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DEVELOPMENT OF RARE EARTH METAL-MODIFIED HEAT-RESISTANT COATINGS FOR GAS TURBINE BLADES

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This paper presents an investigation into the influence of Y-La and Y-Hf-La modifiers on the structure and properties of Ni-Cr-Al consumable cathodes used for heat-resistant coatings on turbine blades via the arc-ion plating method. The study shows that the introduction of these modifiers positively affects the structure formation process of the consumable cathodes. The modifiers contribute to a higher degree of microstructural homogeneity in the cathodes, achieved through the formation of nanosized stabilizing phases between the alloying elements and the rare earth metal groups. This study experimentally confirms that Ni-Cr-Al coatings produced with the Y-La and Y-Hf-La modified cathodes outperform traditional Y-only modified coatings in oxidation tests. Structural analysis of the coatings reveals that samples with Y-Hf-La exhibit greater homogeneity and fewer defects, which is particularly important when depositing coatings with large thicknesses (over 40 μm). It was found that the introduction of the Y-Hf-La complex enables the application of coatings up to 90 μm thick by forming a less defective structure. Additionally, it has been established that Y-Hf-La modification enhances the adhesion of the coating to the substrate and allows for maximum uniformity in the distribution of alloying elements throughout the entire thickness of the applied coating.

Introduction

Among the many tasks in the development of modern aircraft gas turbine engines, the problem of ensuring the reliability and durability of the hot-end turbine blades often stands out as the most critical one. The material and design of a turbine blade largely determine the permissible gas temperature in front of the turbine and its service life, which in turn affects the specific thrust, efficiency, and other characteristics of the engines [1].

Modern aircraft engines frequently have metal temperatures above 1150[0]C on the high-pressure turbine blades. In this case, the use of traditional diffused aluminide coatings is often insufficient due to the melting of the interfacial region between the β-NiAl layer and the base superalloy at temperatures above 1120[0]C [1]. On a macroscopic scale, this causes the protective coatings to wrinkle even before oxidation takes place. Microscopically, thermal fatigue cracks can occur on the coating surface due to the poor ductility of aluminides at lower temperatures. Stressed by the cyclic thermal loads in a gas turbine engine, these cracks, often found on the leading and trailing edges of an airfoil, can propagate quickly, ultimately resulting in the complete failure of the blades [2]. To overcome the limitation of diffused aluminide coatings, overlay heat-resistant coatings were developed. The most effective and common overlay heat-resistant coatings are those based on an M-Cr-Al formulation (M stands for metal, i.e. Ni, Co, or a combination of

these two metals). The durability of such coatings is primarily determined by the aluminum content in the surface layer, as well as the presence of various stabilizing modifiers [3].

A popular and robust process for applying the overlay heat-resistant coatings is the use of arc-ion plating (AIP), where a coating material is made into consumable cathodes. The cathodes are evaporated and ionized by electric arcs in a vacuum. The ions of the coating material are subsequently attracted to the workpiece due to the existence of a bias voltage, bombarding and condensing on the surface of the workpiece. The controlled particle energy of the ions and the high temperature at which they are deposited have a combined effect to ensure an increased adhesion and the formation a fine-grained structure for the coating [4]. Among many AIP equipment types, the multi-arc configuration shown in Fig. 1 was developed by the Ukrainian gas turbine manufacturer, which is optimized for coating turbine blades. Similar designs are also widely used by the aircraft and industrial gas turbine manufacturers elsewhere in the world [5]. In this equipment, up to eight disk-shaped cathodes are placed at selected locations in a vacuum chamber. The turbine blades are placed on a rotary carrier that spins about the central axis of the vacuum chamber. If required, the turbine blades are also able to rotate about their own axis to ensure an even exposure to the charged ions. A high degree of coating uniformity can be achieved on the turbine blades by such multi-arc design. A typical process cycle takes less than 4 hours to deposit a coating with up to 60 μm thickness on the turbine blades.
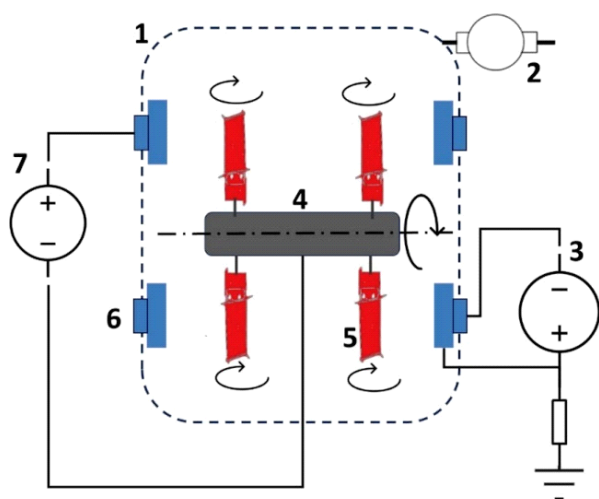

Fig. 1. Schematic diagram of multi-arc AIP equipment: 1 − vacuum chamber; 2 − vacuum pump; 3 − power source for arc discharging; 4 − rotary carrier; 5 − workpieces; 6 − cathodes; 7 − power source for bias voltage

Damages to overlay heat-resistant coatings on in-service turbine blades can frequently be attributed to the defects introduced during the AIP deposition process, such as heterogeneous microstructure and element segregation [6]. These defects are largely affected by the homogeneity of the microstructure and chemical composition of the consumable cathodes. In nickel-based superalloys, rare earth metal (REM) including Y, Hf and La are often introduced into the alloying composition as modifiers in order to form nanosized phase precipitates that stabilize the structure of the alloy and increase the high-temperature properties [7]. These elements actively interact with impurities (mainly with carbon and oxygen) to form stable carbides and oxides at various interfaces (phase-to-phase boundaries, dislocation clusters, etc.). Similarly, in coating applications, M-Cr-Al heat-resistant coating materials are almost invariably produced by microalloying with Y elements [8] as a modifier. The effects of other REM doping in the alloy and coating compositions have also been studied and proved to increase the anti-oxidation property of the coatings by delaying the delamination of the aluminum and chromium oxide films from the engine part surfaces [9,10].

