ISSN 1027-5495. Functional Materials, 33, No.1 (2026), p. 115-120
doi:http://dx.doi.org/10.15407/fm33.01.115

Optimization of thermal diffusion technology of Ni–Al–Si–Cr–La coatings for the protection of heat-resistant nickel alloys

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Received October 18 2025, approved January 20, 2026

An industrial technology for thermal diffusion deposition of complex-alloyed protective coatings of the Ni–Al–Si–Cr–La system on heat-resistant nickel alloys ChS-70VM and EP-539LM has been developed and optimized. Saturation was carried out in powder mixtures based on ferroaluminum, ferrosilicon, and specially smelted Fe–Al–Si master alloys (20–30% Al, 15–25% Si) with the activator Na_2SiF_6 at 900–950 °C. The influence of temperature and time conditions of saturation and subsequent annealing on the coating growth kinetics, phase composition, and microstructure of the base was determined. It was shown that the optimal conditions (quenching at 1160°C + aging at 1050°C + saturation at 950°C, 6–8 hours) provide a coating thickness of 40–60 μm, a microhardness of 7500–9000 MPa, and the preservation of the structural stability of the γ′-phase of the alloy. The coatings demonstrate twice the heat resistance and corrosion resistance in diesel fuel combustion products with the addition of sea salt compared to traditional aluminide coatings. Bench and engine tests (600 hours) confirmed the resource efficiency of the developed technology.

Keywords: protective coatings, thermal diffusion saturation, Ni–Al–Si–Cr–La, heat-resistant nickel alloys, gas turbine engines, saturation kinetics, corrosion resistance.

Оптимізація термодифузійної технології покриттів Ni–Al–Si–Cr–La для захисту жаростійких нікелевих сплавів. *Д.Б. Глушкова, В.М. Волчук*

Розроблено та оптимізовано промислову технологію термодифузійного нанесення комплекснолегованих захисних покриттів системи Ni–Al–Si–Cr–La на жаростійкі нікелеві сплави ЧС-70ВМ та ЕП-539ЛМ. Насичення проводили в порошкових сумішах на основі фероалюмінію, феросиліцію та спеціально виплавлених лігатур Fe–Al–Si (20–30 % Al, 15–25 % Si) з активатором Na_2SiF_6 при температурах 900–950 °C. Встановлено вплив температурно-часових режимів насичення та наступного відпалу на кінетику росту покриття, фазовий склад і мікроструктуру основи. Показано, що оптимальні умови (закалка при 1160 °C + старіння при 1050 °C + насичення при 950 °C протягом 6–8 годин) забезпечують товщину покриття 40–60 мкм, мікротвердість 7500–9000 МПа та збереження структурної стабільності γ′-фази сплаву. Покриття демонструють удвічі вищу жаростійкість і корозійну стійкість у продуктах згоряння дизельного пального з добавкою морської солі порівняно з традиційними алюмінієвими покриттями. Стендові та двигунні випробування (600 годин) підтвердили ресурсну ефективність розробленої технології.

1. Introduction

The application of heat-resistant protective coatings is the primary means of combating corrosion and erosion damage to metals [1-4]. High operating temperatures (up to 950–1000°C) and aggressive gas environments containing combustion products and salts cause intense oxidation, sulfidation, and erosive wear, thereby reducing the reliability of new-generation functional materials [5-9].

Traditional aluminosilicide coatings formed by thermal diffusion saturation in powder mixtures provide high protective capacity due to the formation of dense oxide films of Al_2O_3 and SiO_2 [10-12]. However, the effectiveness of such coatings is largely determined by the saturation process parameters: temperature, holding time, diffusant composition, and subsequent diffusion annealing modes [13], [14]. These parameters directly affect the layer growth kinetics, phase composition, microstructure, and, consequently, the performance properties—microhardness, heat resistance, and durability [15-18].

An industrial technology for applying complex Ni–Al–Si–Cr–La alloyed coatings has been developed. By regulating the content of the main components (Al, Si) and complex alloying with lanthanum, it has been possible to significantly reduce the content of harmful phases based on refractory base elements (W, Mo), increase corrosion resistance severalfold, and lower the brittle-ductile transition temperature by 20°C compared to traditional aluminizing.

This paper presents the results of further optimization of the technology, taking into account the influence of thermal coating application conditions on the structure and properties of the ChS-70VM heat-resistant nickel alloy. The goal of the study is to ensure compatibility between thermal diffusion saturation processes and strengthening heat treatment of the alloy, improve the processability and performance characteristics of the coatings, and confirm their effectiveness under real-world conditions in bench and engine tests.

