Oxidation Resistance of NiCrAlY Coating Prepared by Arc Ion Plating

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With the rapid development of the aviation industry, the working conditions of the aero-engines have become even worse. With a rise in inlet temperatures, the aero-engine blades are easily oxidized at high temperature, which consequently reduces their service life. MCrAlY coating (M refers to Fe, Ni, Co, etc.) has good high temperature oxidation resistance. It is considered to be an effective method to solve the problem of high temperature oxidation of aero-engines blades, and has gradually become the current domestic and foreign scholars focus. Arc ion plating is a mature technology for the preparation of MCrAlY coating, which has been paid much attention. Based on the arc ion plating process, and Ni-base superalloy GH4133 for aero-engines blades, coating the surface with NiCrAlY coating, after heat treatment, high temperature oxidation resistance test, at a constant temperature of 920 °C, continuous oxidation test for 192 h, the micro-morphology, microstructure and oxidation process of the samples were analyzed by SEM, XRD, and EDS. The results: 1) NiCrAlY coating has a high density, there are many fine particles distributed on the surface of the coating, the surface is smooth, no micropores, pits and other defects, the coating and the matrix material formed a close bonding state; 2) after heat treatment, the particles on NiCrAlY coating surface are arranged more closely, the coating changed from NiAl phase to Ni_3Al phase, which improved the high temperature oxidation resistance of the coating; 3) during the initial stage of high temperature oxidation, a uniform and dense protective layer of Cr_2O_3 was formed on the surface of the coating, which effectively prevented the oxygen from diffusing into the inner of the coating, thus greatly improving the anti-oxidation performance; 4) during the oxidation process, the interdiffusion of Cr occurs at the interface between the coating and the substrate, mainly in the form of Cr element diffusing from the coating to the substrate. When the oxide layer Cr_2O_3 cracks or peels off, a new oxide layer can be formed after the substrate contacts with oxygen, which decreases the rate of oxidation weight gain and continues to play the role of high temperature oxidation protection.
Keywords: arc ion plating, coating preparation, NiCrAlY, high temperature oxidation resistance.

1. INTRODUCTION

Superalloys play an important role in the modern energy industry and aerospace industry, it is widely used and in great demand. It are found in gas turbines, spacecraft, rocket engines, nuclear submarines, petrochemical equipment, and fossil-fuel power stations. The most interesting thing is that it is widely used as the blade material of aero-engines.

For a long time, in order to continuously improve the performance of the aero-engines, it is necessary to constantly raise the temperature of the disaster wheel inlet. The blade of the disaster wheel is subjected to the erosion of high temperature gas for a long time. The metal materials used in such a bad environment should have good high temperature mechanical properties and good oxidation resistance.

At present, the high-temperature alloy used in aero-engines blades can not meet the needs of practical applications [1, 2]. Therefore, the use of advanced technology to improve the performance of the blade against high temperature has become increasingly important.

GH4133 nickel-base superalloy is widely used in aero-engines blades. In practical application, the alloy blades need to bear higher temperatures and periodic loads, which makes the surface of the superalloy blade more susceptible to oxidation corrosion phenomenon, thus causing strength failure of the blade, and reducing its service life [3 – 8].

In order to improve the high-temperature resistance of GH4133 Ni-base superalloy, the coating of MCrAlY (m means Fe, Ni, Co, etc.) is considered an effective solution. Arc ion plating (AIP) is an important method for the preparation of MCrAlY coatings in industry, which can achieve a high ionization rate of 70 % ~ 80 %, making it a process with the highest ionic density among all coating technologies [9 – 11].

Domestic and foreign scholars have carried out relevant research, and made some research results [12 – 14]. However, there are few analytical reports about the MCrAlY coating of GH4133 nickel base superalloy, especially the analysis of its high temperature oxidation resistance mechanism.

