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of flux-cored wires.

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Existing techniques for applying

intermetallide layers are characterized by low productivity, difficulties associated with the maintenance and opera-

tion of technological equipment, as well as significant costs for the purchase of

materials for spraying. Therefore, modern science shows considerable interest

in the development of new, highly effective technologies to form intermetallide

coatings on the surface of articles. Such promising techniques include the tech-

nology of plasma-arc spraying (PAS)

the affordability of equipment and mate-

rials for coating. This paper reports a

study into the structure and proper-

ties of coatings obtained by flux-cored wire PAS, in which the steel sheath and

aluminum powder filler interact when

heated with the exothermic effect of

Fe<sub>3</sub>Al synthesis. The influence of tech-

nological parameters of PAS process on the structure and properties of Fe-Al

coatings was investigated by means of

mathematical planning of the exper-

iment. It was found that in all sam-

ples the main phase is an intermetallide

of the Fe<sub>3</sub>Al type. Tests for gas-abrasive wear resistance at room tempera-

ture showed that the wear resistance

of coatings exceeds the stability of steel

S235 by an average of 2 times. As a

result of studying the electrochemical properties in a 3-% aqueous solution

of NaCl and in a 0.5-% solution of

 $H_2SO_4$ , the score of corrosion resistance

for these media was determined, which

was, respectively, 4 and 5 (coatings

belong to the group of "resistant"). In this regard, the practical use of coat-

ings based on the Fe<sub>2</sub>Al intermetallide

is recommended for protection against

oxidation, corrosion, and gas-abrasive

wear of components and assemblies in

the heat power industry (heat exchanger pipes, catalytic converters, steam

Keywords: plasma-arc spraying,

flux-cored wires, intermetallide type

turbine blades, shut-off valves, etc.)

coatings, corrosion resistance

This technique has a number of significant advantages, namely high performance, relative simplicity, as well as MATERIALS SCIENCE

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# EFFECT OF THE TECHNOLOGICAL PARAMETERS OF PLASMA-ARC SPRAYING OF FLUX-CORED WIRE ON THE STRUCTURE AND PROPERTIES OF INTERMETALLIDE COATINGS BASED ON Fe<sub>3</sub>AI

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#### 1. Introduction

Transition metal aluminides (Ni, Fe, Ti) have some physical, mechanical, and corrosive properties, which distinguish them from other types of Ni-, Fe-, and Ti-alloys. The main characteristics of intermetallide alloys are high melting point, high thermal conductivity, low specific gravity, and high strength-to-density ratio. They also include

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resistance to oxidation at high temperatures (up to 1000 °C and above) and resistance in aggressive sulfur-containing environments [1, 2]. The properties of these materials make their use promising as a substitute for heat-resistant steels (AISI 316) and superalloys (INCOLOY 800), which are rapidly destroyed under corrosion conditions even at 700 °C.

Among intermetallide compounds, the researchers paid special attention to iron intermetallides, which began to be considered as substitutes for heat-resistant nickel alloys, the competitiveness of which in relation to these alloys is ensured by the availability and low cost of the main component – iron.

Iron aluminides Fe<sub>3</sub>Al and FeAl are among the most studied intermetallides due to their low specific gravity, high wear resistance, ease of processing, high resistance to oxidation and corrosion. These properties predetermined the areas of potential use of these alloys. These can be heating elements, heat exchange pipes, sintered porous "gas-metal" filters, parts of automotive fittings systems, as well as components of installations working with salt melts [3].

However, the disadvantages of iron intermetallides are their low ductility and resistance to shock at room temperature, as well as insufficient creep resistance in areas of moderate temperature [4]. In this regard, the manufacture of articles and structures from Fe-Al alloys by conventional metallurgical methods (casting, forging, rolling) is limited.

In world practice, a number of scientific and technical efforts are underway in the field of searching for new, highly effective technologies to form protective coatings, which include iron intermetallides, on the surface of articles. These coatings must have the above functional properties and provide effective protection against the destructive effects of external factors, namely mechanical and thermal loads, and aggressive gas environment. In addition, the gas-thermal spraying technique should ensure a reduction in material and production costs, make it possible to quickly and efficiently apply intermetallide coatings under production conditions to parts of complex shape.

The results of such studies are necessary in practice because the development of new high-performance techniques for the formation of intermetallide layers will help eliminate the existing difficulties associated with their application. Namely, the maintenance and operation of technological equipment for applying layers, low productivity, and significant costs for the purchase of materials for spraying.

That will significantly expand the application of Fe-Al coatings in industry by eliminating the problems associated with their manufacture using standard technologies (casting, forging, and rolling). The use of such intermetallide coatings instead of expensive heat-resistant nickel alloys operating under difficult corrosion conditions will help reduce the material and production costs of their manufacture.

#### 2. Literature review and problem statement

Work [5] provides a detailed review of the results of studies of intermetallide coatings applied by methods of high velocity oxygen fuel (HVOF) and plasma spraying (PS) using specialized powders. It was shown that HVOF and PS techniques provide high adhesion strength and microhardness of applied intermetallide Fe-Al coatings, as well as high resistance to erosion at elevated temperatures. However, for the case of PS, there is a significant porosity of the resulting coating. In [6], it was shown that HVOF coatings based on the Fe<sub>3</sub>Al intermetallide have better corrosion resistance in sulphuration and cementation atmospheres at 600 °C for 500 hours compared to steel 304 and alloy 800H. In addition, such coatings show adequate resistance to thermal shock and better resistance to erosion by quartz particles at 600 and 900 °C than the steel 316L. However, it should be noted that in both cases, expensive fine powders of 5,562 microns fraction obtained by the technology of gas spraying of the melt were used to spray Fe<sub>3</sub>Al coating on the IN718 alloy substrate. In [7], the possibility of forming layers with nanocrystalline Ni-Al structures using high velocity oxygen fuel of intermetallide powders is shown. It was found out that during spraying, dense intermetallide layers are formed with crystallite sizes of 20 nm and microhardness of the coating at the level of 5.4-6.1 GPa. Intermetallide powders obtained by mechanochemical synthesis of commercial aluminum powders (purity 99.7 % and particle size  $50-70 \,\mu\text{m}$ ) and nickel (purity 97.5 % and particle size 30–110  $\mu m)$  were used for spraying. In general, issues related to the economic feasibility of using these methods remain unresolved. The reasons for this may be objective difficulties associated with significant costs for the purchase of materials for spraying and, accordingly, the high cost of the coating. Also, it should be noted that the productivity of installations for HVOF is 2-10 kg/h. An option to overcome the corresponding difficulties may be the use of more economical technologies where instead of specialized powders, available materials are used - solid or flux-cored wires, as well as more efficient sources of heating, such as an electric arc.