2. Materials and methods

The chemical composition of the studied alloys is given in Table 1.

Before applying the coatings, the alloys were subjected to heat treatment according to the modes indicated in Table 2 (mode 3 is recommended).

Saturation was carried out in stainless steel containers (volume 200 g and 3 kg) in powder mixtures of the following compositions (mass %):

- Composition 1 (basic): ferroaluminum (50% Al) + ferrosilicon (75% Si) + Na_2SiF_6 (2%);
- Composition 2: alloy Fe–30Al–15Si + Na_2SiF_6 (2%);
- Composition 3: alloy Fe–20Al–25Si + Na_2SiF_6 (2%).

Ferroalloy ingots were crushed to a fraction of <2.0 mm. The components were calcined: activator −at 150 °C for 1.5–2 h; powder mixture −at 900–920 °C for 3–4 h. Container tightness was ensured by a silicate fusible seal.

Diffusion annealing was carried out in air at 950 °C for 2–3 hours (coating quality control).

Heat resistance was assessed for the weight of 20×10×2 mm samples at 950°C for up to 500 h. Corrosion resistance was assessed −in molten salts (25% NaCl + 75% Na_2SO_4, 900°C, 25 h)

Table 1. Chemical composition of alloys, mass %

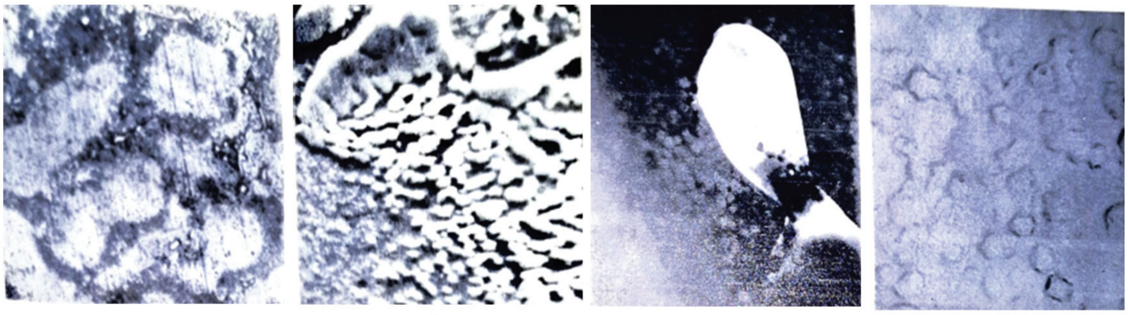
Alloy	Ni	Cr	Al	Ti	Co	Mo	W	Nb	C	B	Zr	Y
ChS-70VM	warp	16.69	3.08	5.00	11.06	2.07	5.00	0.23	0.25	0.04	0.02	0.015
EP-539LM	warp	19:00	3.22	3.08	6.93	4.23	3.25	0.27	0.14	0.08	-	-

Table 2. Heat treatment modes for alloys

Alloy	Heat treatment mode
ChS-70VM	Quenching 1160±10 °C, 4 h, air + aging 1050±10 °C, 5 h, air + aging 850±10 °C, 16 h, air
EP-539LM	Quenching 1180±10 °C, 3 h, air + aging 950±10 °C, 3 h, air

Table 3. Results of quantitative analysis of γ′-phase particles after different heat treatment modes

Compound	Alloy ChS-70VM (h, μm / H_μ, MPa)	Alloy EP-539LM (h, μm / H_μ, MPa)
1	45 / 7650	61 / 8500
2	50/8000	45 / 8500
3	50 / 7650	45 / 7650



a *b* *c* *d*

Fig. 1. Microstructure of the ChS-70VM alloy in the quenched state (a − dendritic structure, ×400; b − secondary electron image, ×2200; c − enhanced image in the superstructure reflection, ×30000; d − morphology of γ′-particles, ×19000)

and synthetic ash (900°C, 200 h). Thermal cycling strength was assessed −on wedge-shaped samples (T_{max} = 1020 and 980°C, up to 2500 cycles). Mechanical properties −were measured on an Instron 1251 universal testing machine (900°C). The microstructure was studied using optical and electron microscopy, and the phase composition was analyzed using X-ray diffraction analysis.

3. Results and their discussion

The ChS-70VM alloy exhibits pronounced −dendritic segregation. In the as-cast state, three types of γ′-phase are observed: eutectic, dispersed in the interdendritic spaces, and dispersed in the dendrite axes.

