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Aluminizing of the EP648 alloy by hot-dipping

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Abstract. The structure and phase composition of coatings formed on the surface of the EP648 alloy were studied using the hot-dip aluminizing method with subsequent heat treatment. Aluminizing allows one to form a continuous aluminide coating with a clearly distinguishable layered structure, in which the upper layer is represented by (Al) with eutectic inclusions, the middle layer consists of (Al) and intermetallics mixture, and the thin boundary layer has a $\varphi + \kappa / \varphi / Ni_2Al_3(Cr)$ structure. Treatment of the aluminized EP648 alloy at 1100 °C allows one to get rid of the (Al) phase and form an intermetallic coating due to the diffusion redistribution of chemical elements.

1 Introduction

The EP648 alloy is used for the production of cold-rolled sheet metal; for tubular blanks and pipes by hot pressure treatment; for the manufacture of highly loaded parts, fittings, flanges, sheet metal parts for a limited service life at temperatures up to 950 °C; for the manufacture of aircraft parts (discs, gas turbine blades) [1].

The relatively low operating temperature of this alloy causes low efficiency of its oxidation. It is possible to ensure a longer service life and higher operating temperature of products made of EP648 alloy by applying an aluminide coating to its surface, due to which a stable Al2O3 oxide film is formed, which will be effectively resist high-temperature oxidation [2].

Currently, there are many different approaches to applying aluminide coatings, including pack cementation [3], cold spraying [4], hot-dip aluminizing [5], and many others [6-7]. The use of the hot-dip aluminizing method is one of the most common and easy to implement, guaranteeing the formation of a continuous aluminum coating over the entire surface of the product in a short period of time.

We have thoroughly studied the features of aluminizing various iron- and nickel-based alloys, ranging from technically pure alloys to alloys with a complex alloying system [2].

The purpose of this work was to explore, on the basis of accumulated experience, the formation features of aluminide coatings on the EP648 alloy surface, its structure and phase composition.

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2 Materials and methods

The studies were carried out on aluminized samples made of EP648 alloy (CrNi50WMoTiAlNb). The EP648 alloy chemical composition is shown in Table 1. To carry out aluminizing, samples measuring 10x20 mm were cut from the alloy sheet and a hole was drilled in them for hanging on a wire. The samples were pre-ground on 320-grit sandpaper and degreased. In graphite crucibles, the AD31 aluminum (Table 2) melt was heated in a SNOL 8.2/1100 furnace to a temperature of 700 - 760 °C, then the samples were dipped into it. The samples were kept in the melt for 1-10 min and then removed and cooled in air.

Table 1. Chemical composition of EP648 alloy (wt.%) TU 14-1-1072-74.

Ni	Cr	Fe	Mo	Ti	Nb	W	Ce	Si	Mn	C	S	P	Al	B
base	32 - 35	≤ 4	2.3 - 3.3	0.5 - 1.1	0.5 - 1.1	4.3 - 5.3	≤ 0.03	≤ 0.4	≤ 0.5	≤ 0.1	≤ 0.01	≤ 0.015	0.5 - 1.1	≤ 0.008

Table 2. Chemical composition of AD31 aluminum alloy (wt.%) GOST 4784–2019.

Al	Si	Mg	Fe	Mn	Zn	Cu	Ti	Cr
base	0.2-0.6	0.45-0.90	0.5	0.1	0.2	0.10	0.15	0.1

The aluminized samples were heat treated in an air atmosphere in a LOIP LF-7/13-G1 furnace at 1100 °C for up to 10 min. The structure and chemical composition of the coatings were studied using Versa 3D Dual Beam electron microscope with an attachment for energy-dispersive spectral analysis (EDS). The phase composition of the coatings was determined using X-ray phase analysis (XRD) on Bruker D8 diffractometer. The shooting was carried out with a collimated (diameter 1 mm) X-rays from a copper anode (λ=1.5418Å). A Goebel mirror was used to convert a divergent beam into a parallel beam of low divergency. Position-sensitive detector SSD160 was used for measuring and recording of diffraction patterns.

