Study of high-temperature heating effect on transformation of structure and phase composition of coatings of Al-Fe system

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Abstract. It is shown that the phase transformations in the coating of the Fe-Al system produced by explosion welding of an aluminum AD1 with steel St3 and double occur in the following sequence: Fe$_2$Al$_5$ → FeAl$_2$ → FeAl → Fe$_3$Al → Fe (Al). They are accompanied by an increase in its thickness. The layered FeAl / Fe$_3$Al / Fe (Al) coating on the inter-phase FeAl / Fe$_3$Al boundary formed on a steel basis is porous, and inclusions of sulphides of manganese are present in alumina Fe$_3$Al. The layered FeAl / Fe$_3$Al / Fe (Al) coating on the steel base has a porous structure at the FeAl / Fe$_3$Al interface, and inclusions of manganese sulfides in the Fe$_3$Al aluminide region.

1. Introduction
The most universal coating for the protection against oxidation of steel products for various purposes at elevated temperatures in air and in some corrosive environments is aluminide [1-8]. However, the aluminide coatings obtained by conventional methods are mainly composed of very hard and brittle intermetallic compounds, such as Fe$_2$Al$_5$, FeAl$_3$ and FeAl$_2$, which cause their cracking and, correspondingly, deterioration of the protective properties [1, 9-12]. Aluminides FeAl and Fe$_3$Al possess excellent corrosion and oxidation resistance [1, 6-8]; therefore, at the stage of coating production, it is necessary to provide technological methods ensuring the presence of FeAl and Fe$_3$Al in their composition.

In this paper, the sequence and kinetics of phase and structural transformations in the coating of the Fe-Al system after high-temperature heating are investigated.

2. Materials and methods
The studied material was a coating of the steel surface, the obtainment of which included: explosion welding of an aluminum AD1 with steel St3 (10 + 10 mm); double heat treatment of the obtained composition at 660 °C, 3h for growing a diffusion zone of the required thickness (Figure 1, a) and at 640 °C, 3h to form a main crack at the interface Al+FeAl$_3$ / Fe$_2$Al$_5$ (Figure 1, b) and mechanical separation of the unreacted aluminum layer along the formed crack (Figure 1, c).
Figure 1. The microstructure of a diffusion zone after double heat treatment at 660 °C, 3h + 640 °C, 3h (a) and after mechanical separation of an unreacted aluminum layer (b)

To study the transformation of the structure, Subsequent Heat Treatment (SHT) of the samples with coatings was carried out in a LOIP LF-7/13-G1 furnace at a temperature of 1100 °C. Metallographic studies were performed on a modular metallographic microscope Olympus BX-61. The phase composition of the diffusion zone was evaluated by comparing the data obtained with the Bruker D8 ADVANCE ECO diffractometer and the Versa 3D raster dual-beam electron microscope. The microhardness was measured on a PMT-3M tester with a load of 100.

3. Results and discussion
After separation of the unreacted aluminum layer, a coating (Figure 1, b) with a thickness of 270 µm with microhardness ~ 12 GPa was obtained on the steel surface. The data comparison of of X-ray diffraction and energy-dispersion analysis made it possible to establish that the coating is a homogeneous intermetallide layer Fe$_2$Al$_5$ with an Al content of ~ 73 at. %.

The experimental data analysis of heat threated samples with coatings at 1100 °C (Figure 2) allows one to establish the following.
After holding for 10 minutes (Figure 2a), between the steel and the Fe$_2$Al$_5$ layer (which hardness decreased to 10 GPa), a transition zone consisting of two interlayers separated by a porous boundary is formed.

Increasing the holding time to 1 hour leads to a growth of the transition zone thickness (Figure 2, b). The energy dispersive analysis data (Figure 3) showed that the presence of aluminum in the surface layer decreased from 73 to 68% at., and X-ray diffraction data showed a change in its phase composition from Fe$_2$Al$_5$ to Fe$_2$Al$_5$ + FeAl. The first interlayer of the intermediate zone is FeAl aluminide with needle inclusions FeAl$_2$ (Figure 4, b), the number of which decreases as it moves away from its surface. The hardness of the interlayer varies from 6 to 4.5 GPa because of its variable chemical composition (from 55 to 35 at.% Al). Behind the porous boundary, there is an interlayer with a phase composition - Fe$_3$Al $\rightarrow$ Fe (Al), with no visible boundaries between phases. The microhardness of the aluminide Fe$_3$Al is 3.5 GPa, and the Fe (Al) solid solution is 2.5 GPa.

Figure 2. The microstructure of coating after heat treatment at 1100 °C for 10 min (a) and 1 h (b)

Figure 3. The distribution of chemical elements over the thickness of the coating after heat treatment at 1100 °C for 1 h (Figure 4)
Figure 4. SEM images of coating after heat treatment at 1100 °C for 1 h

An increase in the holding time to 1.5 hours (Figure 5) leads to a further transformation of the phase composition of the coating, in which the presence of brittle aluminides Fe$_2$Al$_5$ and FeAl$_2$ is not established. It consists of aluminides FeAl, Fe$_3$Al and a solid solution Fe (Al). The coating thickness is ~ 370 µm, which is almost 100 µm thicker than the original coating. After 2 hours of exposure, the coating thickness is 440 µm.

Figure 5. The microstructure of coating after heat treatment at 1100 °C for 1.5 h

The porous boundary dividing the diffusion zone in two layers is presumably the boundary of the FeAl and Fe$_3$Al phases, and, judging from the distance from the surface, it is located in the region where the Fe$_2$Al$_5$ and the steel boundary was located before the transformation of the coating. Below this boundary, a large number of inclusions are present in the structure. The data of the point energy-dispersive analysis (Figure 6, Table 1) showed that these are manganese sulfides, which are present in large quantities in St3, but no inclusions was found in porous boundary. The data of point energy-dispersive analysis (Figure 6, Table 1) allows to suggest that these are manganese sulfides, which are present in large quantities in St3, at the same time no of such inclusions is found in a porous boundary.
Figure 6. SEM images of pores and inclusions in the interlayer Fe$_3$Al

Table 1. Results of point energy-dispersive analysis (Figure 6)

| № | Element | Weight % | Atomic % | Error % |
|---|---------|----------|----------|---------|
| 1 | AlK     | 13.51    | 24.43    | 7.86    |
|   | MnK     | 1.37     | 1.21     | 8.75    |
|   | FeK     | 85.12    | 74.36    | 1.58    |
| 2 | AlK     | 13.82    | 24.53    | 7.68    |
|   | S K     | 2.39     | 3.57     | 5.6     |
|   | MnK     | 3.99     | 3.48     | 3.44    |
|   | FeK     | 79.79    | 68.42    | 1.6     |

4. Conclusion
The phase transformations of the coating in the Fe-Al system obtained by explosion welding of aluminium AD1 with steel St3 followed by heat treatment at 660 °C, 3 h + 640 °C, 3 h, at 1100 °C proceed in the following sequence: Fe$_2$Al$_5$ → FeAl$_2$ → FeAl → Fe$_3$Al → Fe (Al). And they are accompanied by an increase in its thickness from 270 to 440 µm during 2 hours of exposure.

It has been established that the layered FeAl / Fe$_3$Al / Fe (Al) coating with a hardness of ~ 6 GPa on the interphase FeAl / Fe$_3$Al boundary, formed on a steel base, has a porous structure, and the presence of inclusions of manganese sulfides in the aluminide Fe$_3$Al is found.

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