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STUDYING THE IMPACT OF DURATION OF TECHNOCHEMICAL SYNTHESIS OF NANOSTRUCTURE (Fe, Ti)$_3$Al POWDER ON CHARACTERISTICS OF PLASMA COATINGS

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ABSTRACT

The impact of duration of high-energy processing of the mixture of 60.8Fe + 39.2TiAl powders (wt.%) on structure, phase composition and mechanical characteristics of plasma intermetallic (Fe,Ti)$_3$Al coatings was studied. As powders, for plasma spraying powders of (Fe,Ti)$_3$Al intermetallic were used, which were produced by the method of mechanochemical synthesis (MChS) in a high-energy mill during 3 and 5 h. As a result of plasma spraying, coatings with a nanocrystalline structure with the size of crystallites of 60 and 45 nm are formed, respectively. It was shown that during spraying of MChS-powder, produced during 5 h, thin-lamellar coatings with a maximum thickness of lamellae of 23 μm are formed, whereas in the case of spraying MChS-powder produced during 3 h, the thickness of lamellae reaches 42 μm. At the same time, in the case of spraying MChS powder, produced during 5 h, more dense coatings are formed, the porosity of which is reduced by 2.3 % compared to the coating from MChS-powder, produced during 3 h. It was established that mechanical characteristics (hardness and modulus of elasticity) of a plasma coating were increased when using a powder produced by processing during 5 h. This allows predicting higher wear resistance of these coatings, operating in the conditions of wear unlike the case of spraying MChS-powder, produced during 3 h.

KEYWORDS: iron aluminides, mechanochemical synthesis, plasma spraying, nanostructural coatings, size of crystallites, mechanical characteristics

INTRODUCTION

One of the tasks of surface engineering at the present stage of the technology development is the development of protective coatings that provide protection of parts and units of equipment in the conditions of elevated operating temperatures and mechanical stresses and influence of aggressive and abrasive environments. Intermetallic based coatings can be attributed to perspective ones, in particular, those on the base of iron (Fe_3A1, FeAl) aluminides. These intemetallics are characterized by relatively low specific mass (5.51–6.65 g/cm^3), wear resistance, resistance to oxidative and sulfidating media at 1000 °C and higher. The areas of their potential application include heating elements, furnace reinforcement, heat exchanger pipes, sintered porous "gas–metal" filters, parts of valve systems of cars, components of installations operating with salt melts [1]. However, the practical use of iron aluminides is limited due to their tendency to hydrogen brittleness and low creep at temperatures of > 500 °C [2]. An improvement in mechanical characteristics of iron aluminides is achieved by their alloying (B, Si, Cr, Ti etc.), as well as by reduction in the grain size [3–5]. An increase in ductility during alloying can be achieved as a result of a decrease in the covalent bond component, creation of a favourable dislocation structure with a higher dislocation mobility, providing the action of more favourable sliding systems, change in crystalline or phase composition and structural state. The use of titanium as an alloying element helps to raise the temperature of phase transitions, increase the yield strength and improves tribotechnical properties of FeAl intermetallic [6].

To spray coatings based on iron aluminides, thermal spraying methods, namely high-velocity oxygen fuel (HVOF), plasma, electric arc and detonation spraying [7–10] are used. The use of powders produced by the method of mechanochemical synthesis (MChS) as sprayed materials allows forming coatings, which are homogeneous as to their chemical composition and have a nanocrystalline structure.

The aim of this work is studying the structure, phase composition and mechanical properties of plasma coatings when spraying MChS-powders of (Fe, Ti)$_3$A1 produced during different time of processing.

