20 vol.%, respectively).

X-ray diffraction phase analysis (Figure 4) found that the phase composition of the SPGAS-coating from the powders Fe_3A1, FeAl and Fe_2A1_5, as in the case of plasma spraying, does not coincide with the composition of the initial powders, but this difference bears different nature because of the different composition of the plasma jet, its velocity and temperature and the conditions of formation of the

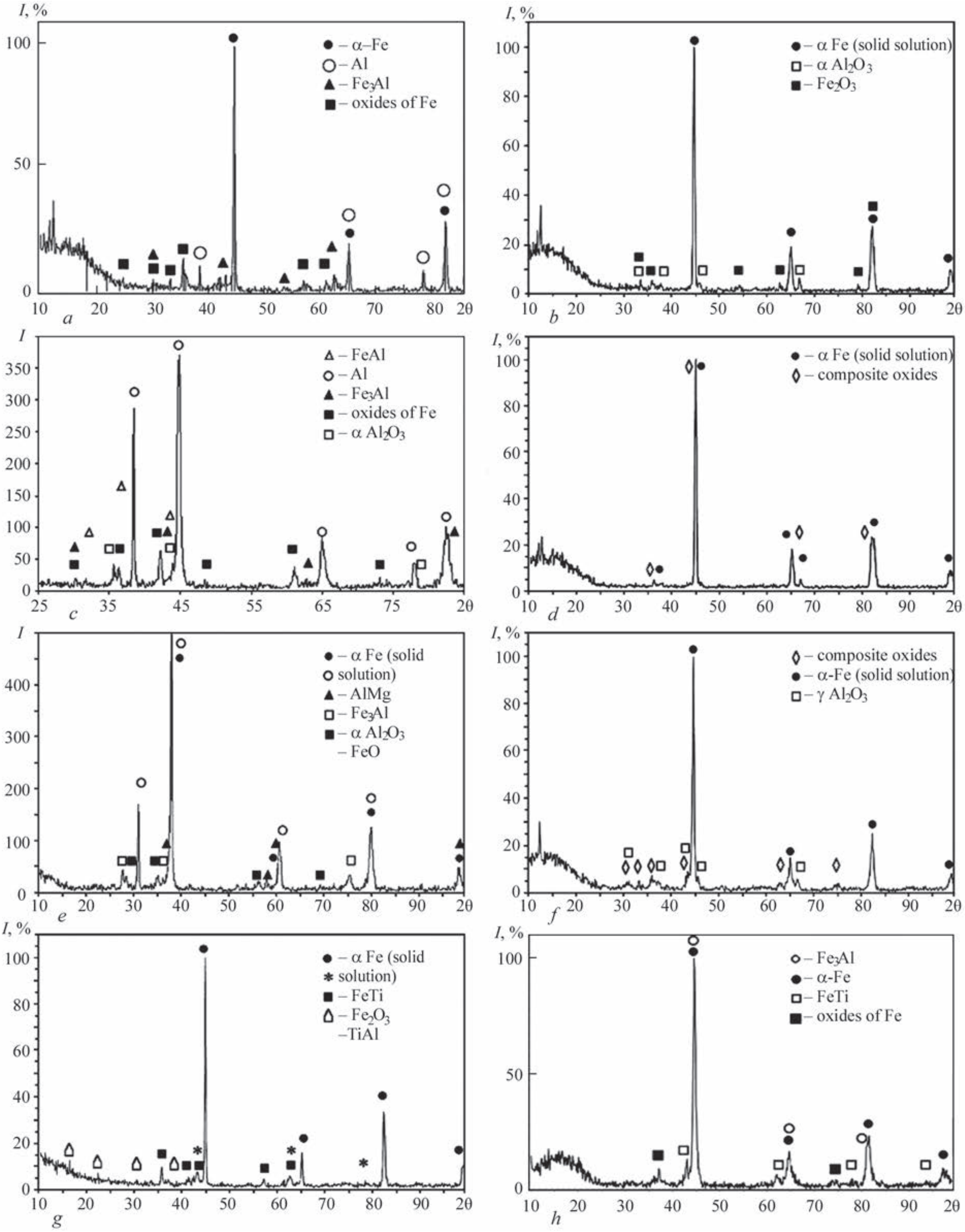


Fig re 6 X-ray patterns of plasma coatings: from mechanical mixtures Fe + AlCrZr (*a*), Fe + AlMg (*c*), Fe + AlMgLa (*f*), Fe + TiAl (*g*); from MChS-powders Fe–AlCrZr (*b*), Fe–AlMg (*d*), Fe–AlMgLa (*f*), Fe–TiAl (*h*)