In view of this, a layer of MCrAlY coating was deposited on the surface of GH4133 Ni-base superalloy by arc ion plating. The performance and mechanism of high temperature oxidation resistance of coatings are analyzed, which provides a theoretical and data basis for the selection of high temperature protective coatings.

2. EXPERIMENT

2.1. Pretreatment of matrix material

Using GH4133 directionally solidified superalloy as matrix material, according to HB 5258-2000 standard [15],

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a standard sample of 30 mm × 30 mm × 5 mm was processed by wire cutted machine. The surface of the samples was sanded with 80#, 600# and 1000# sandpaper respectively, and then wet sprayed with 120# white corundum to make the surface roughness *R*a = 1.0 μm, under which condition, the sample coating has the highest preparation efficiency and the best preparation effect. Rinse the sample after wet spraying, and wash away the white corundum grains, then it was cleaned by the fully-enclosed single-arm automatic ultrasonic cleaning machine, blow-dried by an air gun, and put into the oven of 100 ℃ for 30 min.

2.2. Coating preparation

Using AS-700 ion-assisted vacuum coating equipment (Beijing Danpu Surface Technology Co., Ltd., China), the surface of GH4133 was coated with NiCrAlY coating, the thickness of which is about 65 – 70 μm, the arc current is 110 A, the bias voltage is -100 V and the pressure was 1Pa. During coating, argon ions ionized by high voltage were deposited on the substrate in a discontinuous manner under the action of pulse bias voltage. Table 1 shows the nominal composition of the prepared coatings.

Table 1. Nominal ingredients of NiCrAlY

Num	Element	Percentage
1	Al	13.8 %
2	Cr	21.2 %
3	Y	1.2 %
4	Ni	Allowance

2.3. Heat treatment

The coating deposited by arc ion plating is compact and uniform, but the internal stress is relatively high. In order to eliminate the internal stress, improve the microstructure of the coating and homogenize the alloy composition in the coating, a heat treatment process for the coating is required.

VH-446MMI vacuum heat treatment furnace was used. The deposited samples were placed in a high temperature resistant corundum ark, and then placed in the center of the furnace chamber where the temperature was uniform, ensuring a consistent heat treatment state of each sample.

The heat treatment process is: 1080 ℃ heat diffusion for 4 h, 870 ℃ aging for 32 h, cooled to 100 ℃ under the protection of N_2, then cooled to room temperature with the furnace. In the thermal diffusion process, the temperature rises in a gradient, that is, the temperature rises to 950 ℃ at first, and then to 1080 ℃ after holding for a certain time. This is because of the temperature difference at each point in the furnace, the temperature in the furnace chamber is slightly higher than the two ends, and gradient heating can ensure the furnace temperature as far as possible to become uniform.

2.4. High temperature oxidation test

SX2-12-17 box resistance furnace was used to carry out the oxidation experiment. According to the HB 5258-2000 standard [15] for Determination of Oxidation Resistance of Steel and High Temperature Alloy, the surface of the sample was treated and ground with 1000# sandpaper until the roughness *R*a = 0.63 ~ 1.25 μm. The NiCrAlY alloy samples after heat treatment were put into a 10ml corundum crucible, and the matching plates were covered to prevent the oxide scale from splashing out of the crucible and causing weight loss, which affected the experimental results.

The oxidation test was carried out in a box resistance furnace at 920 ℃ for 192 h. The samples were taken out after oxidation for a certain time, then cooled and weighed, and then put into the furnace for further oxidation, from which the oxidation time in the furnace was reciprocated until 192 h, the oxidation kinetics curves were obtained by sorting out the corresponding data.

2.5. Laboratory equipment information

The main experimental equipment used in this study is shown in Table 2.

