AIP Conference Proceedings

RESEARCH ARTICLE | NOVEMBER 19 2021

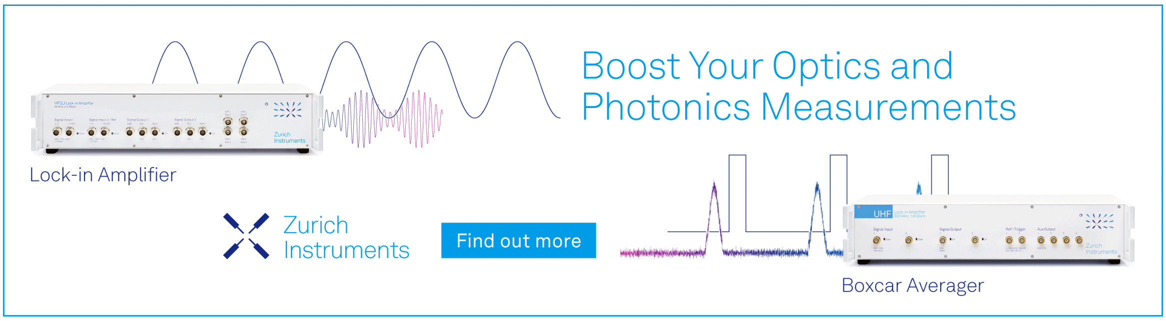
Study of stability of aluminide coating on hastelloy G35 in potassium chloroaluminate melt

K. R. Karimov; J. F. Gordeeva; E. S. Filatov

Check for updates

AIP Conf. Proc. 2388, 040012 (2021)
https://doi.org/10.1063/5.0070028

View Online
Export Citation



AIP Publishing

22 April 2024 07:37:17

Study of Stability of Aluminide Coating on Hastelloy G35 in Potassium Chloroaluminate Melt

K. R. Karimov[1], J. F. Gordeeva[2, a)] and E. S. Filatov[1,2]

[1] *The Institute of High Temperature Electrochemistry of the Ural Branch of Russian Academy of Sciences, 20, Akademicheskaya St., 620990 Yekaterinburg, Russia.*
[2] *Ural Federal University, 19, Mira St., 620002, Yekaterinburg, Russia.*

[a)] Corresponding author: julia100990@inbox.ru

Abstract. The process of aluminizing was studied by the method of thermodiffusion saturation with mechanical activation of the surface. Corrosion tests of structural elements of the equipment were carried out.

22 April 2024 07:37:17

INTRODUCTUION

The alloy Hastelloy G35 is used for manufacturing of equipment used in technology of pure zirconium production by purification from hafnium in chloroaluminate melt [1, 2]. During operation of the equipment, the surface of the devices and pipelines were not resistant to corrosion. Preliminary tests of the alloy using the method suggested in previous [3] showed positive results on corrosion resistance. The problem is that these studies were carried out in salt solutions at low temperature (up to 100 °C) and did not give a complete data on corrosion when using alloys in molten salt [4]. Therefore, we carried out tests at real temperature ranges and with real melt. Previously corrosion reduction was also made by the purification of melt using an aluminum protector [4].

METHODS AND INVESTIGATION STRATEGY

The study of aluminizing process of structural elements of the equipment was carried out. Aluminum coating was made by thermodiffusion saturation method with mechanochemical activation of the surface [5]. This method is a low-waste method of applying continuous diffusion coatings on steels using a batch plant. This method reduces both labor efforts and power consumption along with a simultaneous increase of saturation capacity of mixture without its caking. The saturating mixture consists of a saturated metal powder - 0.100-0.200 kg/m^2 and an electrocorundum powder - 50-70% of the weight of the elements. It constantly exists in a closed technological process and is regenerated by adding fresh portions of saturated metal powders to the mixture necessary to create a diffusion layer of a certain thickness. Saturation is carried out in a rotating reactor with a sealed compacting.

The installation diagram is shown in Figure 1. The procedure involves the purification of the elements surface from oxides and scale, degreasing, elements loading with a saturating mixture into a reactor; creation of vacuum and filling with inert gas. The process of thermodiffusion saturation was carried out in the temperature range 600 – 800 ºC during 5 hours without taking into account the time for heating the elements. The saturating mixture consists of corundum (fraction 160 μm), aluminum powder (5 wt% to corundum) and technical ammonium chloride (up to 1 wt% to corundum). The process is carried out in an argon atmosphere.