This approach is used in [8], where the technique of Arc spraying (AS) of wire materials was used to obtain intermetallide coatings. The process of arc spraying of a pair of wires "Steel AISI 1065 aluminum AS-AW 1050A" was investigated. It was shown that with the AS of heterogeneous solid wires Fe and Al, the coating composition, depending on the process parameters, can be a mechanical mixture of Al and Fe particles without the formation of intermetallides. An option to overcome these difficulties is the formation of a layer of intermetallide coating, followed by its subsequent heat treatment. In [9], it was shown that the heat treatment of the Fe-Al coating carried out at a temperature of 650 °C for two hours followed by cooling in water leads to the formation of Fe<sub>2</sub>Al<sub>5</sub>, Fe<sub>3</sub>Al, and FeAl intermetallides in the coating. However, this requires significant economic costs for additional technological heat treatment operations and significantly complicates the process of forming intermetallide layers on the surface of articles using the AS technique. An option to overcome these difficulties may be the development of appropriate compositions of flux-cored wires, which, due to the selection of powder filler, will make it possible to obtain the necessary coating structure. Thus, in [10], it is shown that the use of flux-cored wires (FWs) has become a new stage in the development of arc metallization technology, which began to be used for coatings of a wide range of functional purposes (tribotechnical, corrosion-resistant, etc.). That has made it possible to obtain coatings with different types of structure (composite, nanocrystalline) and a wide range of functional purposes, resistant to wear and corrosion in various environments and heating temperatures. However, with AS of flux-cored wires, a significant part of the sprayed particles intensively interacts with oxygen in the air, which leads to the formation of oxides in the coating structure. This, in

turn, impairs the mechanical and wear-resistant characteristics of the coating and does not fully make it possible to realize the potential inherent in them. An option to overcome the corresponding difficulties is the use of plasma-arc spraying technology of conductive wires, where the melting and dispersion of flux-cored wire material occurs in argon (inert) plasma [11]. This makes it possible to significantly reduce the degree of oxidation of particles and obtain a coating structure that is similar in chemical composition to or coincides with the material of the wire.

Thus, the accumulated experience in the development of practical technologies for gas-thermal spraying of coatings based on Fe<sub>3</sub>Al and FeAl intermetallides includes coatings obtained by various spraying techniques, except for plasma-arc. These methods include plasma, detonation, high velocity oxygen fuel, which give the best results in terms of wear resistance and corrosion resistance of coatings based on intermetallides of the Fe-Al system. However, these techniques have significant drawbacks - low productivity and high cost of the process. Separately, it is necessary to distinguish the electric arc method of spraving heterogeneous solid and flux-cored wires. Cheaper AS technology of heterogeneous solid wire does not ensure the formation of intermetallide layers in the coating and requires further heat treatment of articles, which greatly complicates the process of coating.

Therefore, there is an urgent need to devise new high-performance and technological techniques for forming intermetallide layers on the surface of parts. Such promising methods include the technology of plasma-arc spraying of conductive flux-cored wires, which requires further research into the formation of layers with an intermetallide structure.

#### 3. The aim and objectives of the study

The aim of this work is to study the influence of technological parameters of PAS process on the structure and operational properties of intermetallide coatings based on Fe<sub>3</sub>Al, obtained by spraying a flux-cored wire consisting of a tubular steel sheath and a filler made of aluminum powder.

To accomplish the aim, the following tasks have been set:

 to investigate the structure and phase composition of the obtained coatings under different modes of flux-cored wire PAS;

 to investigate the microhardness and porosity of the obtained coatings under different modes of flux-cored wire PAS;

- to determine the indicators of heat resistance, gas-abrasive wear resistance at elevated temperatures, corrosion resistance of the obtained intermetallide Fe-Al plasma coatings, to formulate recommendations for their use for protection against oxidation, corrosion, and wear.

#### 4. The study materials and methods

The object of our research is the process of plasma-arc spraying of flux-cored wire. The subject of the study is the influence of technological parameters of the plasma-arc spraying process on the structure and properties of the resulting coatings.

The choice of materials for this study was based on the fact that the selected composition of flux-cored wire should provide an intermetallide structure in the coating. The main requirements for the coating, in addition to technological characteristics (density, low porosity, high adhesion strength, etc.), were to ensure heat resistance, corrosion resistance, and high resistance to wear. In the study, the following assumption was accepted – the flux-cored wire steel sheath and aluminum powder filler will interact when heated with the exothermic effect of synthesis until an intermetallide of the Fe<sub>3</sub>Al type is formed.

Fe-Al coatings were sprayed at the RLAZER 30 PL-W installation (Ukraine) [11], which is designed to apply wear-resistant and corrosion-resistant coatings, as well as to restore worn parts of machines by spraying electrically conductive materials. The spraying process is carried out according to the "wire-anode" scheme.

Argon and compressed air are used as working gases. In the plasma torch, the arc passes between the tungsten cathode blown by argon and the consumable conductive wire, which is continuously fed into the arc behind the cut of the plasma torch nozzle. The high-speed flow of associated gas (air) emanating from the annular gap between the nozzles of the plasma torch provides compression and acceleration of the plasma jet, as well as protection of the sprayed material from oxidation. The scheme of operation of the plasma torch with the external "wire-anode" is shown in Fig. 1 and described in works [12–14].