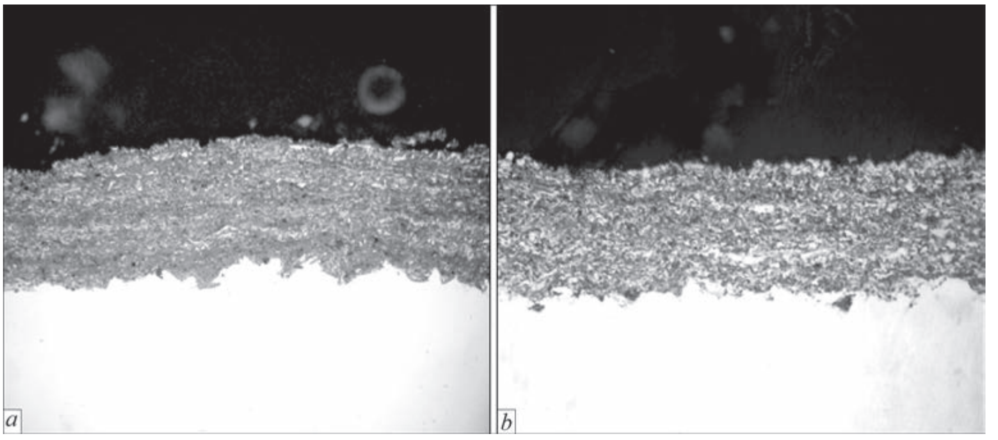


Figure 7 Microstructure of SPGAS-coatings from alloyed powders: *a* — Fe–AlCrZr; *b* — Fe–AlMg

coating layer. In all cases, in the coatings a solid solution of aluminium in iron and the intermetallic phase FeAl are formed. In the coating Fe_2A1_5 the intermetallic phase Fe_3A1 was also detected. In all coatings, a composite oxide $Fe_xA1_2O_4$ is present, and in the FeAl coating iron oxide FeO, aluminium oxide $A1_2O_3$, and in the coating Fe_2Al_3 aluminium oxide $A1_2O_3$ are present.

Measuring the microhardness of PS- and SPGAS-coatings showed that its value is higher in the coatings with MChS powder Fe_2A1_3, as compared to the coatings with MChS powders Fe_3A1 and FeAl (Table 3), which is agreed with the literature data, according to which the hardness of all intermetallic phases decreases with increasing iron content.

Figure 5 shows the microstructure of plasma coatings from the powders alloyed with Fe aluminides, produced by mechanical mixing and from MChS-powders using Al-alloys of AlCrZr, AlMg, AlMgLa, as well as titanium aluminide.

In the microstructure of plasma coatings produced in the case of spraying mechanical mixtures of powders Fe + AlCrZr, Fe + AlMg, Fe + AlMgLa and Fe + TiAl, separate particles of iron and particles based on aluminium are observed (Figure 5, *a*, *c*, *e*); in the case of spraying the coating from the mechanical mixture Fe + TiAl, no particles of titanium aluminide are observed in the coating (Figure 5, *g*).

When using powders of the systems Fe + AlCrZr, Fe + AlMg and Fe + AlMgLa produced by the MChS method, a dense lamellar structure is formed (Figure 5, *b*, *d*, *f*), and in the case of Fe−TiAl powder, spalling elements are observed (5−6 vol.%) (Figure 5, *h*), which indicates the presence of brittle phase inclusions in this coating.

The results of XRD of plasma coatings from alloyed powders (Figure 6, Table 3) showed that in the case of spraying mechanical mixtures of iron with aluminium alloys, as in the case of mixtures of iron with aluminium, the reaction of intermetallic phases formation does not develop noticeably. In the coatings, except for the expected phase of intermetallic Fe_3A1 (for Fe−AlCrZr, Fe−AlMg and Fe−AlMgLa compositions) or $Fe_{1-x}TiA1_x$ (for Fe−TiAl composition), solid solutions based on iron, aluminium, oxides of aluminium and iron were found.