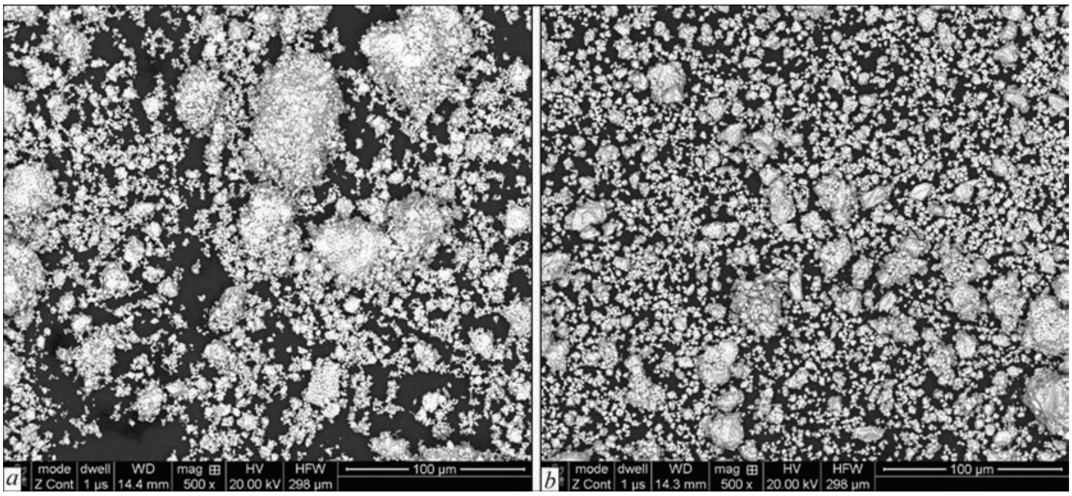

Figure 1. Appearance of powders produced by the MChS method of a mixture of iron and TiAl alloy powders during: *a* — 3; *b* — 5 h

Table 1. Characteristics of MChS (Fe, Ti)$_3$A1 powders for plasma spraying

Mixture components, wt.%	Processing time τ, h	Phase composition	Size of crystallites, nm	Particle size, μm		
				D_{10}	D_{50}	D_{90}
60.8Fe–39.2TiAl	3	(Fe, Ti)$_3$Al	25	6	17	41
	5		10	3	9	30

MATERIALS AND RESEARCH PROCEDURES

As materials for spraying, intermetallic nanostructure (Fe, Ti)$_3$A1 powders were used, produced by the method of mechanochemical synthesis of mixtures of iron and Ti37.5Al alloy powders. The MChS process was carried out in the planetary mill Activator 2SL during the processing time of 3 and 5 h. As was established in previous studies [11], after 3 h of processing, in the MChS product, an intermetallic (Fe, Ti)$_3$Al phase with crystallites of 25 nm is formed, and with an increase in the processing time to 5 h, the size of crystallites decreases to 10 nm and the size of powder particles is somewhat reduced. The appearance of powder particles after MChS is shown in Figure 1, the characteristics of powders is given in Table 1.

For the use of these powders, in the technology of plasma spraying, their conglomeration by mixing the resulting MChS products with a 5 % polyvinyl alcohol solution in water was carried out until producing a homogeneous suspension. The resulting suspension was dried with the subsequent wiping through a sieve and selecting particles of 40–80 μm.

Plasma spraying of coatings was carried out in the UPU-8M installation using the following parameters: $I = 500$ A, $U = 40$ V, $Q_{\mathrm{Ar+N_2}} = 25$ l/min, spraying distance — 120 mm. Preliminary investigations [9] showed the feasibility of using the mentioned plasma spraying parameters to form a fine-lamellar dense structure of coatings based on FeAl intermetallics.

Determination of element composition of coatings by the method of scanning electron microscopy (SEM) was carried out on the basis of an analytical complex consisting of a scanning electron microscope JSM-35 CF of JEOL Company (Japan) and energy dispersion spectrometer (model INCA Energy-350 of Oxford Instruments Company, Great Britain). X-ray diffraction phase analysis (XRD) of coatings was performed using DRON-3 diffractometer in CuK_α-radiation with a graphite monochromator at a step movement of 0.1° and a time of exposure at each point of 4 s with the subsequent computer processing of the obtained digital data. The phase identification was performed using the international database ICDD PDF-2 or PDF-4. The size of crystallites in the coatings was evaluated using the Debye–Scherrer formula:

$$d = \frac{K\lambda}{\beta\cos\theta},$$

where d is the average size of coherent scattering regions (domains, crystallites), which may be smaller or equal to the grain size; K is the dimensionless particle shape factor (Scherrer constant); λ is the wavelength of X-ray radiation; β is the reflex width at half maximum (in radians and in units 2θ); θ is the diffraction angle (Bragg's angle).

Mechanical characteristics (hardness H, modulus of elasticity E) of the coatings were determined by the microindentation method with the use of the Micron-Gamma device [12]. The value of characteristics was calculated automatically according to ISO 14577-1:2002 standard.