The main operating parameters of the RLAZER 30 PL-W installation are as follows [11]:

- power consumption, kW no more than 30;
- operating current adjustment range, A 100-250;
- operating voltage adjustment range, V 30-80;
- consumption of associated gas (air) 0.6 MPa,  $nm^3/h 50$ ;
- argon consumption at a pressure of 0.1 MPa, nm/h 2;
- wire feed rate, m/min 5–15;
- cooling of the plasma torch air.

A flux-cored wire (FW) of the Fe-Al system was made in a shell of AISI 1008 steel with a thickness of 0.4 mm, filled with Al powder (bulk density of  $1.3 \text{ g/cm}^3$ ) with a filling degree of 25–27 vol. %, FW has a composition of 86 wt. % Fe+14 wt. %Al, which corresponds to an intermetallide of the Fe<sub>3</sub>Al type.



Fig. 1. Installation PLAZER 30 PL-W for plasma-arc spraying of conductive "wireanode" [14]: *a* – general view of the plasma torch; *b* – process diagram

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The study of the coating structure was carried out by metallography methods using the Neophot-32 microscope (Germany). The phase composition of the coating was investigated by X-ray diffraction analysis (XSA) on the DRON-3 diffractometer in CuK $\alpha$ -radiation with a graphite monochromator. The study of the chemical composition of the coating was performed by X-ray structural microanalysis (XSMA) in the CAMEBAX installation (Great Britain). The porosity of the coating was measured on the OM-NIMET device, and the microhardness was measured on the LECO device at a load of 50 g.

The heat resistance of coatings separated from the base was determined using a Q-1500D derivatograph in air when heated to 1000  $^{\circ}$ C at a speed of 10  $^{\circ}$ C/min according to the characteristic of the intensity of change in the specific increase in the mass of the sample.

Tests of coating thickness under gas-abrasive wear conditions for mass loss under the influence of a corundum jet with a dispersion of  $850-1000 \,\mu\text{m}$  were carried out at 20 and 550 °C. The abrasive wear resistance of coatings was investigated when rubbing against a non-rigidly fixed abrasive.

The test for gas-abrasive wear of the sprayed coating was carried out according to the ASTM G7695 standard methodology. The sample was heated by a burner flame to a temperature of 550...600 °C. After reaching the required level of sample temperature, when compressed air was supplied, we began to conduct tests. The following parameters were selected for the test: compressed air pressure, 0.5 MPa; nozzle diameter 8 mm; gas abrasive flow rate (calculated) – 200 m/s. Gas-abrasive jet was formed by supplying a crumb of electro corundum of normal grade 14 A with a grain of 90 F according to the main particle size range of 150...180  $\mu$ m with a microhardness of 16.6 Pa. For each test, fresh abrasive material weighing 600 g was used, the distance from the nozzle cut to the sample surface was 50 mm, the angle of inclination (angle of attack) was 10°.

Testing for abrasive wear resistance of coatings during friction on a non-rigidly fixed abrasive was carried out according to the ASTM G6594 standard methodology. The sample was based and clamped in special fastening equipment. With the help of a spring, the force of pressing the sample to the wheel was adjusted. As an abrasive material for testing, an electro corundum with a grain of F 22 with a main fraction of 850...1000  $\mu$ m was used. For each test, a fresh abrasive material weighing 365 g was applied.

Coating wear was determined by the following formula:

 $I_{w} = \Delta i/m, \, \mu m/kg,$ 

where  $I_{w}$  is the intensity of wear,  $\mu m/kg$ ;

 $\Delta i$  – linear wear of the coating,  $\mu$ m;

m – abrasive consumption for testing, kg.

The study of the electrochemical properties of coatings was carried out by the potential-dynamic method in the potentiostat P-5827M in the media of aqueous solutions of 3 % NaCl and 0.5M H<sub>2</sub>SO<sub>4</sub>. As a characteristic of corrosion resistance, weight (W, g/cm<sup>2</sup>·h) and deep (P, mm/h) corrosion characteristics were calculated, and the service life of the protective coating ( $\tau$ , years) was determined. To compare the characteristics of corrosion resistance of the coating, we used a ten-point scale according to GOST 308–85.

When selecting factors for analyzing the processes of gas-thermal spraying, a set of constant factors is usually used – the main parameters of the mode. These factors include current (I) and voltage (U) of the power supply, the flow rate and composition of working gases, powder consumption, and spraying distance. However, in the case of GTN technologies based on the method of arc spraying of wire materials, such as AS and PAS, it is necessary to ensure stable combustion of the working arc. To form a continuous and stable arc, it is necessary to equalize the wire feed rate ( $W_{wize}$ ) at the rate of its melting, providing a constant arc length. Thus, the factors of current and wire feed rate are interdependent when choosing their values. The characteristic of their interdependence can be designated as  $K_g=I/W_{wize}$ , which in each case depends on the characteristics of the wire (composition, diameter, melting point, etc.). It is determined by the empirical dependence of the combustion stability of the arc on the relationship I and  $W_{wize}$ .

In this regard, the factors selected for inclusion in the plan of the experiment on spraying Fe-Al-coating using PAS, as well as the matrix of the experiment, are given in Tables 1–3.

The value  $K_g$  of the characteristic under the conditions of the PAS Fe-Al coating was established as a result of previous experiments with the determination of the combination of current, wire feed rate, and process stability; it was equal to  $180/4.8=230/6\approx38$ .

The factors chosen were spraying distance -h, air flow rate  $-Q_{air}$  current strength -I, wire feed rate  $-W_{wize}$ .

To analyze the coating formation process during PAS spraying of Fe-Al wire and select the spraying mode, the method of mathematical planning of the experiment using a complete factor experiment  $2^3$  was used. This made it possible to investigate the structure and determine the properties of Fe-Al coatings sprayed by the PAS method.