After thermodiffusion saturation, high temperature annealing was performed to relieve stresses in the coating. Each sample was placed in a separate alundum crucible and then placed in a muffle furnace heated to 950 ° C. The samples were kept for 1.5 - 2 hours at this temperature, and then the furnace was turned off and cooled to 800 ° C. Further cooling was carried out with an opened door in the furnace.

The obtained sample was examined by gravimetric and metallographic methods. Weighing was carried out using the Sartorius Research analytic balance, and the preparation of metallographic specimens was carried out using equipment and technology of Struers company. Etching of the specimens was made with a solution of the following composition: 10 mL HNO_3 + 1 mL HF + C_2H_5OH to 100 mL. Metallographic measurements were carried out using a microhardness tester PMT-3. Photographs were obtained using a Reichert microscope with a CanonEOS350D camera.

Modern Synthetic Methodologies for Creating Drugs and Functional Materials (MOSM2020)
AIP Conf. Proc. 2388, 040012-1–040012-5; https://doi.org/10.1063/5.0070028
Published by AIP Publishing. 978-0-7354-4142-2/$30.00

Also corrosion tests of the aluminized sample were carried out. The sample was placed into a molten salt and kept during 100 hours at 450 °C.

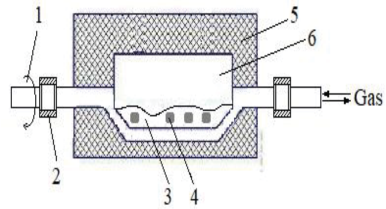


FIGURE 1. Installation diagram for thermodiffusion coating applying:
1 – rotator; 2 – joint bearing; 3 – saturated mixture; 4 – elements; 5 –furnace;
6 – barrel-type reactor

Preparation of the Equipment

The coating applying was performed in a sealed reactor filled with inert gas (pure argon). The construction should not prevent the free moving of treated elements and saturated mixture in the reactor. At the same time a good mixing is ensured, there should be no stagnant zones.

The material of the reactor must be heat resisting and heat proof. The process is carried out at 800 °C during 5 hours without taking into account the heating and cooling time.

Before starting the procedure, the inner surface of the reactor is purified from contaminants, washed and dried.

The process must be carried out in a furnace providing long-term operation at temperature of 800 °C. There should be no temperature gradients in various zones of the reactor.

Preparation of Saturating Mixture

The saturating mixture consists of three components: electrocorundum (brown aluminum oxide), aluminum powder, ammonium chloride.

Electrocorundum 14A GOST 28818-90. Fraction of 160 μm was used. Additionally the fraction of 63 μm was refined. The powder was washed and calcined at 400 ° C.

Aluminum powder PAP 1 GOST 5494-95.

Comercially pure ammonium chloride GOST 2210-73.

The main component of the mixture was electrocorundum. Aluminum was taken in amount of 5 wt% to corundum, ammonium chloride - 0.5-1 wt%. The amount of electrocorundum was chosen to fill the reactor a little less than half. The obtained mixture was suitable for multiple uses with the addition of fresh portions of aluminum and ammonium before each loading.

The mixture is thoroughly mixed. It was placed in a cold reactor. The elements were placed in the reactor with a loaded mixture.

Coating Applying

The reactor with loaded mixture and elements was sealed and placed in the furnace, where it was heated up to 200 °C. At the same time, the atmosphere was pumped out of the reactor by a vacuum pump. There should be no leakage into the reactor. The reactor was kept during half an hour. Then the pumping was stopped, and the reactor was filled with argon. Next the pumping continues up to the forvacuum. The procedure was repeated, and just the third filling with argon was considered to be operational.

The reactor was then disconnected from the forvacuum pump and rotated at 5 rpm. The temperature was raised up to 500 °C in the furnace and kept during half an hour. Then the temperature was raised up to 800 °C and kept during 5 hours.

In 5 hours the reactor was air cooled. Cooling was performed to 500 °C the rotation turned on.