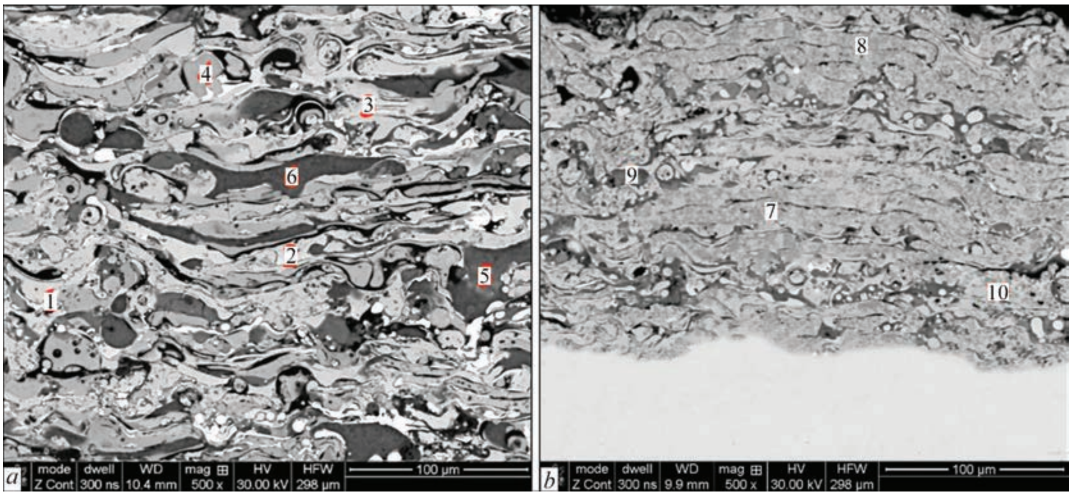

Figure 2. Microstructure of plasma coatings from nanostructural intermetallic (Fe, Ti)$_3$A1 powder, produced by the MChS method during 3 h (*a*) and 5 h (*b*)

RESEARCH RESULTS AND THEIR DISCUSSION

Metallographic analysis found that as a result of plasma spraying nanostructural powders of (Fe, Ti)$_3$Al intermetallic, the coatings with a characteristic layered structure (Figure 2, *a*, *b*) are formed. An increase in duration of mechanosynthesis of powders from 3 to 5 h leads to the formation of a structure with a smaller thickness of lamellae. In spraying MChS-powder (τ = 3 h), in the coating, lamellae of ~42 μm thick are present, whereas in spraying MChS-powder (τ = 5 h), the maximum thickness of lamellae is ~23 μm. This is associated with the presence of composite particles of up to 70 μm (Figure 1, *a*) in MChS-powder after 3 h of processing, as far as at the initial stages of the MChS-process, the process of "cold" welding of particles of initial powders with each other prevails. As the processing time increases to 5 h, between grinding and welding of particles, the equilibrium is achieved and the size of MChS-powder particles decreases.

It is noted that both types of coating are characterized by the presence of particles in the layer structure, unmolten in the plasma jet. A number of such particles in the coating of MChS-powder (τ = 3 h) is somewhat larger, which is also associated with the presence of particles of > 40 μm in the powder. This, in turn, leads to the formation of a coating with a slightly greater porosity. Thus, the porosity of the coating from MChS-powder (τ = 3 h) amounts to 6.8 ± 0.8 %, the coating from MChS-powder (τ = 5 h) is –4.5 ± 1.0 %.

According to the analysis of the chemical composition, bright lamellae of the coatings consist of the initial components containing oxygen ~2 wt.% (Table 2). In this case, in terms of chemical composition, the regions 1–4, 7, 8 (Figure 2) are close to (Fe, Ti)$_3$Al compound. A high content of oxygen (up to 20 wt.%) in individual microvolumes is caused by the presence of oxides formed by spraying coatings as a result of interaction of powder components with a plasma jet. Such lamellae in terms of their chemical composition correspond to the iron (FeO) oxide with a small amount of titanium and aluminium (spectra 5, 6, 9, 10 — Figure 2).

By means of energy dispersive analysis it was established that the time of the MChS process of powders affects the uniformity of elements distribution over the thickness of the coating (Figures 3, 4). In the coating sprayed from MChS-powder (τ = 3 h), the distribution of initial components is non-uniform. Here, oxygen is distributed in the form of interlayers between the metal lamellae (Figure 3). An increase in the time of MChS to 5 h allowed producing a coating with a more uniform distribution of both initial elements as well as oxygen over the thickness of the coating (Figure 4). This difference in the distribution of elements over the thickness of the coating is obviously associated with the distribution of elements in MChS-powders. When conducting the MChS process during 5 h, smaller particles are formed and the distribution of elements in the powder particles is averaged.