Table 2. List of experimental equipment

No.	Content of the experiment	Equipment required	Models
1	Coating preparation	Ion assisted vacuum coater	AS-700
2	Heat treatment	Vacuum heat treatment furnace	VH-446MMI
3	High temperature oxidation test	Box type resistance furnace	SX2-12-17
4	Analysis of coating morphology	Scanning electron microscope	JEOL JSM 4800F
5	Element content analysis	Electron backscatter X-ray spectrometer	Ultim Max 40
6	Phase analysis	X-ray diffractometer	Rigaku D/max-II B X-ray diffractometer

3. DISCUSSION AND ANALYSIS

3.1. Analysis effect of heat treatment of coating

Although arc ion plating has a series of advantages, the prepared coating (deposition state) can not avoid problems such as relatively high internal stress, and uneven distribution of the composition. In order to prevent the cracking of coatings due to stress during high temperature oxidation, it is necessary to carry out stress reduction experiments. Vacuum annealing and aging heat treatment are considered as an effective solution. On the one hand, it can promote the metallurgical bonding between the matrix and the coating and improve the bonding strength, on the other hand, it can also homogenize the structure and make the distribution of alloy elements in the coating more uniform [15, 16].

3.1.1. Effect of heat treatment on the phase composition of the coating

Fig. 1 shows the surface and cross-sectional topography of NiCrAlY coating before and after heat treatment. As can be seen from Fig. 1 a, the deposited coating forms a smooth surface with a high density, and the surface is distributed with numerous fine particles without holes. As can be seen from Fig. 1 b, after heat treatment, the refined surface of the

coating grains is no longer smooth, but becomes slightly rough.

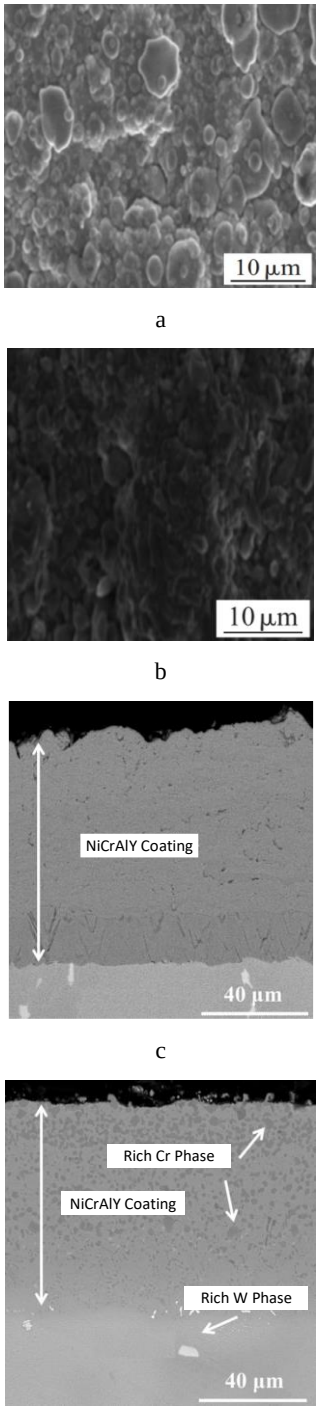


Fig. 1. The surface and cross-section morphology of NiCrAlY coating before and after heat treatment: a – coating surface before heat treatment; b – coating surface after heat treatment; c – coating cross-section before heat treatment; d – coating cross-section after heat treatment

Prepare the sample in accordance with aviation standard HB5258-2000, and completely cover the sample. As shown in Fig. 1 c, the non-obvious coating delamination on the surface of the deposited sample is caused by the process of full-coating treatment, not by the arc ion plating technology itself. No adverse effects on coating composition, structure, adhesion, performance, etc. [17, 18]. After heat treatment, the boundary between the coating and the basic layer has become blurred, which indicates that the coating composition has been fully diffused. The interface between the substrate and the coating is eliminated, and element diffusion occurs between the coating and the substrate. At the same time, the coating is distributed in several uniform fine gray "Spots" as shown in Fig. 1 d. By EDS analysis, the content of Cr was 76.13 % (mass fraction), which was confirmed as rich Cr phase. The content of W in the bright white phase was 50.16 %, which was a rich W phase.