The matrix of the experiment is given in Table 1.

| P                 |   |          |                |          |                  |  |  |  |  |
|-------------------|---|----------|----------------|----------|------------------|--|--|--|--|
| Experiment number |   | Factor   |                |          |                  |  |  |  |  |
|                   |   | $X_1(h)$ | $X_2(Q_{air})$ | $X_3(I)$ | $X_4 (W_{wize})$ |  |  |  |  |
|                   | 1 | _        | _              | _        | _                |  |  |  |  |
| Block No. 1       | 2 | +        | _              | _        | _                |  |  |  |  |
|                   | 3 | _        | +              | _        | _                |  |  |  |  |
|                   | 4 | +        | +              | _        | _                |  |  |  |  |
|                   | 5 | -        | —              | +        | +                |  |  |  |  |
| Block No. 2       | 6 | +        | —              | +        | +                |  |  |  |  |
|                   | 7 | _        | +              | +        | +                |  |  |  |  |
|                   | 8 | +        | +              | +        | +                |  |  |  |  |

#### Experiment matrix

Table 2 gives the values of factors depending on their level.

Table 2

Table 1

Factor levels

| Level | $X_1$ , $h$ , mm | $X_2$ , $Q_{air}$ , m <sup>3</sup> /h | <i>X</i> <sub>3</sub> , <i>I</i> , A | $X_4, W_{wize}, m/min$ |
|-------|------------------|---------------------------------------|--------------------------------------|------------------------|
| +     | 220              | 45                                    | 230                                  | 6.0                    |
| -     | 150              | 40                                    | 180                                  | 4.8                    |
| 0     | 185              | 42.5                                  | -                                    | _                      |

Table 3 gives a plan of experiments for Fe-Al coating PAS. Studies of microstructure, phase composition, determi-

nation of microhardness and porosity of coatings on samples were performed for each of the 8 experiments indicated in Table 3. Their purpose was to assess the influence of the

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selected parameters of PAS on the structure and properties of the resulting Fe-Al coating.

Plan of the Fe-Al coating PAS experiment

Table 3

| Emeriment |               | Characteristics                         |              |                                    |       |                |            |
|-----------|---------------|---|--------------|------------------------------------|-------|----------------|------------|
| number    | <i>h</i> , mm | Q <sub>air</sub> ,<br>m <sup>3</sup> /h | <i>I</i> , A | <i>W<sub>wize</sub></i> ,<br>m∕min | $K_t$ | K <sub>w</sub> | $K_{\tau}$ |
| 1         | 150           | 40                                      | 180          | 4.8                                | 4.5   | 37.5           | 3.75       |
| 2         | 220           | 40                                      | 180          | 4.8                                | 4.5   | 37.5           | 4.9        |
| 3         | 150           | 45                                      | 180          | 4.8                                | 5.75  | 37.5           | 3.3        |
| 4         | 220           | 45                                      | 180          | 4.8                                | 5.75  | 37.5           | 3.75       |
| 5         | 150           | 40                                      | 230          | 6.0                                | 5.11  | 38.3           | 3.75       |
| 6         | 220           | 40                                      | 230          | 6.0                                | 5.11  | 38.3           | 5.6        |
| 7         | 150           | 45                                      | 230          | 6.0                                | 5.11  | 38.3           | 3.3        |
| 8         | 220           | 45                                      | 230          | 6.0                                | 5.11  | 38.3           | 4.9        |

To assess the course of the processes occurring during the formation of a jet of particles that make up the FeAl coating layer, sets of factors have been formed. These factors are the qualitative characteristics of individual phenomena of the spraying process, so they were calculated for each of the 8 experiments conducted within the framework of the selected experiment plan (Table 3):

1. Specific heat capacity per unit of sprayed material:  $K_w = I/W_{wize}$ , [A·min/m] (a characteristic of the stage of melting of wire in the arc zone).

2. Specific heat capacity of a unit of working gas volume:  $K_t=I/Q_{ain}$  [A·h/m<sup>3</sup>] (a characteristic of the particle heating process).

3. Estimation of the duration of stay of particles in the volume of the jet:  $K_{\tau}=h/Q_{ain}$  (a characteristic that takes into account the duration of the particle stay in the jet volume).

The results of the experiment plan were separated in two blocks according to the current value (180 and 230 A).

#### 5. Results of investigating the properties and structure of Fe-Al coatings

## 5. 1. Investigation of microstructure and phase composition of coatings

Fig. 2, 3 show the microstructure and RFA of the coatings, which are obtained by the method of Fe-Al flux-cored wire PAS.

Fig. 4 shows the XSMA analysis of coatings that are obtained by the method of Fe-Al flux-cored wire PAS.



Fig. 2. Investigating the microstructure of coatings obtained by plasma-arc spraying using Fe-Al flux-cored wire:
 a - microstructure of coating mode No. 1; b - microstructure of coating mode No. 2; c - microstructure of coating mode No 3;
 d - microstructure of coating mode No. 4; e - microstructure of coating mode No. 5; f - microstructure of coating mode No. 6;
 g - microstructure of coating mode No. 7; h - microstructure of coating mode No. 8



Fig. 3. Investigating the phase composition of coatings obtained by plasma-arc spraying using Fe-Al flux-cored wire: *a* – radiograph of coating No. 1; *b* – radiograph of coating No. 2; *c* – radiograph of coating No. 3; *d* – radiograph of coating No. 4; *e* – radiograph of coating No. 5; *f* – radiograph of coating No. 6; *g* – radiograph of coating No. 7; *h* – radiograph of coating No. 8