The coatings from powders produced by MChS method from a mixture of iron with aluminium alloys AlCrZr, AlMg and AlMgLa, contained a large amount of oxides (up to 30 vol.%). Moreover, in addition to oxides of aluminium and iron, composite oxides were also detected, for example, $MgFeAlO_4$. Probably

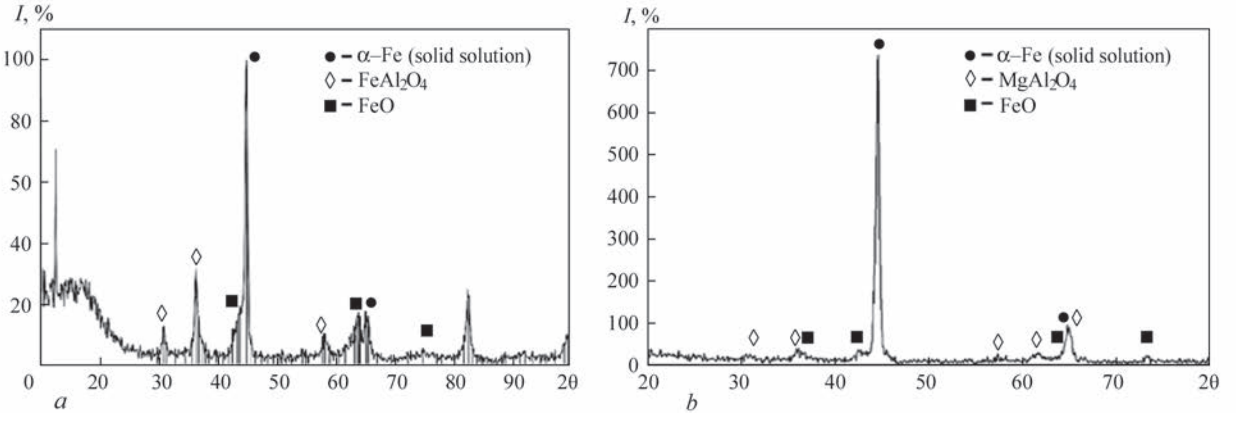


Figure 8 X-ray patterns of SPGAS-coatings: *a* — Fe−AlCrZr; *b* — Fe−AlMg

Tb le 4 Characteristics of PS and SPGAS-coatings of powders alloyed by iron aluminides produced by mechanical mixing and MChS method with the use of Fe- and Al-alloys

Powder		Coating		
Composition	Method of producing	Method of spraying	H_μ, MPa	XRD
86Fe + 14(Al1.5Cr1Zr) (wt.%)	Mechanical mixing	PS	Based on Fe – 3080±400 Based on Al – 620±100	α-Fe, Al, Fe_3Al, Fe_3O_4, FeO, Fe_2O_3 (Figure 6, *a*)
Fe–AlCrZr	MChS	PS	3830±630	Solid solution of Cr and Zr in Fe; γ-Al_2O_3, Fe_2O_3 (Figure 6, *b*)
		SPGAS	6180±740	Solid solution based on α-Fe, $FeAl_2O_4$, FeO, traces of Al_9Cr_4, $AlCr_2$, Cr_2Zr, $AlZr_2$ (Figure 8, *a*)
86Fe + 14(Al5Mg) (wt.%)	Mechanical mixing	PS	Based on Fe – 2800±460 Based on Al – 630±90	FeAl, Al, Fe_3Al, α-Al_2O_3, Fe_2O_3 (Figure 6, *c*)
Fe–AlMg	MChS	PS	3280±450	Solid solution of Al in α-Fe, $MgAl_2O_4$, $MgFeAlO_4$ (Figure 6, *d*)
		SPGAS	4460±740	Solid solution based on α-Fe, $MgAl_2O_4$, FeO, traces of Al_2Mg, MgO, MgO_2 (Figure 8, *b*)
86Fe + 14(Al5MgLa) (wt.%)	Mechanical mixing	PS	Based on Fe – 3080±400 Based on Al – 630±90	Solid solution of Mg in Al, α-Fe, Fe_3Al, α-Al_2O_3, FeO (Figure 6, *e*)
Fe–AlMgLa	MChS	PS	5040±780	Solid solution of Al and La in α-Fe, Fe_3O_4, Fe_2O_3, γ-Al_2O_3, $MgFeAl_2O_4$ (Figure 6, *f*)
61Fe+39(TiAl) (wt.%)	Mechanical mixing	PS	3670±870	α-Fe, FeTi, TiAl, Fe_2O_3 (Figure 6, *g*)
F–TiAl	MChS	PS	6910±1640	Fe_3Al, Fe, FeTi, Fe_2O_3, Fe_3O_4, FeO (Figure 6, *h*)