Table 2. Chemical composition of plasma coatings produced by spraying intermetallic (FeTi)$_3$A1 powder

Spectra	Chemical composition, wt.%			
	Fe	Ti	Al	O
1	59.93	28.79	10.31	0.97
2	54.99	30.87	12.61	1.53
3	54.48	31.05	13.24	1.23
4	49.33	36.36	12.39	1.92
5	71.66	8.35	1.17	18.82
6	69.98	7.71	2.23	20.08
7	62.4	24.97	11.69	0.94
8	62.63	22.22	13.8	1.35
9	56.06	19.57	6.98	17.4
10	78.01	5.54	8.05	8.4

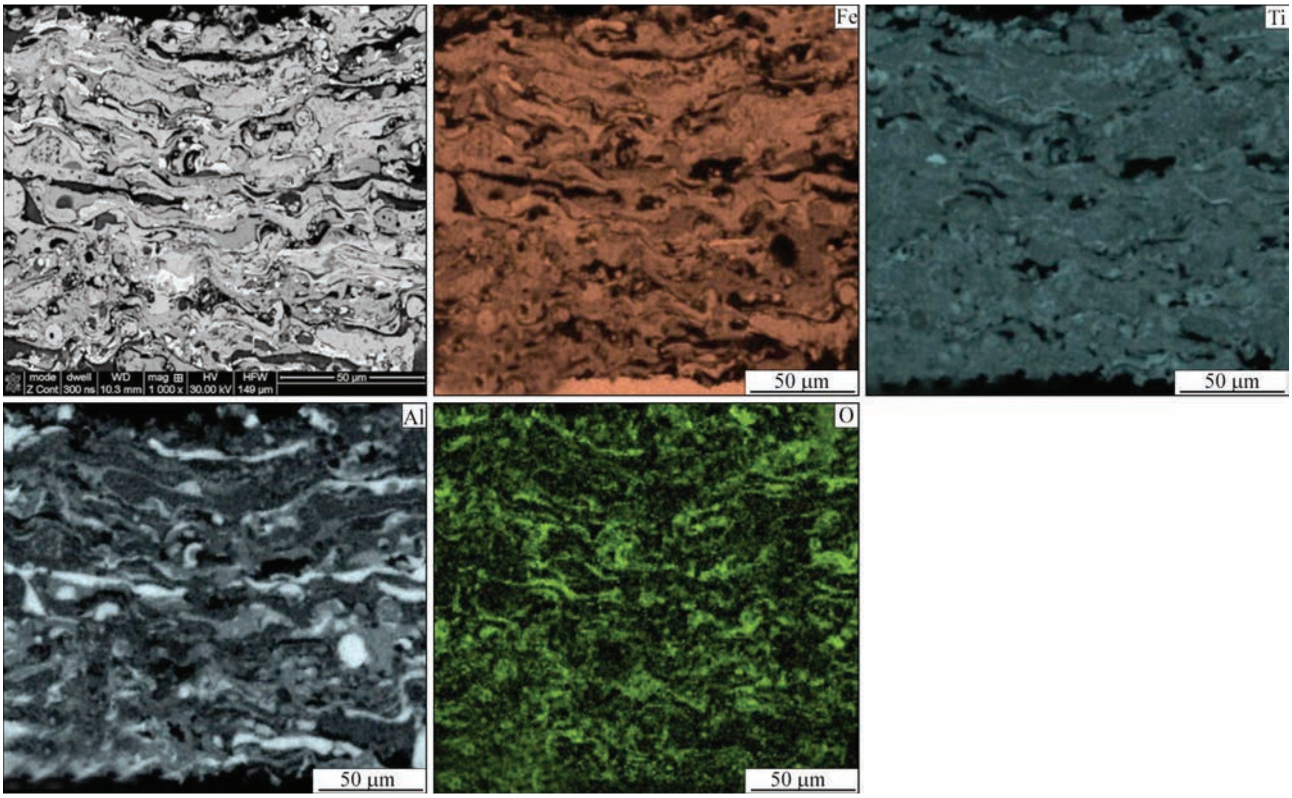

Figure 3. Distribution of elements in the plasma coating from intermetallic (Fe, Ti)$_3$A1 powder, produced by the MChS method during 3 h

As to their phase composition, the coatings do not differ significantly and in both cases the main phase in the coatings is (Fe, Ti)$_3$Al intermetallic (Figure 5). Except of the main intermetallic phase, in the coatings also the lower FeO oxide is identified, the appearance of which is associated with the interaction of iron in the plasma jet with oxygen of the environment. This is consistent with the results of chemical analysis of the