Since the concentration of the REM modifiers is at the level of tenths of a percent, ensuring their uniform distribution is a challenging task in the manufacturing process of the consumable cathodes. The authors previously investigated the effects of REM modifiers and different types of manufacturing processes on the quality of consumable cathodes. The experimental findings were reported elsewhere [4,11,12]. In the present study, we manufactured Y-La and Y-Hf-La modified cathodes and characterized the coatings via both metallurgical examination and high-temperature cyclic oxidation tests. The results were compared to those of the baseline production Ni-Cr-Al-Y coating and reported in this paper.

Experimental

To study the effect of modifiers, three coating material variants were manufactured into alloy ingots. The first two variants were doped with Hf-La-Y and La-Y modifiers, respectively, and the last variant with only Y as a reference (baseline production coating, known as SDP-2). Each variant of the ingots was then cast into disk-shaped cathodes, which were subsequently used in the AIP equipment to produce coatings on coupon specimens and turbine blades made from ZhS32-VI single crystal superalloy. Metallurgical examinations were carried out on both the ingots and the finished coatings. Finally, the coated specimens and turbine blades were subject to high- temperature cyclic oxidation tests in an air furnace to compare

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their heat resistance properties.

Manufacture of ingots

To obtain the three variants of coating alloy ingots, a vacuum-arc skull remelting unit [12] was used, which allowed the fine dispersion of the raw materials (in the form of powders or pellets) to optimize the homogeneity of the resulting ingots. The target elemental contents (wt.%) of each alloy variant are shown in Table 1.

Melting was carried out by applying the following process parameters: current of 450 to 1800 A; voltage of 35 to 40 V; mixed inert gases Ag/He (70/30); and pressure of 0.5 atm. For each ingot variant, the melt was held at 1550 to 1750^0C for 25 to 30 minutes before being poured into a preheated steel mold. The use of the above parameters ensured the homogeneous mixing of all alloying elements to produce high-quality remelting stocks.

Subsequently, the coating cathodes were made by remelting the ingots in a graphite crucible and casting into a graphite mold. The castings were then machined into disk- shaped cathodes using a lathe to the final dimensions shown in Fig. 2. The recess on the arc-ionization surface and the slight taper on the cathode side surface relative to the central axis are both due to a consideration of process know-how, which is not disclosed in this paper.

Chemical analysis and metallurgical examinations

The determination of the chemical composition of the coating materials was carried out by X-ray spectrometry on an Olympus DPO-2000 portable spectrometer. Additionally, the chemical composition was double-checked on an optical emission SPECTROMAX spectrometer to rule out any spurious measurement.

Optical metallographic examinations for the macro and microstructures were carried out on a Zeiss Axio Observer optical microscope and a Nikon SMZ 745T binocular stereomicroscope.

The chemical composition, microstructure and the distribution of the main alloying elements were also investigated on a Joel JSM- IT300LV multipurpose scanning microscope equipped with an X-Max 80 X-ray spectral energy-dispersive microanalysis (XMSMA) system. When examining the cross section of a sample, the system is capable of producing color maps for individual chemical elements where a denser color on a map corresponds to an increased content level of the element under study.

Manufacture of coated samples

Coated samples were produced in the AIP equipment using the three variants of cathodes. After aqueous rinsing and sand blasting operations, a combination of coupon specimens and turbine blades were loaded into the vacuum chamber of the AIP equipment where the pressure was evacuated to $6.65 \cdot 10^{-8}$ MPa or below. In this study, the sample surfaces were subject to ionic cleaning before the coating deposition process began. The ionic cleaning process lasted 3 minutes by applying an arc discharge current of 250 ± 10 A on the cathodes and a bias voltage of 1500 V between the cathodes and samples. The coating deposition process took 120 minutes under an arc discharge current of 300 ± 10 A and a bias voltage of 15 ± 1 V. A 30 to 50 µm thick layer of coating was deposited on the sample surfaces, which was within the turbine blade specifications. The coated samples were then left in the vacuum for 60 to 90 minutes to cool down before removal into the ambient.

Table 1

Chemical composition of the experimental coating alloys

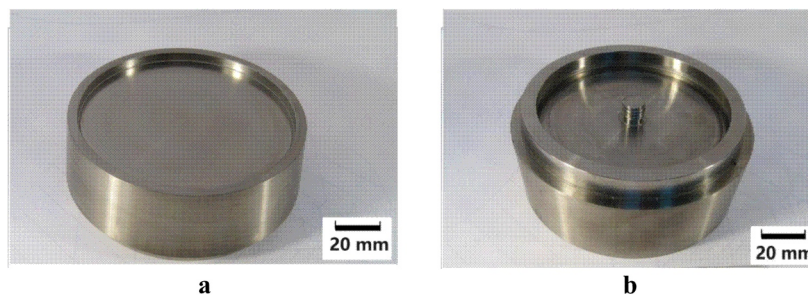| Alloy variant	Chemical composition, wt.%						
	Ni	Cr	Al	Hf	La	Y	other (impurities)
1	balance	18–22	11–13	–	0.2–0.4	0.3–0.6	≤1.5
2	balance	18–22	11–13	0.2–0.4	0.1–0.3	0.3–0.6	≤1.5
3	balance	18–22	11–13	–	–	0.3–0.6	≤1.5

Fig. 2. Cathodes for AIP process: a − arc-ionization surface; and b − back surface with fixing boss

Results and discussion

Structure and chemical composition of cathodes

The chemical analysis results for the three variants of cathodes are presented in Table 2. It can be seen that the elemental contents of the cathodes broadly comply with the target levels (Table 1), with minor deviations for Al, Hf and La. Due to the experimental nature of the work, all cathodes were deemed acceptable for testing purposes. All three variants of the cathodes were subject to production inspection procedures for macro-level defects. The inspection results showed that the cathodes were dense castings, free of macro-porosity (piping) and cracks.