The optimal regime (quenching 1160°C + aging 1050°C + saturation 950°C, 6–8 h) ensures a bimodal distribution of the γ′-phase while maintaining a high volume fraction (42–45%) and minimal coagulation.

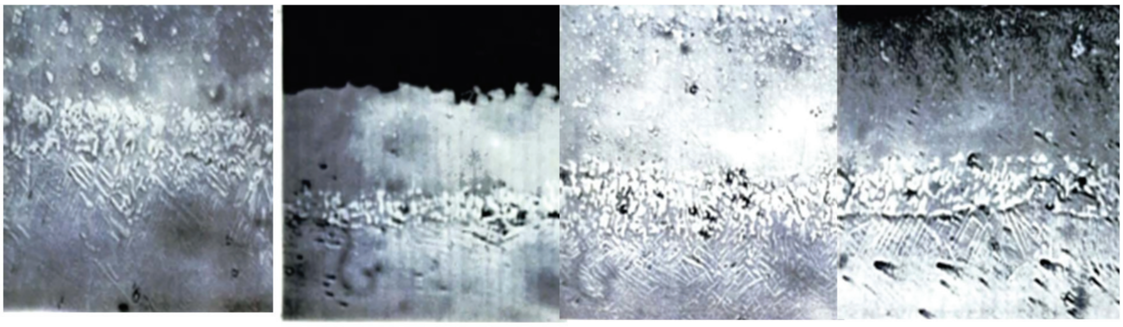
The saturation kinetics in multicomponent ligatures (compositions 2 and 3) allows obtaining coatings with a thickness of 40–60 µm in 6–8 hours at 950 °C.

The kinetics of thermal diffusion saturation in multicomponent master alloys (compositions 2 and 3) allows for the formation of uniform coatings 40–60 µm thick in 6–8 h at a temperature of 950°C. The experimental data obtained for the thickness (h) and average microhardness (Hµ) of the coating surface on ChS-70VM and EP-539LM alloys, depending on the temperature and saturation time, are presented in Table 4.

An analysis of the table shows that with an increase in the saturation temperature from 900 to 950 °C and an increase in the holding time from 4 to 12 hours, the coating thickness increases by 1.5–2.0 times for all compositions. The most intense growth is observed with base composition 1 (ferroaluminum + ferrosilicon). Switching to Fe–Al–Si master alloys (compositions 2 and 3) allows for a slight decrease in the coating growth rate, which has a positive effect on its ductility and adhesion to the

Table 4. Thickness (h) and average microhardness (Hµ) of coatings on alloys ЧС-70ВМ and ЭП-539ЛМ

Alloy	Temp., °C	Time, h	Composition 1		Composition 2		Composition 3	
			h, µm	Hµ, MPa	h, µm	Hµ, MPa	h, µm	Hµ, MPa
EP-539LM	900	4	44	7650	27	7350	29	7000
EP-539LM	900	6	61	8500	35	7650	33	7350
EP-539LM	900	12	81	9050	65	8500	37	8500
EP-539LM	950	4	47	8500	40	8000	44	7350
EP-539LM	950	6	77	9500	45	8500	45	7650
EP-539LM	950	12	88	–	76	8750	77	8000
ChS-70VM	900	4	34	7350	24	7000	28	7000
ChS-70VM	900	6	45	7650	33	7000	30	7350
ChS-70VM	900	12	63	8750	36	8500	34	7650
ChS-70VM	950	4	50	8000	45	7650	42	7350
ChS-70VM	950	6	66	9050	50	8000	50	7650
ChS-70VM	950	12	85	–	76	8500	71	8000



a *b* *c* *d*

Fig. 2. Microstructure of the surface layer of EP-539LM and ChS-70VM alloys after 500-hour heat resistance tests at 950 °C (a, b – composition 1; c – composition 3; d – composition 2

substrate. Surface microhardness consistently ranges from 7000 to 9500 MPa and correlates with the layer thickness. Maximum values (9050–9500 MPa) are achieved on the EP-539LM alloy at 950 °C and a holding time of 6–12 hours, which is associated with a higher chromium content in this alloy and, accordingly, the formation of a greater number of strengthening silicide phases. The obtained results confirm that the optimal conditions for obtaining coatings of a given thickness (40–60 μm) and microhardness (7500–9000 MPa) are temperatures of 950 °C and holding times of 6–8 hours.

The microhardness of the coatings is 7500–9000 MPa. The concentration of Al in the coating is 26–28%, Si 4–6%, La up to 1%.