3 Results and discussion

The change in the main parameters (temperature and time) of the aluminizing process showed the following (Figure 1). The change in the process temperature is accompanied by a change in the thickness of the resulting coating, the volume fraction of intermetallic phases and the ratio of the thicknesses of the layers that make up the coating. The phase composition of the coating remains unchanged. An increase in temperature facilitated the process and guaranteed a more stable result when aluminizing a batch of samples.

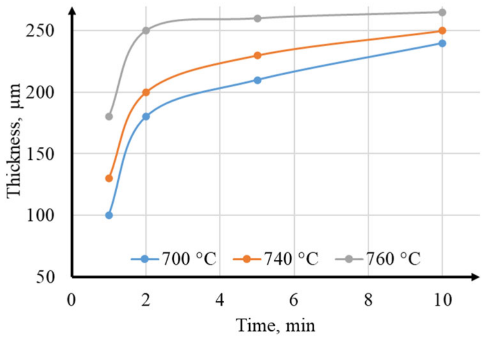
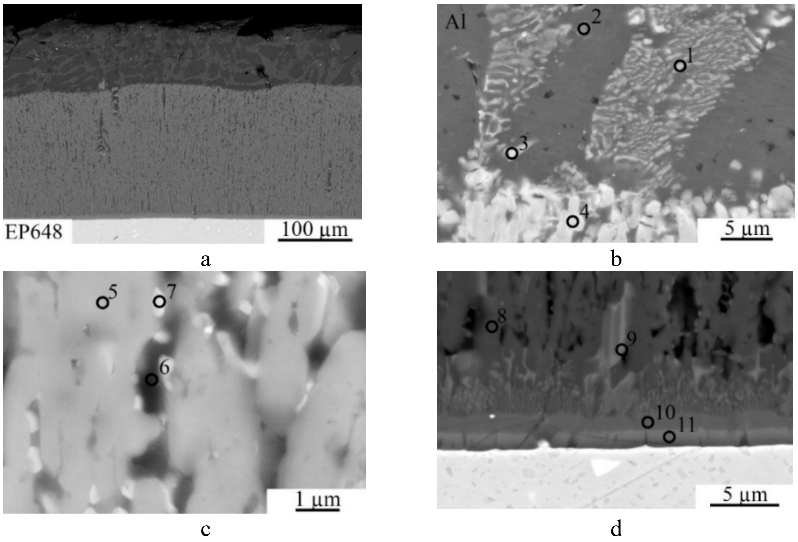


Fig. 1. The temperature-time dependence of coating thickness.

Changing the aluminizing time affects the continuity of the resulting coating and the dissolution degree of the substrate material in the melt. Thus, a short holding time (less than 1 min) may be insufficient for uniform wetting of the substrate surface with the melt and, as a consequence, a discontinuous coating (with exposed substrate) may form. A significant increase in the holding time in the melt is accompanied by active dissolution of the substrate material in the melt, while the thickness of the final coating remains virtually unchanged.

As a result of the conducted preparatory tests, it was established that it is optimal to carry out aluminizing at 740 °C with a holding time of 2 min.

Aluminizing of the EP648 alloy resulted in the formation of a thick coating (~200 µm) (Figure 2). The coating has a surface layer consisting of aluminum and eutectic structures and an extended zone consisting of finely dispersed intermetallic inclusions.



a b

c d

Fig. 2. The structure of the coating on the EP648 alloy after aluminizing (a) and EDS analysis points location (b, c, d).

The upper layer eutectic structure consists of a mixture of the (Al) phase and the metastable phase Ni_2Al_9 (Table 3, point 1, Figure 3) [8]. The middle layer, which occupies the main part of the coating, consists of (Al), $CrAl_7$ and the κ-phase (Table 3, points 4-8). In this case, we can definitely state the formation of $CrAl_7$, since according to EDS analysis, no other alloying elements dissolve in this phase.