3.1.2. Effect of heat treatment on the phase composition of the coating

Fig. 2 shows the XRD pattern of NiCrAlY coating before and after heat treatment. As can be seen from Fig. 2, the main phase of the deposited coating is γ′-Ni_3Al and contains a small amount of β-NiAl. After heat treatment, the main phase in NiCrAlY coating does not change. However, the decrease of β-NiAl phase value and the disappearance of some small peaks accompanied by the formation of new peaks of γ′-Ni_3Al phase indicate that the transformation of β-NiAl to γ′-Ni_3Al occurs, γ-Ni＋β-NiAl→γ′-Ni_3Al.

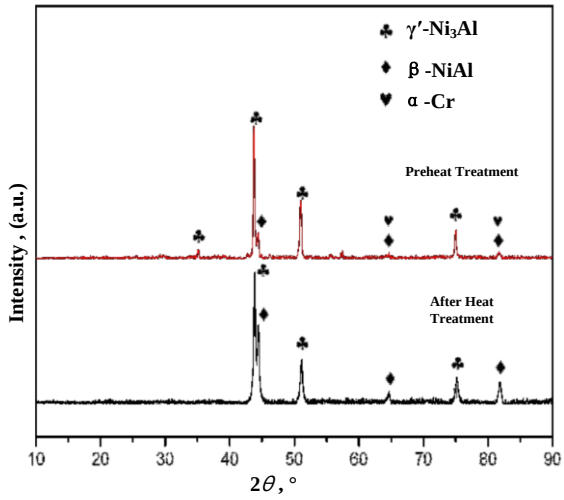


Fig. 2. The XRD patterns of NiCrAlY coating before and after heat treatment

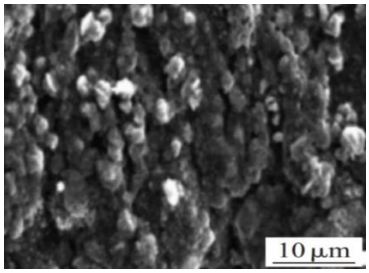
At the same time, a new characteristic peak of α-Cr phase appears in the figure, which is consistent with the detection of cross-sectional topography, and further verifies the accuracy of the above analysis. Because γ′-Ni_3Al belongs to a kind of strengthening phase [19, 20], with the heat treatment, the γ′-Ni_3Al strengthening phase in the coating increases gradually. It can be analyzed that the high temperature oxidation resistance of the coating after heat treatment is improved compared with that of before heat treatment.

3.2. Effect of high temperature on coating

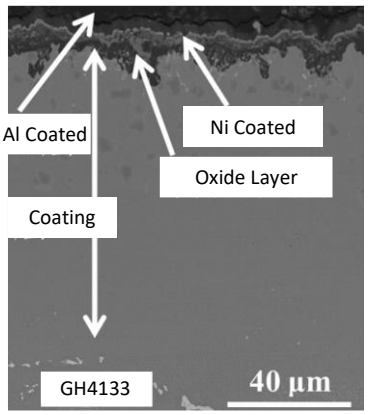
In order to study the effect of active elements on the high temperature oxidation behavior of the coating, the coating was oxidized at constant temperature for 192 hours in static air at 920 °C. Employing SEM, XRD and EDS, the microstructure, phase composition, and alloying element distribution of the coating were studied.