The results for cathode with Y-modifier only (Variant 3) are presented in Fig. 3. It can be seen that the cathode material is a Ni-based solid solution with the presence of intermetallic Ni-Cr-Al phases. The crystallite sizes of the solid solution and intermetallic phases are between 25 and 150 microns. In contrast, the Y phase distributed over the cathode material takes the form as individual inclusions within the separation boundaries of other phases, typically less than 5 μm in size. The even distribution of the small Y inclusions between the larger intermetallic phases demonstrated the good solubility of Y modifier in the alloy and the homogeneity of the structure.

The same analysis was performed on the cathode samples with Y-La modifier (Variant 1) and the results are shown in Fig. 4. There were no discernable macro- or microstructure defects discovered in the analysis. Compared to Variant 3, a more refined and balanced alloy structure was obtained in the cathodes for Variant 1 with the Y-La phase evenly distributed over the entire volume of the body. The sizes of the solid solution and intermetallic phases range from 8 to 140 μm, with some phases exceeding 200 μm. As with Variant 3, the Y and La inclusions are mainly distributed within the boundaries between other phases, ranging from 4 mm and 10 mm in size. Overall, it is clear that the Ni-Cr-Al-Y-La cathodes had a homogeneously dispersed microstructure.

The analysis results for the Y-Hf-La-modified cathode (Variant 2) are presented in Fig. 5. It can be seen that, similar to the Y and Y-La-modified variants,

the microstructure of the coating also has a dual-phase structure (solid solution and intermetallics), with their phase sizes ranging from 90 μm to 150 μm. The Y-Hf-La inclusions are 2 μm to 10 μm in size distributed across the entire dual-phase volume. It is worth noting that La and Y are also partially dissolved in the composition of the solid solution and intermetallic phases, while Hf is mainly located within their boundaries.

When comparing the results of all three variants of the cathodes, it can be concluded that the introduction of the Y-La and Y-Hf-La modifiers into the composition of the Ni-Cr-Al alloy has a greater influence on alloying structure formation than the Y only modifier. The Y-La and Y-Hf-La modifiers both allow the formation of a homogeneous structure in the samples. In particular, the introduction of the Y-Hf-La modifier has resulted in a structure characterized by a greater degree of homogeneity and fineness in comparison with that of Y and Y-La modifiers. There is also a noticeable positive effect of Y-Hf-La modifier on the uniformity of distribution of Al and Cr in the bulk of the material compared to the samples with Y and Y-La modifiers. The higher degree of distribution uniformity in the Y-Hf-La-modified material may lead to reduced segregations, compared to the Y and Y-La-modified materials, when being thermally cycled in turbine blade applications.

Characterization of coatings

In the study, each of the three cathode variants (with Y-La, Y-La-Hf and Y-only) was used to coat nine samples (6-off coupon specimens and 3-off turbine blades) in the AIP equipment, producing 27-off coated samples in total.

For the baseline Ni-Cr-Al-Y coating (Variant 3), as shown in Fig. 6, it can be seen that the structure of the coating sample is uniform, and free of defects such as porosity, cracks, and delamination. The Y modifier is distributed evenly over the entire thickness of the coating. A thin separation line is visible in the transition zone between the substrate and coating due to the alumina particles impinged into the surface of the samples during the sand blasting process. Frequently referred to as the

Table 2

Chemical analysis results for cathodes

Variant of cathodes	Chemical composition, wt.%						
	Ni	Cr	Al	Hf	La	Y	Impurities
1	65.18	20.5	13.7	–	0.16	0.46	≤1.5
2	67.18	19.06	13.19	0.12	0.09	0.37	≤1.5
3*	–	–	–	–	–	–	–

Note: * − production variant, known to comply with specification; no chemical analysis was performed in this study.

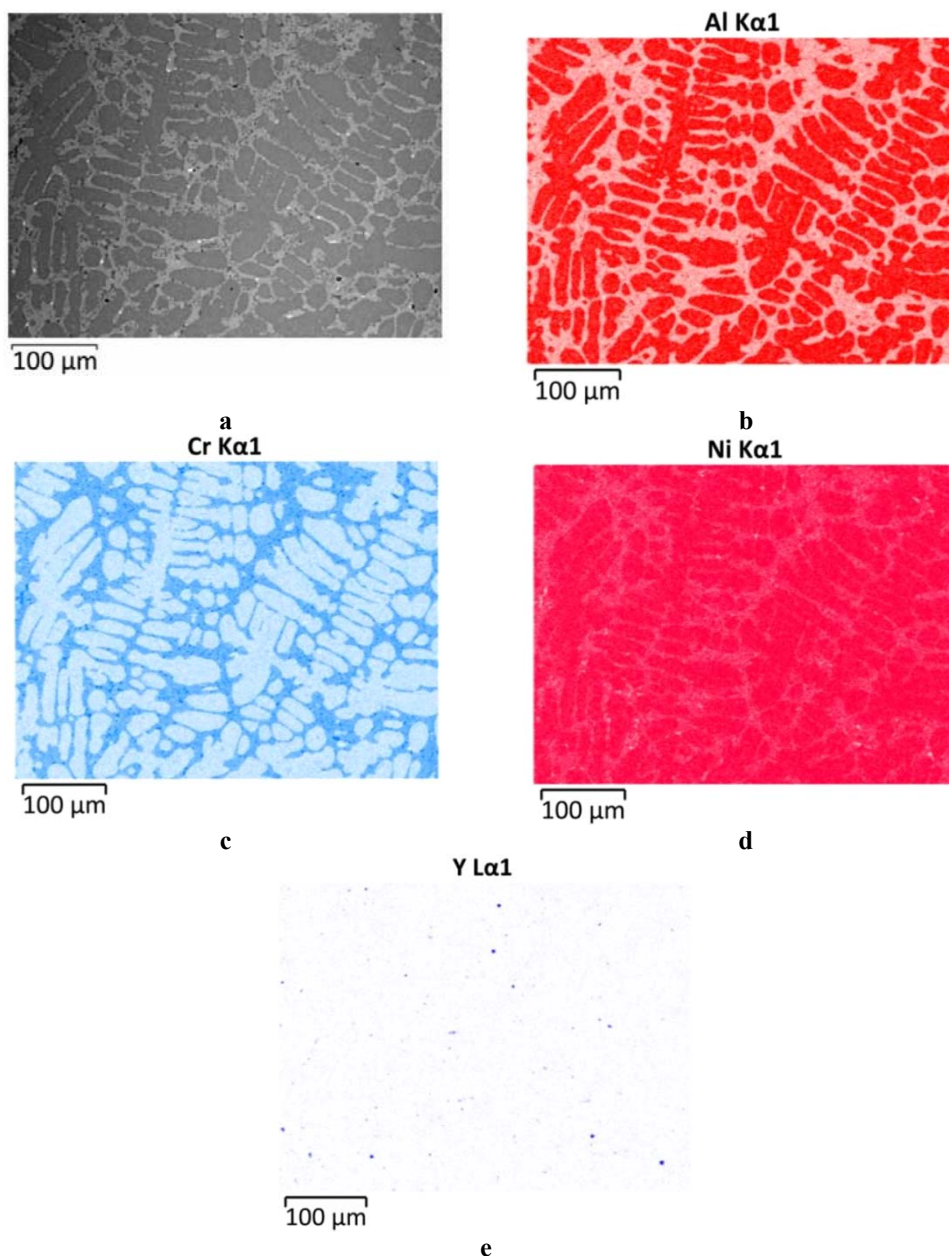*V.S. Yefanov, M.O. Gnatenko, H.M. Laptieva, Y.F. Basov, K.M. Sukhyy, S.V. Kovalyov, S.M. Popov*