Table 3. Point EDS analysis results

Point (Figure 2)	**Chemical element, at. %**							**Phase**
	Al	**Ni**	**Fe**	**Cr**	**Mo**	**Ti**	**W**	
1	93.53	6.47	-	-	-	-	-	(Al)+Ni_2Al_9
2	98.50	1.50	-	-	-	-	-	(Al)
3	93.95	6.05	-	-	-	-	-	(Al)+Ni_2Al_9
4	85.10	1.65	0.46	11.10	0.43	0.47	0.77	κ
5	84.06	1.48	0.44	13.32	0.32	0.38	-	κ
6	92.45	-	-	7.55	-	-	-	$CrAl_7$
7	83.13	4.43	0.44	10.77	0.29	0.37	0.56	κ
8	84.38	3.17	0.41	11.72	-	0.32	-	κ
9	76.42	19.70	-	3.88	-	-	-	$NiAl_3$
10	72.15	13.52	0.28	12.63	0.26	0.46	0.71	φ
11	61.60	22.09	-	15.40	0.35	0.56	-	Ni_2Al_3(Cr)

According to the literature data, the mentioned phases have the following composition: κ-Al76Cr18Ni6 and φ-Al77.5Cr12.5Ni10 [9]. According to EDS analysis, a total of up to 2 at.% Fe, Mo, Ti and W are dissolved in these phases.

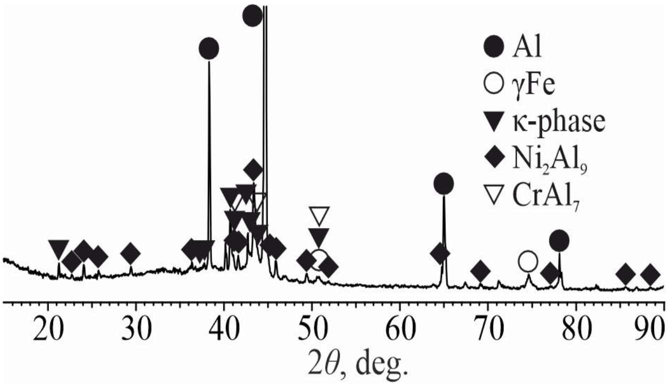


Fig. 3. XRD pattern from the aluminized coating on the surface of the EP648.

The lower thin layer, bordering the substrate, does not contain (Al) in its pure form and has a layered structure. The layer on the coating side is a two-phase φ + κ, the middle layer is represented by the φ-phase, and the layer on the substrate side is solid solution of Cr in Ni_2Al_3 intermetallic (Table 3, points 10-11).

Heating of the aluminized EP648 alloy for 10 min at 1100°C, aimed at transforming the coating structure, led to a change in structure to a multiphase one with globular intermetallic inclusions (Figure 4).