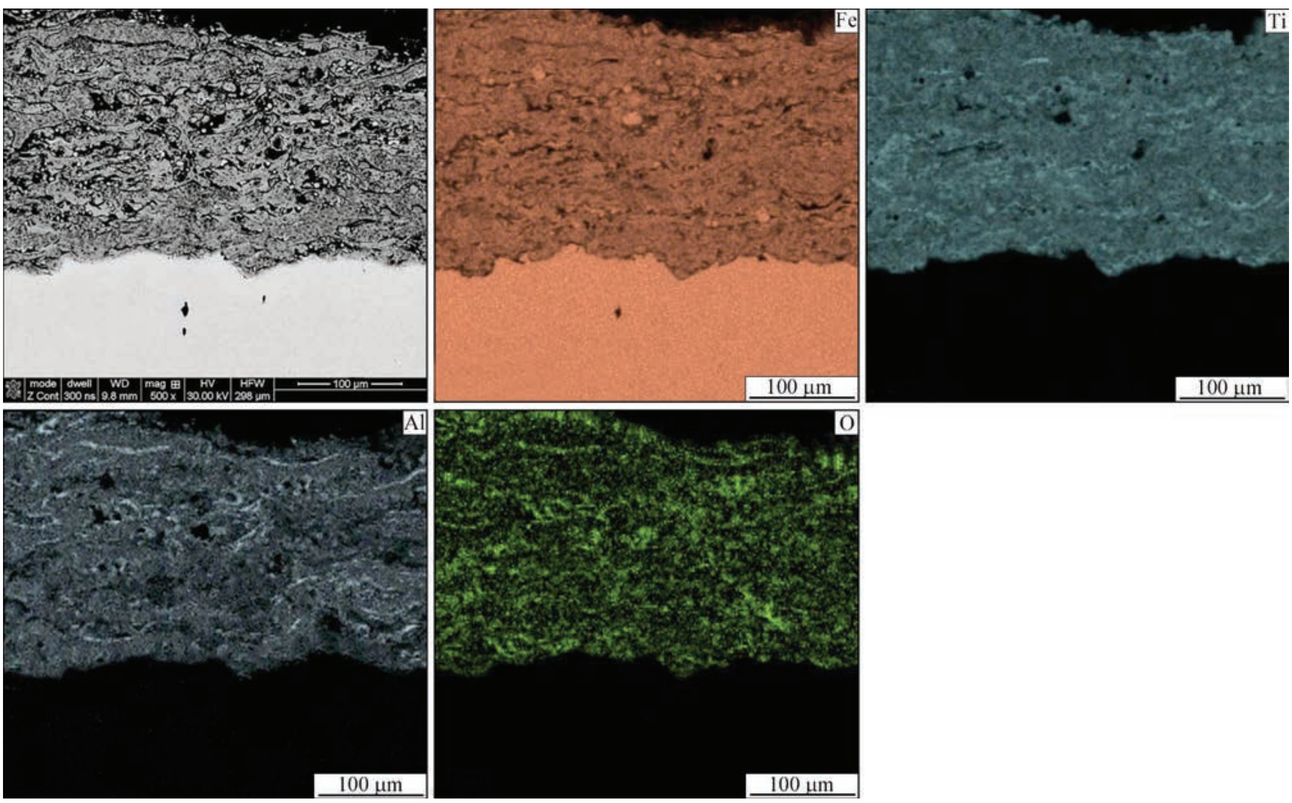

Figure 4. Distribution of elements in the plasma coating from intermetallic (Fe, Ti)$_3$Al powder, produced by the MChS method during 5 h

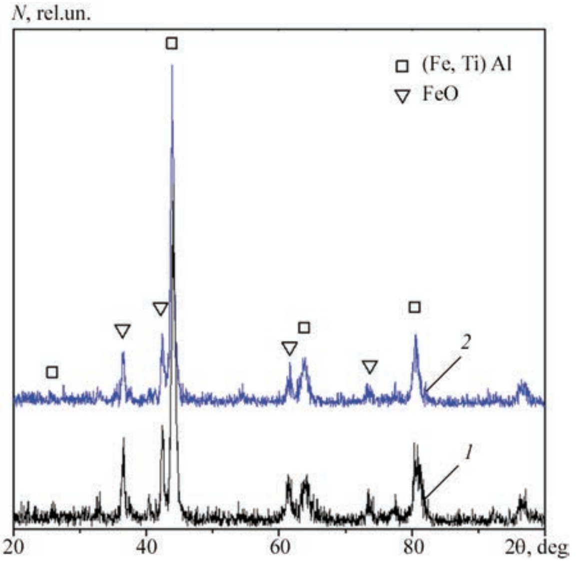

Figure 5. X-ray patterns of plasma coatings from intermetallic (Te, Ti)$_3$A1 powder, produced by the MChS method during 3 h (*a*) and 5 h (*b*)

coatings (Table 2). In the X-ray patterns, a decrease in the intensity of iron oxide peaks is observed when using MChS-powder produced during 5 h, which may indicate a lower amount of iron oxide in this coating.