Completely cooled reactor was dismantled; the elements were purified from mixture residues and subjected to high temperature annealing. The mixture can be used for the treatment of next set of elements.

22 April 2024 07:37:17

High Temperature Annealing

Annealing was carried out in a muffle furnace heated to 950 °C. The samples were kept at this temperature for 1.5 – 2 hours, and then the furnace was turned off and cooled to 800 °C. Further cooling was carried out with an opened door in the furnace.

Results of the Study

Study of aluminide coating formation was carried out at 600, 700 and 800 °C.

At temperature of 600 °C homogeneous continuous coatings without pores were obtained on the alloy Hastelloy G35 (Figure 2). Coating thickness reaches 30 μm. Several phases were observed in the coating. X-ray phase analysis determined that the main coating phase is Al_3Ni_2. A small amount of chromium and iron aluminides were present as well as complex mixed molybdenum and nickel aluminides.

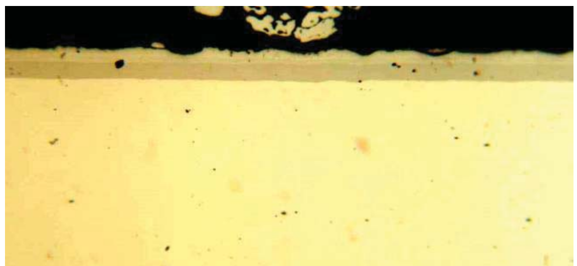


FIGURE 2. Aluminized alloy Hastelloy G35 at 600 °C. Coating thickness is 30 μm x 250.

22 April 2024 07:37:17

The coatings obtained on the alloy Hastelloy G35 at 700 °C are homogeneous, continuous, without pores (Fig. 3). The coating thickness is 45 – 50 μm. The coating is multiphasic and longitudinal cracks are formed at the obtaining of a specimen. X-ray phase analysis determines the presence of Al_3Ni_2, AlNi, Ni_5Al_3 phases and complex mixed nickel and molybdenum aluminides. Microhardness Hv_{50} of the base and the coating is 2920 MPa and 10235 MPa, respectively. It is not possible to measure the surface microhardness at weighting of 100 g as cracks are formed.

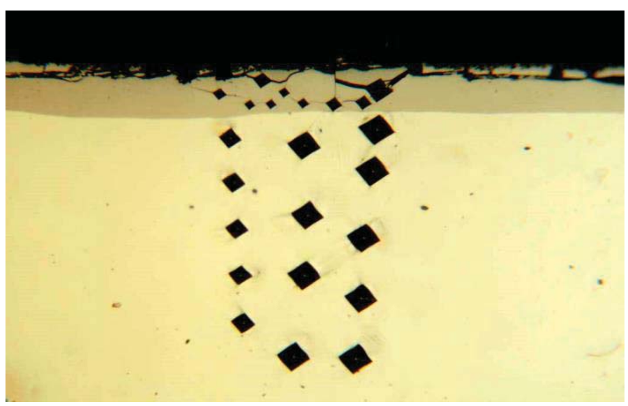


FIGURE 3. Aluminized alloy Hastelloy G35 at 700 °C. Coating thickness is 50 μm.x 250.

To obtain maximum thickness of protective coatings aluminizing was carried out at 800 °C.

A number of inner holes were considerably less in the alloy G35 (Fig. 4, 5), there were no pores on the surface, microhardness was homogeneous.

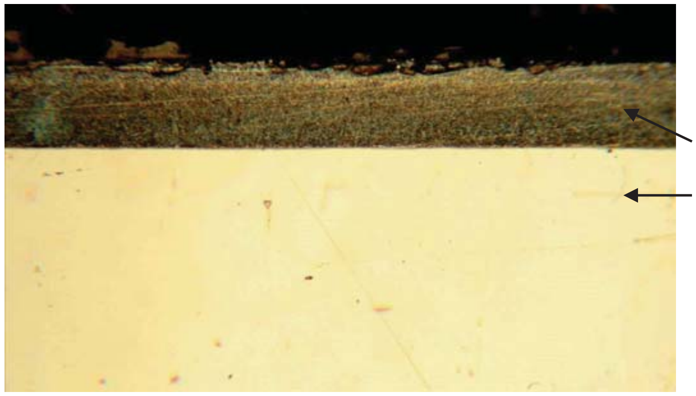


FIGURE 4. Aluminized alloy Hastelloy G35 at 800 °C. Layer thickness is 70 µm.