Fig. 3. Microstructure and distribution of elements in the structural components of the Ni−Cr−Al−Y cathode (Variant 3): a − microstructure of the sample; b − Al distribution; c − Cr distribution; d − Ni distribution; e − Y distribution

«grit line», such defects are deemed as acceptable according to the quality standard of turbine blades. It was observed during the deposition process that the cathode material evaporated uniformly and the arc was stable without any disruptions.

The results concerning the Ni-Cr-Al-Y-La-modified coating samples (Variant 1) are shown in Fig. 7, where some localized areas with increased porosity can be seen, both in the coating itself and in the transition zone with the substrate. Such high porosity level is likely to negatively affect the strength and heat resistance properties of the coating in real service conditions. The presence of such a large number of voids with various shapes and sizes in the transition zone may result in poor adhesion between the applied coating and the base material. It should also be noted that during the coating process, it was observed that the electric arc often dwelled in localized zones on the cathode surface. Although the underlying mechanism is yet to be understood, this localized arc dwelling is clearly undesirable in that it tends to result in uneven material evaporation rates during the deposition process.
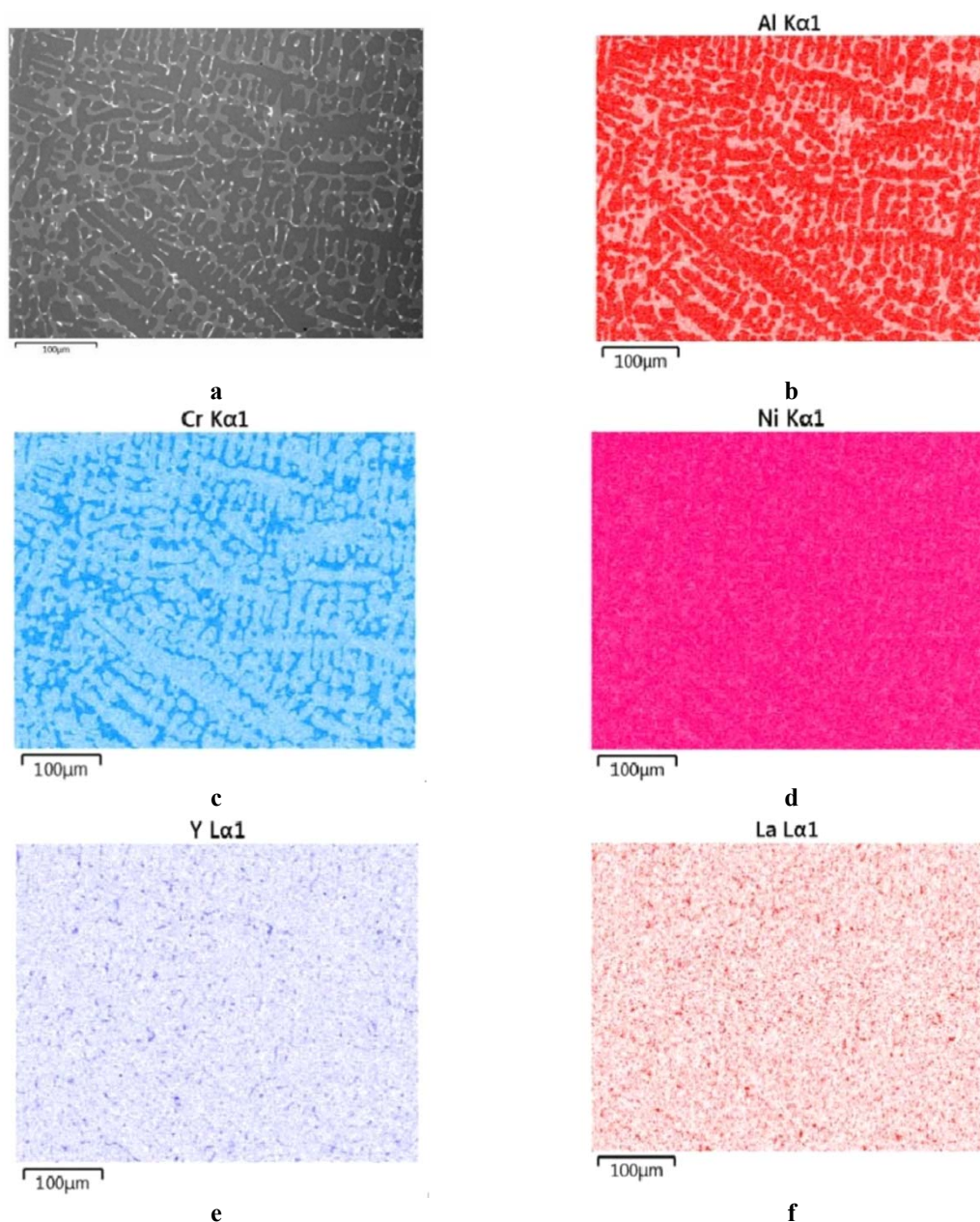
Fig. 4. Microstructure and distribution of elements in the Ni−Cr−Al−Y−La cathode (Variant 1): a − sample microstructure; b − Al distribution; c − Cr distribution; d − Ni distribution; e − Y distribution; f − La distribution