Ni–Al–Si–Cr–La coatings are 2 times more heat-resistant than traditional ones at 950 °C (weight gain after 500 h < 14 g/m²).

The high protective properties of the developed coatings have been confirmed by heat and corrosion resistance tests. Table 5 shows the coating thickness and microhardness values in the initial state and after testing at 950°C for 130 hours.

As can be seen from the table, after long-term high-temperature testing, the coating thickness increases by 30–80% due to the formation of an additional oxide layer, while microhardness either remains unchanged (compositions 1 and 2) or decreases slightly (composition 4). Coatings obtained using Fe–Al–Si master alloys (compositions 2 and 3) demonstrate the most stable results: the change in microhardness does not exceed 10–12%, indicating high thermal stability of the phase composition. The weight gain of samples with Ni–Al–Si–Cr–La coatings after 500 hours at 950°C does not exceed 14 g/m², which is half that of traditional aluminide coatings.

On a gas-dynamic test rig (Tmax = 1,020 °C, 2,500 cycles), the coating effectively suppressed the development of thermal fatigue cracks. During 600-hour engine tests, nickel alloy with the applied coating showed no signs of corrosion damage (Figure 2).

Analysis of the structure of the samples in Figures 1 and 2 confirmed the fractal nature of their structure, due to the complexity of the geometric configuration of the surface layer of the EP-539LM and ChS-70VM alloys [18-24].

Table 5. Thickness (h) and microhardness (Hμ) of thermal diffusion coatings on alloys ЧС-70ВМ and EP-539ЛМ (after testing) (according to the 1985 report, Table 4.2)

No. of saturating mixture	Alloy	In its original state		After testing T = 950 °C, 130 h	
		h, μm	Hμ, MPa	h, μm	Hμ, MPa
1	ChS-70VM	30–35	7500	65–75	8500
1	EP-539LM	50–55	8500	65–75	8500
2	ChS-70VM	40–45	8000	80–85	8000
2	EP-539LM	40–45	8500	–	–
3	ChS-70VM	55	8800	90–100	7400
3	EP-539LM	50–55	8500	90–100	8000
4	ChS-70VM	45–55	8000	60–65	6000
4	EP-539LM	50–55	8500	70–80	6500

Comprehensive bench and engine tests have definitively confirmed the high operational efficiency of the developed coatings. On a gas-dynamic rig, at a maximum temperature of 1020°C and 2500 thermal cycles, the coating significantly inhibits the development of thermal fatigue cracks in the blade surface layer. After 600 hours of operation, heat-resistant nickel alloys with the Ni–Al–Si–Cr–La coating exhibit no visible signs of corrosion-erosion damage, whereas uncoated control samples are characterized by significant oxidation and pitting. The obtained data indicate that the optimized technology not only ensures the required coating thickness and microhardness, but also provides high durability of the material under conditions as close as possible to actual operating conditions. Thus, the results of the experimental studies fully confirm the correctness of the chosen approach to the compatibility of alloy saturation and heat treatment processes and open the possibility of widespread industrial implementation of the developed protective coatings.

4. Conclusions

1. The technology for thermal diffusion application of complex-alloyed protective coatings of the Ni–Al–Si–Cr–La system on heat-resistant nickel alloys ChS-70VM and EP-539LM has been optimized. It is fully compatible with the hardening heat treatment modes of the alloys, which ensures the preservation of the structural stability of the γ′-phase and high mechanical properties of the base.

2. The transition from traditional ferroalloys to multicomponent Fe–Al–Si ligatures (20–30% Al, 15–25% Si) significantly improves the technological efficiency of the saturation process, allows control over the kinetics of coating growth and the formation of more flexible and uniform protective layers with improved adhesion to the substrate.

3. The developed coatings provide a twofold increase in heat resistance and corrosion resistance compared to traditional aluminide coatings: weight gain after 500 hours at 950 °C does not exceed 14 g/m^2, and corrosion damage in molten salts and synthetic ash is virtually absent while maintaining surface microhardness at a level of 7500–9000 MPa.

4. Comprehensive bench tests (2,500 thermal cycles at 1,020 °C and 600 hours) confirmed the high resource efficiency of the coatings: they reliably protect special-purpose materials from corrosion and erosion damage in salt-laden environments, significantly enhancing the reliability of new-generation materials.

5. The obtained results open the possibility of developing a new generation of protective coatings with predetermined kinetic and structural characteristics, which enables the transition from empirical selection of process parameters to a scientifically grounded digital design of protective layers for modern dual-purpose materials.

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