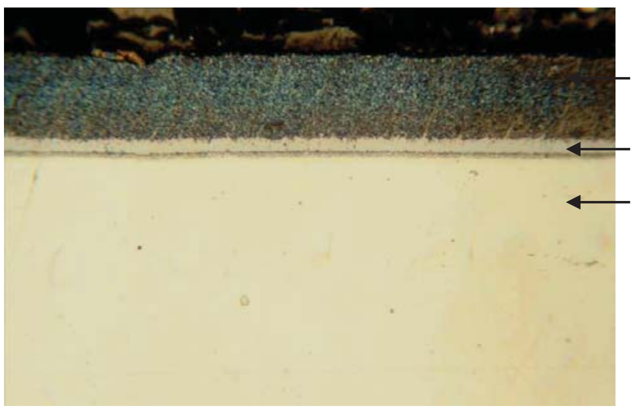


FIGURE 5. Aluminized alloy Hastelloy G35at 800 °C after annealing at temperature 950 °C during 1.5 hours. Layer thickness is 70 µm x 250

Corrosion Tests

Corrosion tests were carried out at temperature of 450 °C during 100 hours in molten salt.

Table 1 shows the results of the corrosion test for aluminized sample of Hastelloy G35 in the melt of potassium tetrachloroaluminate. Test duration was 48 hours at temperature of 450 ºC [3].

TABLE 1. Results of the Corrosion test.

Material	**Coating thickness (δ), мкм**	**S, cm^2**	**M_0, g**	**M_1, g**	**M_0- M_1, g**	**Km, g/m2·h**	**П, mm per year**	**Coating mass before test, g**
Hastelloy G35+Al	30	2,38	1,5954	1,5785	0,0169	1,479	0,002	~ 0,0193
Hastelloy G35	–	6,72	12,5054	12,5011	0,0043	0,130	0,128	–

As it is seen from the Table 1 aluminized Hastelloy 35 has a weight corrosion index (Km) by an order less than nonaluminized alloy. It means that corrosion speed for aluminized sample of alloy 35G indicates increased stability of the sample with the coating in molten salt.

22 April 2024 07:37:17

Final Key Points to Consider

The obtained results of the research lead to the following conclusions:

- Hastelloy G35 is aluminized for the thickness of 70 µm at 800 °C during 5 hours. The coating has a complex phase composition (main phases are AlNi and Al_3Ni_2). Longitudinal cracks are formed in many positions at the obtaining of a specimen;
- Annealing of aluminized samples during 2 hours at 950 °C leads to coating recovery, cracks do not form;
- Obtained coating is tightly connected with the sample and has an increased microhardness compared to metal base;
- Corrosion tests indicate a high chemical stability of the alloy with coating in the operation conditions. Corrosion speed of aluminized samples of Hastelloy G35 is 0,002 – 0,011 mm per year;

REFERENCES

1. M. G. Shtutsa and E. S. Filatov, "Zirconium XXI century", Materials of the scientific and technical conference, (JSC «ChMZ», Glazov, 2014), pp. 5-10 (in Russian).
2. V. V. Karpov and A. V. Bazhenov, Chimica Techno Acta **2**, 131–138 (2015).
3. Nickel Base Alloys, Methods for determining the resistance to intergranular corrosion, Standards Publisher, WD24.200.15-90, Moscow (01 april 1991) (in Russian).
4. E. S. Filatov and J. F. Gordeeva, "Investigation of the Corrosion Mechanism of Components of Structural Materials by Differential Scanning Mass Spectroscopy and Thermodynamic Modeling", Physics. Technology. Innovation. FTI-2020. Abstracts of the VII International Youth Scientific Conference dedicated to the 100th anniversary of the Ural Federal University, (Ural Federal University named after the First President of Russia B. N. Yeltsin, Ekaterinburg, 2020), pp. 516-517 (in Russian).
5. V.V. Chebykin and Ya. B. Chernov, RU Patent No. 2,221,898 (29 January 2004).

22 April 2024 07:37:17