by this fact it is possible to explain the detection of phases based on FeAl and solid solutions of Al in Fe in the structure of the coating instead of the expected intermetallic based on Fe_3A1, containing alloying elements. Only in the coating of the system Fe–TiAl, Fe_3Al is the main phase. In addition to this phase, the coating contains FeTi and iron oxides.

Analysis of the microstructure of SPGAS-coatings (Figure 7) showed that during spraying of alloyed powders of the systems Fe–AlCrZr and Fe–AlMg produced by the MChS method, a dense lamellar structure is formed, in which the content of the oxide component is 60–65 and 30–35 vol.%, respectively.

Using X-ray diffraction phase analysis (Figure 8, Table 3) it was found that during SPGAS-spraying of powders Fe–AlCrZr and Fe–AlMg, produced by the MChS method, the reaction of intermetallic phases formation does not proceed, and in the coating solid solution based on iron, iron oxide FeO and composite oxides $FeAl_2O_4$ and $MgAl_2O_4$ are revealed (Table 3). In addition, in the coating Fe–AlCrZr traces of compounds $A1_9Cr_4$, $A1Cr_2$, Cr_2Zr and $AlZr_2$ were detected, and in the coating Fe–AlMg there are traces of AlMg compound and magnesium oxides MgO and MgO_2.

The microhardness of plasma coatings from the powders of alloyed iron aluminides (Table 4) produced by MChS is much higher as compared to the coatings from the mechanical mixture of Fe- and Al-alloys. The maximum value of microhardness (6910 MPa) was marked in the coating from the powder of the system Fe–TiAl produced by MChS due to the formation of FeTi intermetallic in the coating.

Analysis of the microhardness of SPGAS-coatings from the alloyed powders (Table 4) showed that in the coating Fe–A1CrZr it is 1.6 times higher than in the sprayed initial powder (3840 MPa, Table 1), and the microhardness of the coating Fe–AlMg almost does not differ from the microhardness of the initial powder due to the absence or limited development of the contact interaction of Fe and Al in the process of coating deposition. This is explained by the lower degree of oxidation of Fe–AlCrZr coatings (60–65 vol.%) as compared to Fe–AlMg coatings (30–35 vol.%).

Cn clusin s

The investigation of microstructure, microhardness and phase composition of plasma coatings from Fe-Al-powders produced by the MChS method, showed that in addition to the initial phase (Fe_3A1, FeAl and Fe_2Al_5) in the coatings to a greater or lesser extent oxides of Fe and Al are present, which is reflected on the value of microhardness (Fe_3A1 — 3630 MPa, FeAl — 4150 MPa and Fe_2Al_5 — 5200 MPa). When iron alloying elements are introduced into aluminides by using Al-alloys AlCrZr, AlMg and AlMgLa, microhardness of the coatings amounts to 3830, 3280 and 5040 MPa, respectively. Microhardness of the plasma coating Fe–TiAl (6910 MPa) increases 2 times in respect to the initial powder due to the formation of the intermetallic phase FeTi in the coating.

During plasma spraying of mechanical mixtures of iron with aluminium and iron with aluminium alloys, the reaction of intermetallic phases formation does not develop noticeably due to the lack of development of contact interaction of Fe and Al during the process of coating. The main phases in the coatings are the initial components of iron, aluminium and their oxides.

In the coatings produced by the method of supersonic plasma gas-air spraying intermetallic phases were not detected, the main phase is α-Fe (Al)-solid solution, which is obviously the result of a high rate of hardening the melt particles during the formation of intersections on the base surface.

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Received 30.06.2020