Table 4

| Spectrum | Spectrum | <b>F</b> ' • 4 |       | Chemical com | position, % b | y weight |       | D 11.1.1                       |
|----------|----------|----------------|-------|--------------|---------------|----------|-------|--------------------------------|
| number   | type*    | F1g. 4         | Fe    | Al           | 0             | Mn       | Total | Principal phase                |
| 1        | 1        | a              | 98.24 | -            | 1.23          | 0.53     | 100   | Base                           |
| 2        | 1        | а              | 80.98 | 14.4         | 4.62          | -        | 100   | Fe <sub>3</sub> Al             |
| 3        | 1        | а              | 80.08 | 12.98        | 6.93          | -        | 100   | Fe <sub>3</sub> Al             |
| 4        | 1        | a              | 79.17 | 14.47        | 6.36          | -        | 100   | Fe <sub>3</sub> Al             |
| 5        | 1        | а              | 81.45 | 13.25        | 5.3           | -        | 100   | Fe <sub>3</sub> Al             |
| 6        | 2        | а              | 94.76 | 4.28         | 0.96          | -        | 100   | Fe(Al)                         |
| 7        | 2        | a              | 81.62 | 17.23        | 1.15          | -        | 100   | Fe <sub>3</sub> Al             |
| 8        | 2        | а              | 89.38 | 9.19         | 1.43          | -        | 100   | Fe(Al)                         |
| 1        | 1        | b              | 99.27 | _            | _             | 0.73     | 100   | Base                           |
| 2        | 1        | b              | 87.58 | 12.42        | -             | -        | 100   | Fe <sub>3</sub> Al             |
| 3        | 1        | b              | 84.98 | 15.02        | _             | -        | 100   | Fe <sub>3</sub> Al             |
| 4        | 1        | b              | 85.66 | 14.34        | _             | -        | 100   | Fe <sub>3</sub> Al             |
| 5        | 1        | b              | 87.36 | 12.64        | -             | -        | 100   | Fe <sub>3</sub> Al             |
| 6        | 2        | b              | 75.94 | -            | 23.53         | 0.52     | 100   | FeO                            |
| 1        | 2        | С              | 63.23 | 5.13         | 31.64         | _        | 100   | Mixture of oxides Al and Fe    |
| 2        | 2        | С              | 77.27 | 21.25        | 1.48          | -        | 100   | Fe <sub>3</sub> Al+Al          |
| 1        | 2        | d              | 72.61 | 27.39        | _             | _        | 100   | Fe <sub>3</sub> Al+Al          |
| 2        | 2        | d              | 72.23 | 27.77        | _             |          | 100   | Fe <sub>3</sub> Al+Al          |
| 3        | 2        | d              | 83.08 | 16.92        | _             | _        | 100   | Fe <sub>3</sub> Al             |
| 4        | 2        | d              | 2.29  | 52.73        | 44.98         | _        | 100   | Al <sub>2</sub> O <sub>3</sub> |

Results of Fe-Al coatings XSMA

Note: \*: 1 – scanning the site with averaging the contents of elements over the entire thickness; 2 – measurement of individual structural components with an electron probe



а



h





Fig. 4. Electron micrographs of typical areas of FeAl coatings made by plasma-arc spraying of Fe-Al flux-cored wire (for the chemical composition of the spectra, see Table 4): a – solid solutions of aluminum in iron (spectra 6, 8); b – iron oxide of composition FeO (spectrum 6); c – locations of oxide phases (spectrum 1); d – aluminum oxide Al<sub>2</sub>O<sub>3</sub> (spectrum 4)

Our studies make it possible to establish the structure and phase composition of the coatings formed in the process of plasma-arc spraying.

## 5.2. Investigation of microhardness and porosity of coatings

We studied the influence of process parameters on the microhardness and porosity of Fe-Al coatings. The summary results of the analysis of phase composition, porosity, and microhardness are given in Table 5.

To establish the qualitative influence of the selected parameters on microhardness (HV), a regression analysis of experimental data was carried out and the regression equation was built for two experimental blocks differing in an arc current - 180 A and 230 A, and a wire feed speed - 4.8 and 6.0 m/min:

- block 1 180/4,8: HV`=3851+2,3h-32O;
- block 2 230/6,0: HV<sup>\*</sup>=4435-2,4h-26 $\widetilde{O}$ ;
- block 1 180/4,8: HV'=3851+2,3h-32Q;
- block 2 230/6.0: HV"=4435-2.4h-26Q.

Table 6 gives a comparison of the results of microhardness calculation.

The studies allow us to determine the influence of the technological parameters of the process on the microhardness and porosity of the coating formed in the process of plasma-arc spraying. It is shown that the difference between experimental and calculated data is no more than 1 %.

Table 5

#### Characteristics of the structure and properties of Fe-Al coatings obtained by plasma-arc spraying

| Exper-<br>iment | Spray mode<br><i>h/Q<sub>air</sub>/I/W<sub>wize</sub></i> , | Microhardness,<br>HV <sub>0.05</sub> , MPa        |       | Phase composition  | Poros-<br>ity, % |
|-----------------|---|---|-------|--|------------------|
| number          |   | $H_{\mu}^{\text{mean}} \mid H_{\mu}^{\text{max}}$ |       |  | by voi.          |
| 1               | 150/40/180/4.8  | 2930±<br>±848                                     | 6795  | Fe <sub>3</sub> Al, α-Fe, γ-Fe, Al traces<br>of FeO  | 3.5              |
| 2               | 220/40/180/4.8  | 3058±<br>±721                                     | 9887  | Fe <sub>3</sub> Al, α-Fe, γ-Fe, Al traces<br>of FeO  | 3–4              |
| 3               | 150/45/180/4.8  | 2742±<br>±514                                     | 3575  | Fe <sub>3</sub> Al, $\alpha$ -Fe, $\gamma$ -Fe, Al, Fe <sub>3</sub> O <sub>4</sub> ,<br>Al <sub>2</sub> O <sub>3</sub> , traces of FeO, Fe <sub>2</sub> O <sub>3</sub><br>( $\alpha\gamma$ >2) | 2-2.5            |
| 4               | 220/45/18/4.8   | 2927±<br>±552                                     | 13100 | Fe <sub>3</sub> Al, α-Fe, γ-Fe, Al, Fe <sub>3</sub> O <sub>4</sub> ,<br>traces of Al <sub>2</sub> O <sub>3</sub> , FeO   | 2-2.5            |
| 5               | 150/40/230/6.0  | 3024±<br>±761                                     | 4635  | Fe <sub>3</sub> Al, α-Fe, γ-Fe, Al, traces of<br>Al <sub>2</sub> O <sub>3</sub> , FeO, Fe <sub>3</sub> O <sub>4</sub>  | 1.5              |
| 6               | 220/40/230/6.0  | 2888±<br>±766                                     | 4418  | $\begin{array}{l} {\rm Fe_3Al,  \alpha\text{-}Fe,  \gamma\text{-}Fe,  Al,  Fe_3O_{4,}} \\ {\rm Al_2O_3  (\gamma \!\!>\!\! \sim \!\! \alpha)} \end{array}$                                      | 2–3              |
| 7               | 150/45/230/6.0  | 2930±<br>±656                                     | 4221  | Fe_3Al, $\alpha$ -Fe, $\gamma$ -Fe, Al, Fe_3O <sub>4</sub> , Al <sub>2</sub> O <sub>3</sub>  | 2                |
| 8               | 220/45/230/6.0  | 2723±<br>±880                                     | 5807  | $\begin{array}{c} \mathrm{Fe_3Al,} \ \alpha \mathrm{-Fe}, \ \gamma \mathrm{-Fe}, \ \mathrm{Al}, \ \mathrm{Fe_3O_{4,}} \\ \mathrm{Al_2O_3,} \ \mathrm{Fe_2O_3} \end{array}$                     | 2                |