3.2.1. Effect of high temperature oxidation on the microstructure of the coating

In order to further analyze the transformation of the coating in the oxidation process, the morphology of the coating at 920° oxidation time was analyzed. Fig. 3 shows the micro-morphology of NiCrAlY coating at different oxidation times.



a



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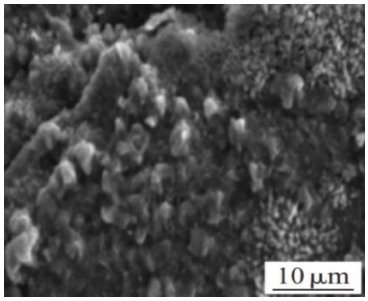

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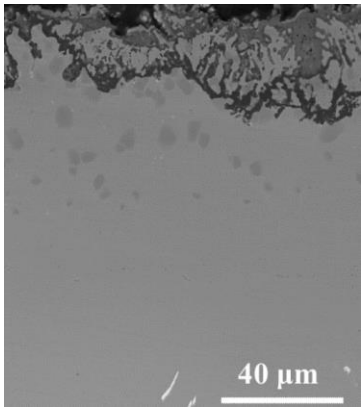

d

Fig. 3. The microstructure of NiCrAlY coating oxidized at different times: a – 48 h surface; b – 48 h section; c – 144 h surface; d – 144 h section

As shown in Fig. 3 a, after 48 h oxidation treatment of the coating, a large number of spinel-like and a small number of granular grains were formed on the coating surface, which, combined with energy spectrum analysis, may be $NiCr_2O_4$ andα-Al_2O_3, respectively. The particles on the coating surface were loose, but no cracks were found.

As shown in Fig. 3 c, after 144 h oxidation treatment, cracks and holes appear in the coating and begin to fall off. Oxygen can enter the coating through these holes, which aggravates the oxidation and crack formation, it also accelerates the spalling of the surface, thus exposing the fresh surface to further oxidation.

In order to observe the cross-section morphology of the oxide coating, an Al protective layer is evaporated on the outer surface of the coating during sample preparation to prevent the oxide layer from spalling off.

As shown in Fig. 3 b, after 48 h oxidation treatment, a dense oxide film was formed on the surface of the coating. XRD analysis showed that the main component was Cr_2O_3, and the thickness was about 4 μm, the oxide layer thickens and becomes loose. In addition, an obvious interdiffusion layer can be observed at the interface between the substrate and the coating, which is due to the non-uniform distribution of elements in the two parts, under the action of high temperature, the interdiffusion appears and the interdiffusion layer is formed.

As can be seen from Fig. 3 d, after 144 h oxidation treatment, the oxide layer thickness of the coating increased by about 5 μm, and it was no longer uniformly dense, and it grew towards the inner of the coating. This is because the high temperature oxidation resistance of Cr_2O_3 is comparable to that of dense Al_2O_3, and the protective effect of Cr_2O_3 on the matrix is limited. However, there are some defects such as vacancy and dislocation in the coating itself, and O_2 infiltrates into the coating where the defects are enriched, which accelerates the oxidation rate. Therefore, the oxidation process of the coating becomes non-uniform, and the surface of the coating appears more serious spalling phenomenon. At the same time, it can be observed that the gray rich Cr in the coating changes from uniformly dispersed in the coating at 48 h to concentrated on the surface of the coating and is constantly consumed. This further shows that the protective effect of the oxide layer with Cr_2O_3 as the main component can be effectively carried out under the current high temperature environment, but when the high temperature environment is intense, good resistance to high temperature oxidation can not be guaranteed [21, 22].

3.2.2. Effect of high temperature oxidation on the phase composition of the coating

The Fig. 4 shows the XRD pattern of NiCrAlY coating oxidized at 920 °C at different times. When NiCrAlY coating was oxidized for 48 h, the oxides such asα-Al_2O_3, Cr_2O_3 and $NiCr_2O_4$ were formed on the surface, and Cr_2O_3 was dominant. This indicates that the Al in the coating is rapidly consumed during the initial oxidation stage, so the protective effect of the coating is limited during high temperature oxidation, and the dynamic infiltration of O can not be effectively prevented. When NiCrAlY coating was oxidized for 144 h, the peak value of Cr_2O_3 decreased, which indicated that Cr in the coating had diffused to the

surface, and the coating was consumed with the spalling of the coating.