As shown in Fig. 8, the introduction of Ni-Cr-Al-Y-Hf-La modifier (Variant 2) has led to a significant decrease in the porosity level both in the coating itself and in the transition area with the substrate. In addition, no visible defects are present in the transition zone, indicating good coating adhesion to the base material. In comparison with the Y and Y-La-modified coatings, Y-Hf-La-modified coating has demonstrated the best metallographic quality, characterized by a greater degree of homogeneity and fewer defects. This is especially important when applying coatings of greater thicknesses (over 40 μm),

since an increase in the coating thickness inevitably leads to an increase in the total number of defects and hence the likelihood of failure. From Fig. 8,c,e, it is evident that the Y-Hf-La modifier reduces the segregations of alloying elements to a minimum, leading to an optimal uniformity of elemental distribution over the entire thickness of the applied coating.

Meanwhile, it is also evident that in the Y-Hf-La-modified coating, the uniformity of Cr distribution was not inferior to that with Y-only modifier. The distribution of Cr is of great importance
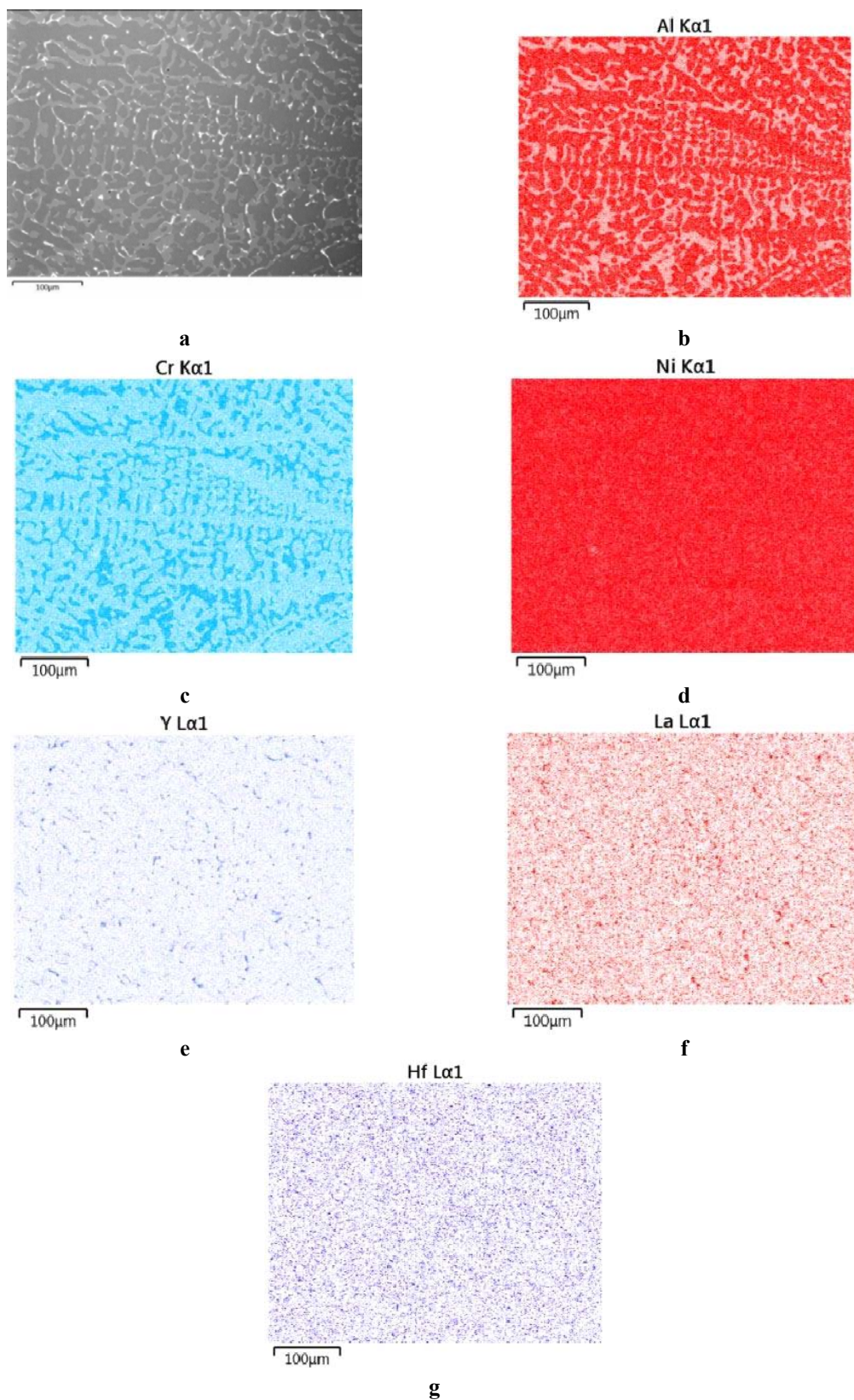
Fig. 5. Microstructure and distribution of elements in the structural components of the Ni−Cr−Al−Y−Hf−La cathode (Variant 2): a − sample microstructure; b − Al distribution; c − Cr distribution; d − Ni distribution; e − Y distribution; f − La distribution; g − Hf distribution
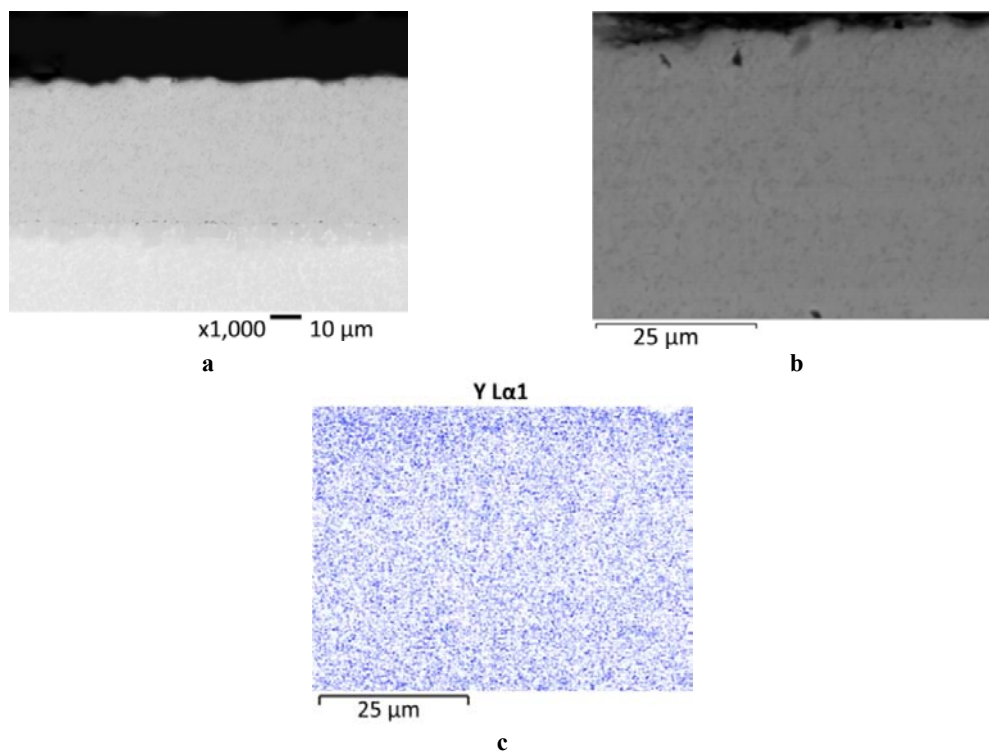
Fig. 6. Microstructure and elemental distribution in the Ni−Cr−Al−Y coating (Variant 3):
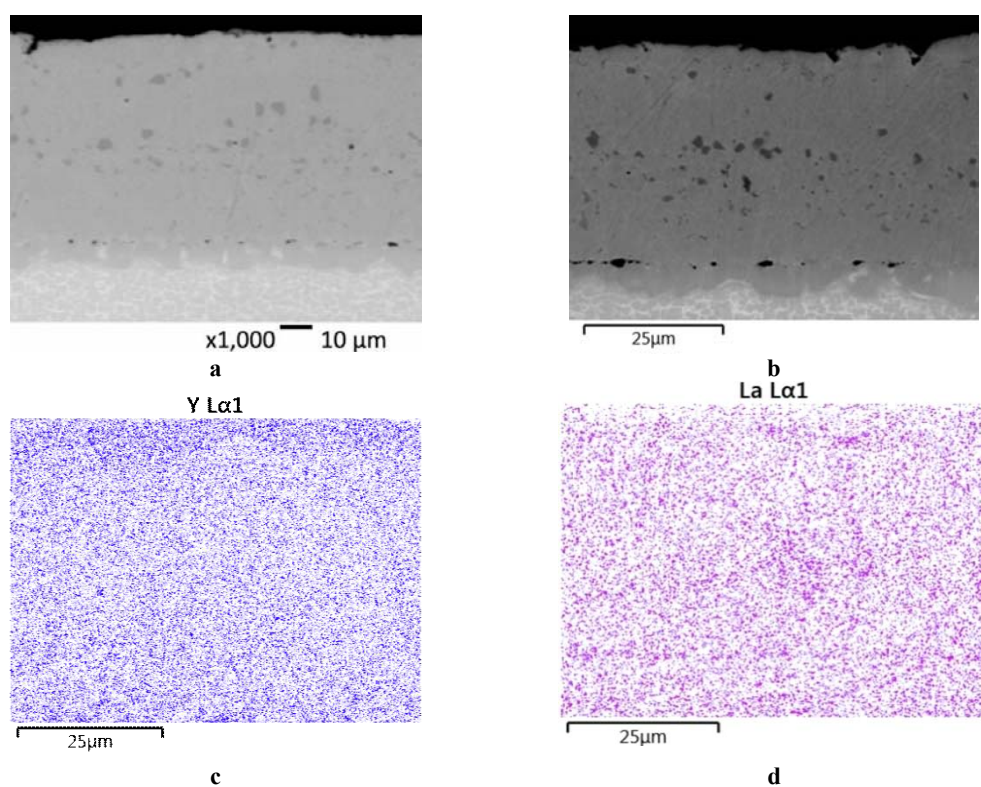a and b − coating microstructure; c − Y distribution [12]