Table 6

#### Results of experiments for PAS of Fe-Al coatings

| Experiment |       | HV, MF | a      | Experiment | HV, MPa |       |          |  |
|------------|-------|--------|--------|------------|---------|-------|----------|--|
| No.        | Exp.  | Est.   | Δ      | No.        | Exp.    | Est.  | Δ        |  |
| 1          | 2930  | 2916   | -14    | 5          | 3024    | -3035 | +11      |  |
| 2          | 3058  | 3077   | +19    | 6          | 2888    | +2867 | -21      |  |
| 3          | 2742  | 2756   | +14    | 7          | 2930    | -2905 | -25      |  |
| 4          | 2927  | 2917   | -10    | 8          | 2723    | 2737  | +14      |  |
| Σ          | 11657 | 11666  | _      | Σ          | 11565   | 11544 | —        |  |
| Mean       | 2914  | 2916   | +33-24 | Mean       | 2891    | 2886  | +25 - 40 |  |

# 5.3. Studying the heat resistance, wear resistance (abrasive, gas-abrasive), and corrosion resistance

We investigated the FeAl coatings obtained under modes 5, 7, and 8 in order to determine their heat resistance,

wear resistance (abrasive, gas-abrasive), and corrosion resistance. The heat resistance of coatings separated from the base was measured, where the main phase is the intermetallide  $Fe_3Al$  (Fig. 5).



Fig. 5. Dependence of change in the sample mass on the test temperature for coatings obtained by plasmaarc spraying (modes 5, 7, 8, Table 3), separated from the base

Coating tests for gas-abrasive wear resistance were carried out at room temperature and heated to 550 °C. Data were obtained on samples made of steel S235 to compare the wear resistance of coatings. Abrasive wear resistance was investigated by rubbing against non-rigidly fixed abrasive particles. According to these data, the following graphical dependences were built; Fig. 6, 7.



Fig. 6. The intensity of gas-abrasive wear of coatings sprayed by plasma-arc where:  $1 - Fe_3AI$ coating (mode 5);  $2 - Fe_3AI$  coating (mode 7);  $3 - Fe_3AI$  coating (mode 8); 4 - steel S235



Fig. 7. The intensity of abrasive wear of coatings sprayed by plasma arc where:  $1 - Fe_3Al$  coating (mode 5);  $2 - Fe_3Al$  coating (mode 7);  $3 - Fe_3Al$  coating (mode 8); 4 - S235 steel

The results of studies into the electrochemical properties of plasma-arc coatings are given in Table 7. Based on these data (Table 7), the resistance and estimated service life of Fe-Al coatings were calculated in 3% NaCl solutions (Table 8), and in 0.5% H<sub>2</sub>SO<sub>4</sub> (Table 9).

Table 7

Results of electrochemical tests of FeAI coatings made from a fluxcored wire by PAS method

| Mode   | Coating            | Spraying mode |                               | 3 %           | NaCl      | $0.5 \text{ M H}_2\text{SO}_4$ |                     |                     |
|--------|--------------------|---------------|-------------------------------|---------------|-----------|--------------------------------|---------------------|---------------------|
| number | thickness, $\mu m$ | <i>I</i> , A  | $Q_{air}$ , m <sup>3</sup> /h | <i>h</i> , mm | $E_s$ , V | $I, A/cm^2$                    | $E_{\rm s}, { m V}$ | $I, A/cm^2$         |
| 5      | 600-700            | 230           | 40                            | 150           | -0.52     | $2.5 \cdot 10^{-6}$            | -0.34               | $2.7 \cdot 10^{-5}$ |
| 7      | 400                | 230           | 45                            | 220           | -0.54     | $3.12 \cdot 10^{-6}$           | -0.33               | $3.0 \cdot 10^{-5}$ |
| 8      | 400                | 230           | 45                            | 150           | -0.55     | $2.3 \cdot 10^{-6}$            | -0.32               | $2.0 \cdot 10^{-5}$ |

#### Table 8

Resistance and estimated service life of FeAI coatings in 3 % NaCI, made of flux-cored wire by PAS method

| Mode<br>number | <i>i</i> , A/cm <sup>2</sup> | W,<br>g/m²h | P,<br>mm/r | Corrosion resis-<br>tance point | Stability<br>group | Service<br>life, years |
|----------------|------------------------------|-------------|------------|---------------------------------|--------------------|------------------------|
| 5              | $2.5 \cdot 10^{-6}$          | 0.026       | 0.030      | 4                               | Stable             | 16.7                   |
| 7              | $3.12 \cdot 10^{-6}$         | 0.033       | 0.037      | 4                               | Stable             | 15.1                   |
| 8              | $2.3 \cdot 10^{-6}$          | 0.024       | 0.028      | 4                               | Stable             | 15.9                   |