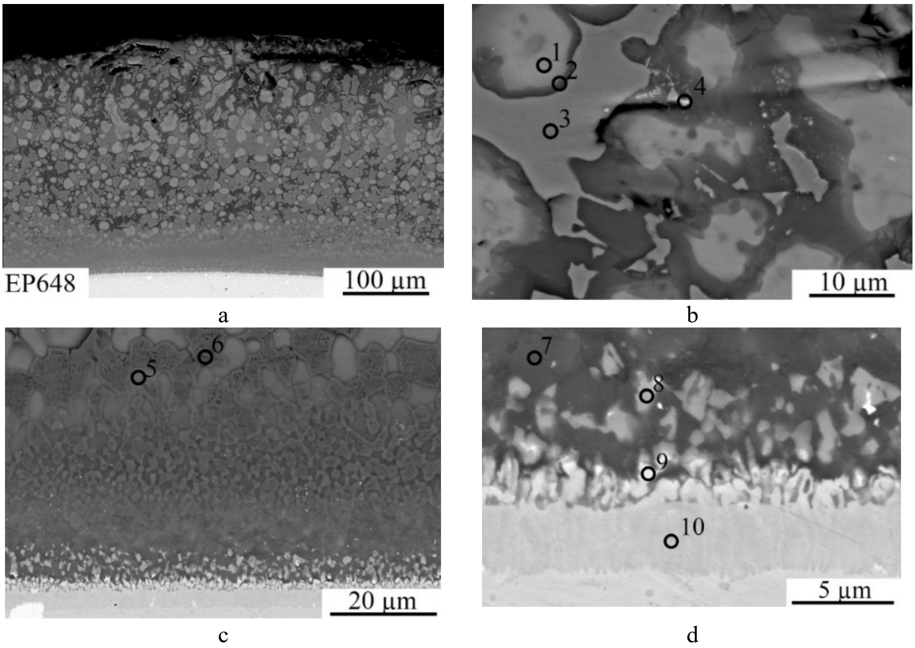


Fig. 4. The structure of the coating on the EP648 alloy after heat treatment at 1100 °C for 10 min (a) and EDS analysis points location (b, c, d).

The upper part of the coating contains Cr_5Al_8 inclusions surrounded by a dark shell of the φ-phase and areas corresponding to the Ni_2Al_3 intermetallic compound (Table 4, points 1-3, Figure 5). The presence of small inclusions rich in Ti and W is also noted (Table 4, point 4).

The central part of the coating consists of Cr_5Al_8 and Ni_2Al_3 aluminides. Most of the alloying elements dissolve in Cr_5Al_8, while in Ni_2Al_3 nothing dissolves except 2-5 at.% Cr (Table 4, points 5-6).

Table 4. Point EDS analysis results

Point (Figure 4)	Chemical element, at. %								Phase
	Al	Ni	Fe	Cr	Mo	Nb	Ti	W	
1	67.48	2.13	0.34	26.49	1.16	0.20	0.24	-	Cr_5Al_8
2	70.42	8.11	-	19.27	0.72	0.17	0.30	1.01	φ
3	60.74	37.20	-	2.07	-	-	-	-	Ni_2Al_3
4	63.17	13.49	0.41	11.71	0.38	0.99	8.26	1.60	Ti-rich phase
5	63.29	4.46	-	29.59	0.96	0.11	0.23	1.36	Cr_5Al_8
6	58.08	37.05	-	4.88	-	-	-	-	Ni_2Al_3
7	54.55	34.67	-	10.78	-	-	-	-	(Ni,Cr)Al
8	47.56	23.67	0.46	25.43	0.79	0.07	0.46	1.55	(Ni,Cr)Al
9	11.60	14.29	0.94	67.42	2.08	0.18	0.81	2.69	(Cr)
10	5.59	37.34	0.67	49.37	1.39	1.13	2.87	1.62	γNi+(Cr)

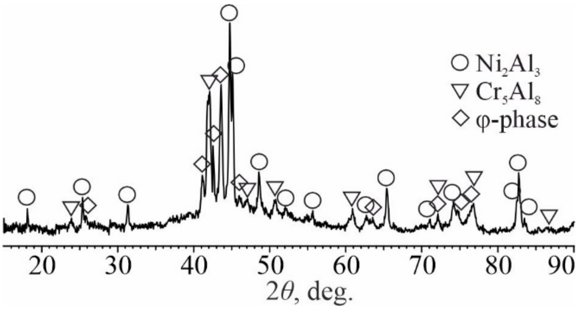


Fig. 5. XRD pattern from the aluminide coating surface after heat treatment at 1100 °C for 10 min.

Layer on the border is represented as a mixture of Cr and γ phase (Table 4, point 10).

An increase in the heat treatment time for this coating is accompanied by a redistribution of chemical elements in the volume; however, the coating retains many phases with a complex chemical composition.

4 Conclusion

Hot-dip aluminizing of EP648 alloy allows to form a continuous aluminide coating on the alloy surface. The resulting coating has a clearly distinguishable laminated structure, in which the upper layer is represented by (Al) with eutectic inclusions, the middle layer consists of the (Al) phase and intermetallics ($CrAl_7$ and the κ-phase) mixture, and the thin boundary layer has a φ + κ / φ / Ni_2Al_3(Cr) structure.

High-temperature treatment of the aluminized EP648 alloy allows to get rid of the (Al) phase and form an intermetallic multiphase coating due to the diffusion redistribution of chemical elements.

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