Fig. 7. Microstructure and elemental distributions in the Ni−Cr−Al−Y−La coating (Variant 1):
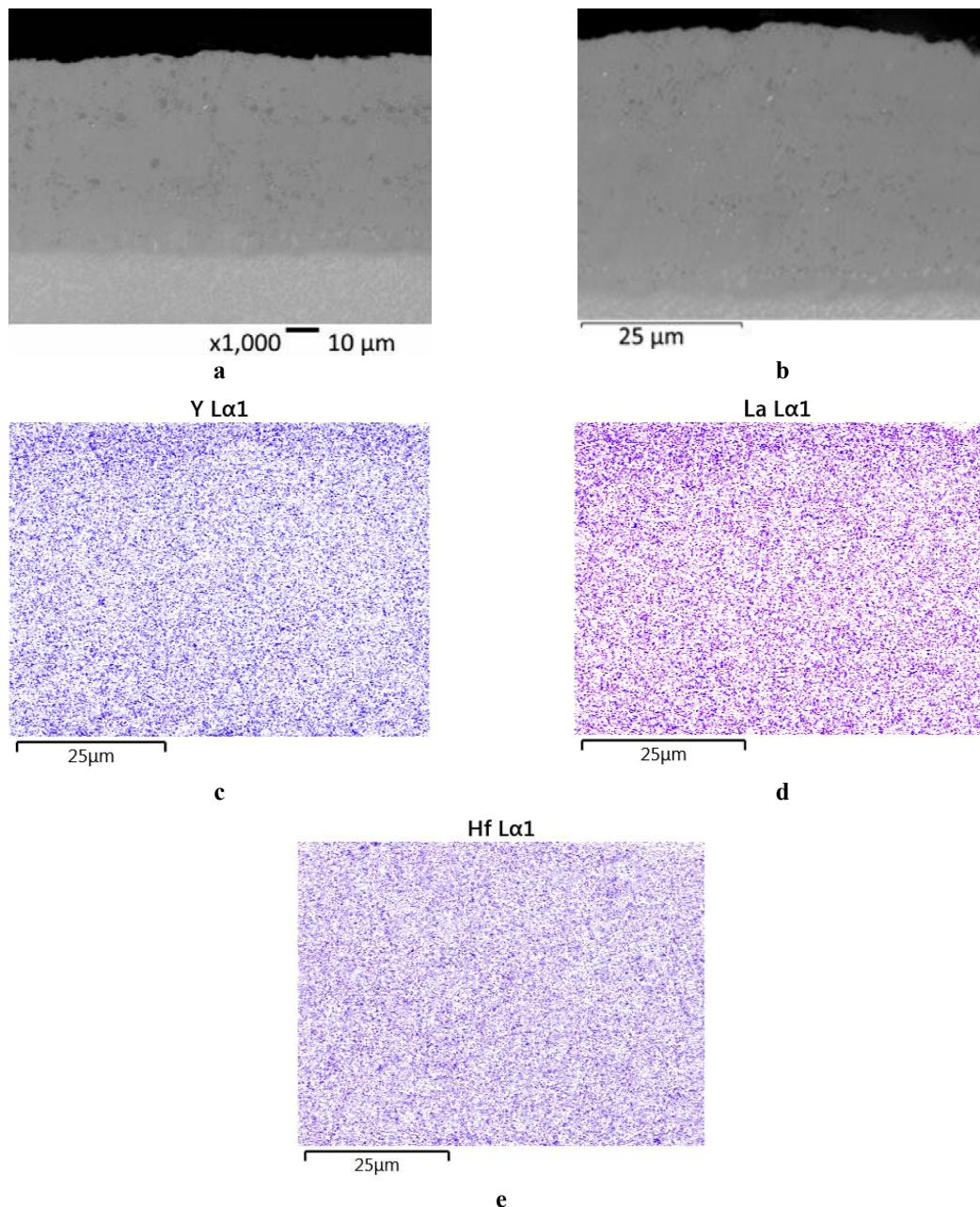a and b − coating microstructure; c − Y distribution; d − La distribution

Fig. 8. Microstructure and elemental distributions in the Ni−Cr−Al−Y−Hf−La coating (Variant 2):
a and b − coating microstructure; c − Y distribution; d − La distribution; e − Hf distribution

in the application of heat resistant coatings due to the fact that Cr is the main element in the alloy that forms a protective film of Cr_2O_3 on the metal surface and thus provides heat resistance. A uniform distribution of Cr in the coating ensures a uniform heat resistance property across the entire coated area and minimizes the chance of any localized premature in-service failures. It is also worth noting that the Y-Hf-La-modified cathodes evaporated stably under a steady electric arc during the deposition process.

Results of heat resistance test

The heat resistance property of the modified coatings was evaluated via cyclic oxidation tests. The tests were carried out on coupon specimens and turbine blades cast from ZhS32-VI single crystal superalloy, as shown in Fig. 9. The coated samples were deposited with Y-Hf and Y-Hf-La-modified coatings using the AIP equipment to a thickness of 30−50 μm. For comparison, uncoated samples and samples with baseline Ni-Cr-Al-Y coating were also manufactured and subject to the same testing procedure.
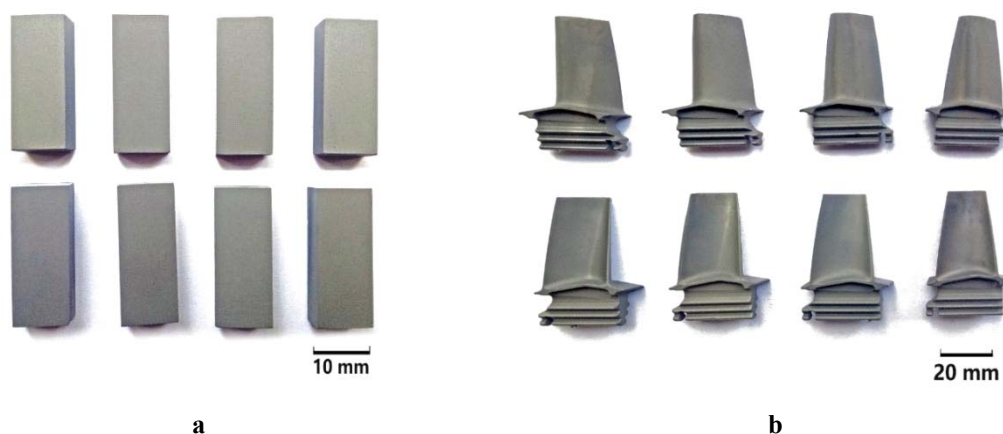
Fig. 9. Appearance of coated samples: a − coupon specimens; b − single crystal turbine blades of a turboprop engine

The cyclic oxidation tests were carried out by heating the samples in alumina crucibles to a temperature of 1050^0C in an electric air furnace. Each cycle included a dwell time of 100 h at this temperature, after which the samples were removed from the furnace and subject to non-forced ambient cooling. The mass changes of the samples were recorded up to four completed cycles (i.e. at 100, 200, 300 and 400 h). Although the findings are not included in this paper, visual and optical examinations were performed during the tests to determine the depth of corrosion damage and to observe the changes in the structure of the coatings. X-ray diffraction technique was also applied to analyze the phase composition of the damaged sample surfaces.