#### Table 9

Resistance and estimated life of Fe-Al coatings made of flux-cored wire by PAS method in 0.5 % H<sub>2</sub>SO<sub>4</sub>

| Mode<br>number | i, A/cm <sup>2</sup> | W, g/<br>m²h | P,<br>mm/r | Corrosion resis-<br>tance point | Stability<br>group | Service<br>life, years |
|----------------|----------------------|--------------|------------|---------------------------------|--------------------|------------------------|
| 5              | $2.7 \cdot 10^{-5}$  | 0.28         | 0.31       | 6                               | Low stability      | 1.7                    |
| 7              | $3.0 \cdot 10^{-5}$  | 0.31         | 0.34       | 6                               | Low stability      | 1.6                    |
| 8              | $2.0 \cdot 10^{-5}$  | 0.20         | 0.23       | 5                               | Stable             | 2.2                    |

The studies have allowed us to establish the heat resistance, wear resistance (abrasive, gas-abrasive,) and corrosion resistance of intermetallide coatings formed in the process of plasma-arc spraying.

#### 6. Discussion of results of investigating the properties and structure of Fe-Al coatings

In contrast to [9] where in the process of arc spraving an intermetallide coating was formed after heat treatment, the PAS of flux-cored wire made it possible to obtain an intermetallide coating without performing this operation. This becomes possible due to the fact that when melting flux-cored wire, the steel shell and aluminum powder filler interact when heated with the exothermic effect of the synthesis of intermetallide Fe<sub>3</sub>Al. Also, the use of the PAS process for the formation of intermetallide layers can significantly reduce the amount of oxides and the amount of unreacted iron and aluminum in the coating [11]. This is achieved due to the fact that the melting and dispersion of the wire material is carried out in inert argon plasma, which leads to minimal oxidation of particles during the spraying process. Unlike [5-7] where the coating process is associated with significant costs for the purchase of materials, the PAS process is characterized by a lower cost of the process. It is shown that the use of more economical materials (wires instead of specialized powders) and heating sources (electric arc instead of gas mixtures) makes it possible to form high-quality intermetallide coatings. The mechanical, wear-resistant, and corrosion-resistant characteristics of such coatings correspond

to the characteristics of coatings applied by high velocity oxygen fuel methods and detonation spraying.

To establish the regularity of the influence of the technological parameters of the PAS process on the structure and properties of the resulting coatings, the method of mathematical planning of the experiment was used using a complete factor experiment  $2^3$ . Analysis of the regression equations (block 1, block 2) showed the difference in how the spray distance, which changes its sign, affects the current changes. This is due to the formation of a finer stream of sprayed particles, full interaction of flux-cored wire components during wire spraying with increasing arc current. Also, the increase in heat supply to the gas jet ( $I/Q_{air}$  increases from  $4.0 \div 4.5$  to  $5.11 \div 5.75$ ) leads to overheating of particles and the formation of oxide phases ( $Al_2O_3$ ,  $Fe_3O_4$ ,  $Fe_2O_3$ ).

The results of a comparative analysis of investigating the microstructure of coatings (Fig. 2) showed that under the conditions of block 180/4.8, coatings with high microheterogeneity and porosity were formed. The structure of coatings obtained under the conditions of the 230/6.0 block is dense, homogeneous, finely dispersed.

For further consideration, the coatings obtained in experiments 5, 7, and 8 were selected. When coatings were applied under these conditions, a constant characteristic of the process was the specific heat investment of the arc into a unit of sprayed material - 2.35 kWh/m, which characterizes the stage of melting of the wire in the arc zone. The second indicator, characterizing the heating of particles of sprayed material, is associated with the temperature of the gas jet. Its assessment can be carried out by calculating the heat investment per unit volume of gas  $I \cdot U/Q_{air}$ ; for experiment 5, this value is  $0.345 \text{ kWh/m}^3$ ; for experiments 7 and 8, it is 0.307 kWh/m<sup>3</sup>. As the characteristic of the spraying process, taking into account the duration of the particle stay in the volume of the jet, the ratio of the spraying distance and gas consumption is taken, which is interconnected by the following ratio  $-h/Q_{air}$ . The value of this characteristic is 3.75 (experiment 5); 3.3 (experiment 7); and 4.9 (experiment 8), respectively.

Thus, experiment 5 can be characterized by the conditions for increasing the thermal activity of a plasma jet at a short spray distance of 150 mm. Under the conditions of experiment 7, there is less heat intake in the jet in combination with a longer particle time in it due to an increase in the jet speed, which limits the degree of heating of the sprayed particles. During experiment 8, a longer time spent by particles in the jet, combined with a decrease in the thermal activity of the jet, creates conditions for the development of oxidation processes of the sprayed material.

According to the results of XSA, it was found that in all samples the main phase is an intermetallide of the Fe<sub>3</sub>Al type. These results confirm the data obtained by scanning electron microscopy in combination with XSMA (Fig. 4, Table 4). The aluminum content obtained in the samples by scanning five to seven sections of different sizes, averaged over the entire cross-sectional area of the coating, was within the homogeneity region of the intermetallide Fe<sub>3</sub>Al. Analysis of individual structural components of the coating, performed using an electronic probe measuring 1.5  $\mu$ m, revealed solid solutions of aluminum in iron (spectra 6, 8; Fig. 4, *a*). Aluminum oxide Al<sub>2</sub>O<sub>3</sub> (spectrum 4, Fig. 4, *d*) and iron oxide of FeO composition (spectrum 6, Fig. 4, *b*) were also found.

It is characteristic that the oxide phases are mainly located at the boundaries of the lamellas and between the coating and the base (spectra 1, Fig. 4, *c*, and Table 6, Fig. 4, *b*).