Evaluation of CSR of the produced coatings from a nanostructural powder of (Fe,Ti)$_3$A1 intermetallic showed that as a result of powder melting in the plasma jet, followed by hardening on the base, the size of the crystallites of the coatings increased from 25 to 60 nm compared to initial powders in case of spraying powder produced by MChS during 3 h and from 10 to 45 nm during spraying powder produced by MChS during 5 h.

The results of determination of such mechanical characteristics of coatings as hardness (*H*) and modulus of elasticity (*E*) by the method of microidentation, are given in Table 3. Table also shows the ratio of H/E and H^3/E^2, which are indices of transition from elastic deformations to fracture (normalized hardness) and resistance to plastic deformation, accordingly.

The comparison of values of mechanical characteristics shows that values of hardness and modulus of elasticity of the coating produced from MChS-powder (τ = 5 h) exceed the coating from MChS-powder (τ = 3 h) by 1.8 and 1.7 times, respectively. The difference between the values of mechanical characteristics of the two coatings is predetermined, apparently, by their microstructure. Thus, the coating from MChS-powder (τ = 5 h) is characterized by a thin lamellar structure, a more uniform chemical composition and has a smaller size of crystallites, which contributes to an increase in mechanical characteristics compared to the coating from MChS-powder (τ = 3 h).

Table 3. Mechanical characteristics of plasma coatings from intermetallic (FeTi)$_3$A1 powder, produced by the MChS method during 3 and 5 h

MChS-powder	*H*, GPa	*E*, GPa	H/E	H^3/E^2
τ = 3 h	3	47	0.064	0.012
τ =5 h	5.4	80	0.068	0.025

According to the method of evaluation of the structural state of the material using H/E index, proposed by the authors of [12], produced coatings belong to nanostructural materials, since H/E index in both cases is within 0.05–0.09.

Such indices as H/E and H^3/E^2 are indicators of a coating resistance to fracture, which are often used as a criterion for evaluation of wear resistance of protective coatings [13–15]. As is seen from Table 3, as to H/E index, the coating from MChS-powder (τ = 3 h) is somewhat inferior to the coating from MChS-powder (τ = 5 h). Whereas the coating from MChS-powder (τ = 5 h) 2.1 times exceeds the coating from MChS-powder (τ = 3 h). The obtained data indicate that the coating sprayed using the powder produced by MChS during 5 h, has a higher wear resistance.

CONCLUSIONS

As a result of spraying nanostructural powders of (Fe, Ti)$_3$Al intermetallic produced by the MChS method during 3 and 5 h, nanostructural coatings are formed, the size of crystallites in which is 60 and 45 nm, respectively. The phase composition of powders is inherited by plasma coatings, the main phase in which is the intermetallic (Fe, Ti)$_3$Al phase with the presence of FeO iron oxide.

It is shown that an increase in time of MChS of (Fe, Ti)$_3$Al intermetallic powder from 3 to 5 h allows forming a coating in the conditions of plasma spraying with a thin lamellar structure and a uniform distribution of initial elements in the volume of the coating. In this case, the porosity of the coating decreases from 6.8 to 4.5 %.

Using the method of micro-indentation, it was found that according to such main indices of mechanical characteristics of a coating as hardness and modulus of elasticity, the coating from MChS-powder, produced during 5 h, exceeds the coating from MChS-powder, produced during 3 h by 1.8 and 1.7 times, respectively. The values of normalized hardness (H/E) and resistance to plastic deformation (H^3/E^2), which are the criteria of wear resistance, are also greater in the coating during spraying MChS-powder produced by processing during 5 h.

The carried out studies indicate the prospects of using mechanically synthesized powder of (Fe, Ti)$_3$Al intermetallic, produced at the time of processing initial mixtures during 5 h, for plasma spraying of coatings operating under wear conditions.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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