The test results are presented in Fig. 10, where the mass change values are the mean value of all nine samples of the same variant after completing each test cycle. As expected, the masses of the coated samples initially increased due to oxygen absorption and decreased subsequently due to the delamination of the formed Al_2O_3 film. In contrast, the uncoated samples showed a significant mass loss at the start of the test after only one cycle (100 h), which prompted an early termination of the uncoated tests.

From the test results, it is clear that the best anti-oxidation, i.e. heat resistance, property was achieved on the samples with Y-Hf-La-modified coating (Variant 2). Compared to the baseline samples with Y-only modifier (Variant 3), the Y-Hf-La-modified samples lost approximately 60% less mass after 400 hours of exposure at 1050^0C. Despite having an increased level of porosity defects seen in Fig. 7, the runner-up is the Y-La-modified coating (Variant 1), achieving a greater than 20% reduction in mass loss after 400 hours, compared to the baseline coating.
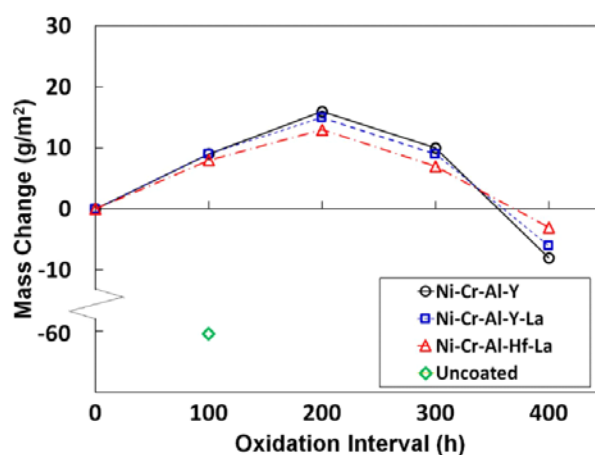

Fig. 10. Oxidation test results at 1050^0C

Conclusions

Based on the comparison of the results obtained on the cathodes and coatings with Y, Y-La and, Y-Hf-La modifiers, the following conclusions can be drawn:

(1) The rare earth metal modifiers help create a more refined and homogeneous structure in the cathodes and subsequently in the coatings.

(2) The Y-Hf-La modifier, in comparison with Y-La modifier, has a more significant and positive effect on the change in the microstructure of the coating material with a minimum segregation of alloying elements, a greater degree of homogeneity and a smaller number of defects.

(3) The oxidation tests have shown that the introduction of the Y-La and Y-Hf-La modifiers into the coatings may decrease the mass loss significantly at prolonged exposure times, indicating improved heat resistance compared to that of the baseline Ni-Cr-Al-Y coating.

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Following this study, it is the view of authors that the demonstrated advantages of the Y-Hf-La modifier makes it a promising candidate for replacing the baseline Ni-Cr-Al-Y coating, in the effort to extend the service life of the turbine blades currently in serial production.

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РОЗРОБКА МОДИФІКОВАНИХ РІДКІСНОЗЕМЕЛЬНИМИ МЕТАЛАМИ ТЕРМОСТІЙКИХ ПОКРИТТІВ ДЛЯ ЛОПАСТЕЙ ГАЗОВИХ ТУРБІН

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У даній роботі наведено результати дослідження впливу модифікаторів Y-La та Y-Hf-La на структуру та властивості витратних катодів Ni-Cr-Al, які використовуються для нанесення термостійких покриттів на турбінні лопасті методом електродугового напилення. Показано, що введення таких модифікаторів позитивно впливає на процес структуроутворення витратних катодів. При введенні модифікаторів можна досягти більшого ступеня однорідності мікроструктури в катодах за рахунок утворення нанорозмірних стабілізуючих фаз між легуючими елементами та групами рідкісноземельних елементів. У цьому дослідженні було експериментально підтверджено, що покриття Ni-Cr-Al, виготовлені з модифікованими катодами Y-La та Y-Hf-La, перевершують традиційне покриття лише з модифікатором Y у випробуваннях на окислення. Аналіз структури покриттів показав, що зразки з Y-Hf-La мають більший ступінь однорідності та менше дефектів, що особливо важливо при нанесенні покриттів великої товщини (понад 40 мкм). Встановлено, що введення комплексу Y-Hf-La дає можливість наносити покриття товщиною до 90 мкм за рахунок формування менш дефектної структури. Встановлено, що модифікація Y-Hf-La підвищує адгезію покриття до основи, а також дозволяє досягти максимальної рівномірності розподілу легуючих елементів по всій товщині нанесеного покриття.

Ключові слова: газова турбіна, лопасть, рідкісноземельний метал, катод, покриття, електроіонне покриття.

DEVELOPMENT OF RARE EARTH METAL-MODIFIED HEAT-RESISTANT COATINGS FOR GAS TURBINE BLADES

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This paper presents an investigation into the influence of Y-La and Y-Hf-La modifiers on the structure and properties of Ni-Cr-Al consumable cathodes used for heat-resistant coatings on turbine blades via the arc-ion plating method. The study shows that the introduction of these modifiers positively affects the structure formation process of the consumable cathodes. The modifiers contribute to a higher degree of microstructural homogeneity in the cathodes, achieved through the formation of nanosized stabilizing phases between the alloying elements and the rare earth metal groups. This study experimentally confirms that Ni-Cr-Al coatings produced with the Y-La and Y-Hf-La modified cathodes outperform traditional Y-only modified coatings in oxidation tests. Structural analysis of the coatings reveals that samples with Y-Hf-La exhibit greater homogeneity and fewer defects, which is particularly important when depositing coatings with large thicknesses (over 40 μm). It was found that the introduction of the Y-Hf-La complex enables the application of coatings up to 90 μm thick by forming a less defective structure. Additionally, it has been established that Y-Hf-La modification enhances the adhesion of the coating to the substrate and allows for maximum uniformity in the distribution of alloying elements throughout the entire thickness of the applied coating.

Keywords: gas turbine; blade; rare earth metal; cathode; coating; arc-ion plating.

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