XSA (Table 5) showed the presence of aluminum oxide in all samples and a small content of iron oxide in the coatings, but there are differences in their qualitative composition. Thus, in samples No. 1 and No. 2, FeO iron oxide (wustite) was found, which is formed on the surface of iron at temperatures of 700 and 800 °C. In other cases, the main oxide phase was Fe<sub>3</sub>O<sub>4</sub> (magnetite) - the most stable iron-oxygen compound. Traces of Fe<sub>2</sub>O<sub>3</sub> (hematite) were found only in sample No. 8 (maximum values of all experimental parameters). Fe<sub>2</sub>O<sub>3</sub> is formed by the oxidation of iron at temperatures of 600-900 °C. Here, Fe<sub>3</sub>O<sub>4</sub> and FeO are present under the Fe<sub>2</sub>O<sub>3</sub> film. In the coating, along with Fe<sub>2</sub>O<sub>3</sub>, traces of Fe<sub>3</sub>O<sub>4</sub> were found. Iron is present in all samples (in fact, as XSMA showed, these are solid solutions of aluminum in iron) in the form of two modifications  $\alpha$  and  $\gamma$ . At the same time, the content of  $\alpha$ -Fe in all samples, except No. 6, is 1.5–5 times higher than in  $\gamma$ -Fe. Only in samples number 6, on the contrary, the content of  $\gamma$ -Fe is almost 7 times higher than the content of  $\alpha$ -Fe. At the same time, the amount of unreacted iron, similar to aluminum, is insignificant in all samples

You should also pay attention to the coloring of individual structural components of the coating, namely the brightest areas – you can identify them as solid solutions of aluminum in iron. At the same time, gray color is characteristic of intermetallide  $Fe_3Al$ , and dark, which has the greatest microhardness, is characteristic of oxide phases.

Thus, according to the results of our work, it was established that the following mode should be considered the most favorable for the formation of FeAl under PAS conditions when spraying Fe-Al flux-cored wire:

- current 230 A;
- air flow rate 40 m<sup>3</sup>/h;
- spraying distance 150 mm;
- wire feed speed 6 m/min.

Under these conditions, a coating was formed consisting of the main phase of Fe<sub>3</sub>Al and solid solutions of Al in Fe with traces of oxides with a porosity of 1.5 vol. % and a microhardness of  $3024\pm761$  MPa.

FeAl coatings obtained under modes 5, 7 and 8 were investigated in order to determine their heat resistance, wear resistance (abrasive, gas-abrasive), and corrosion resistance.

The heat resistance of coatings separated from the base was measured, where the main phase is the intermetallide Fe<sub>3</sub>Al. Our results indicate the influence of the ratio of intermetallide and oxide phases on the initial temperature and intensity of the coating oxidation process (Table 5, Fig. 3). Comparing the oxidation curves, it is possible to notice an increase in the temperature of the oxidation principle depending on the phase composition during the transition from the spraying mode of sample No. 8 to the spraying mode of sample No. 5. Oxidation intensity coatings also differ significantly depending on the modes and phase composition of the coatings. So, for sample number 8 with the maximum values of all experimental parameters, the oxidation intensity increases over the entire temperature range.

Tests for gas-abrasive wear resistance at room temperature showed that the wear resistance of Fe-Al flux-cored wire coatings obtained by the PAS method exceeds the stability of S235 steel by an average of 2 times. Resistance to abrasive wear of PAS coatings made of flux-cored wires based on Fe-Al is 1.8 times higher than that of S235 steel.

When investigating the electrochemical properties of plasma-arc coatings, the density of corrosion current (A/cm<sup>2</sup>) in 3 % NaCl decreased from  $3.12 \cdot 10^{-6}$  to  $2.3 \cdot 10^{-6}$ ; in 0.5 % solution H<sub>2</sub>SO<sub>4</sub> it decreased from  $3.0 \cdot 10^{-5}$  to  $2.0 \cdot 10^{-5}$  (Table 7). The obtained results of corrosion tests of intermetallide coatings (Tables 8, 9) indicate that all coatings studied in these environments belong to the group of "resistant". These coatings have corrosion resistance points of 4 and 5, with a deep corrosion index of 0.23-0.34 (mm/p). The estimated service life of these coatings is, respectively, 15.9-16.7 years in the medium of an aqueous 3-% solution of NaCl and 1.62.2 years in an aqueous medium of a 0.5-% solution of H<sub>2</sub>SO<sub>4</sub>.

#### 7. Conclusions

1. The structure and phase composition of intermetallide-type coatings obtained by plasma-arc spraying of conductive flux-cored wire consisting of a tubular steel shell and a filler made of aluminum powder have been investigated. It was found that the structure of the coatings is dense, homogeneous, and fine, the results of studies using XSA and XSMA showed that the main phase in the coatings is an intermetallide of the Fe<sub>3</sub>Al type.

2. The optimal modes of the PAS process have been established in which dense coatings are formed with a porosity of less than 1.5 vol. % and a microhardness of  $3024\pm761$  MPa.

3. It was found that the gas-abrasive wear resistance of coatings based on the intermetallide  $Fe_3Al$  at room temperature exceeds the resistance of steel S235 by an average of 2 times. Resistance to abrasive wear of PAS coatings made of flux-cored wires based on Fe-Al is 1.8 times higher than that of S235 steel. The results of corrosion tests showed that all

coatings investigated in the medium of a 3-% NaCl solution and an aqueous 0.5-% solution of  $H_2SO_4$  belong to the group of "resistant". They have corrosion resistance scores of 4 and 5, with a deep corrosion index of 0.23–0.34 (mm/p). The estimated service life of these coatings is, respectively, 15.9–16.7 years in an aqueous medium of a 3-% solution of NaCl and 1.6–2.2 years in an aqueous medium of an 0.5-% solution of  $H_2SO_4$ . This allows them to be recommended for practical use for protection against oxidation, corrosion, and gas-abrasive wear of components and assemblies in the heat power industry.

#### **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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