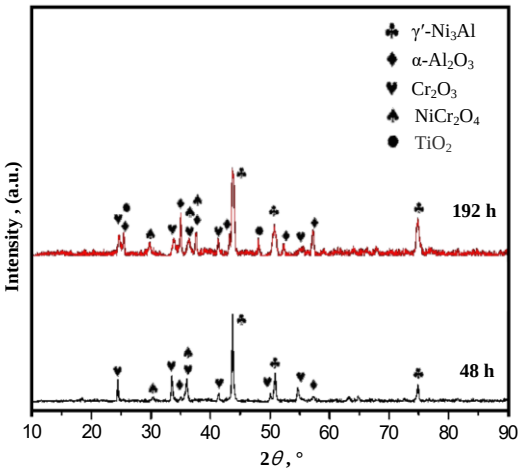

Fig. 4. XRD patterns of 920 °C NiCrAlY coating after different oxidation time

In addition, a small amount of TiO_2 was formed, but no Ti was found in the coating, so it is possible that Ti in the matrix diffused out and diffused to the surface of the coating to form oxide TiO_2. At the same time, the peak value ofα-Al_2O_3 decreases, which indicates that Al in the coating is greatly lost, and there is no longer enough Al to form a uniform and dense oxide layer to protect the coating. The oxidation resistance of α-Al_2O_3 is higher than that of Cr_2O_3, which indicates that the oxidation resistance ofα-Al_2O_3 is weaker than that of Cr_2O_3. The peak value of oxide $NiCr_2O_4$ decreases and some characteristic peaks disappear, which is also due to the large loss of Cr with coating exfoliation, so there is not enough Cr to combine with Ni and O to form $NiCr_2O_4$.

The above analysis also proves that the coating can carry out effective oxidation protection under the current high temperature environment of 920°, but when the high temperature environment is severe, it can not ensure that the coating has good high temperature oxidation resistance.

3.2.3. Analysis of oxidation kinetics at high temperature and constant temperature

The oxidation kinetics of NiCrAlY coating was measured and analyzed in a constant temperature oxidation furnace at 920 ℃ for 192 h. In order to better eliminate the error, during the oxidation process, the uncoated samples are placed in the furnace at the same time as a contrast.

Fig. 5 shows a comparison of the rate of oxidation gain between NiCrAlY coating and GH4133 substrate at 920 ℃. As can be seen from Fig. 5: 1) the oxidation kinetic curve of the matrix material is approximately linear, because the material contains about 5 % Al, which can form a dense Al_2O_3 protective film during the oxidation stage, which hinders further contact between the matrix and oxygen and inhibits the oxidation process[23, 24]; 2) the kinetics curve of the oxidation process of NiCrAlY coating has a slight fluctuation phenomenon. In the early 24 h oxidation stage, the samples gain weight rapidly, and then the rate of gain gradually slows down. The main reason is that the oxidation resistance layer will be formed quickly on the surface of the coating during the high-temperature oxidation stage, which effectively hinders the oxidation reaction.

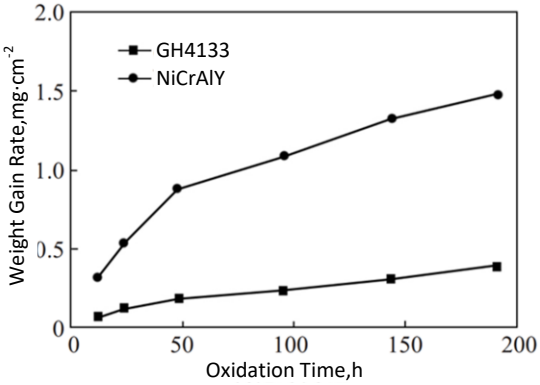

Fig. 5. Growth rate curve of 920 °C oxidation of coating and substrate

With the thickness of the oxidation layer increasing gradually, the oxide layer will crack and peel off. When the inner substrate contacts with oxygen, a new oxide layer can be formed, which makes the rate of gain weight decrease again and plays a protective role against high-temperature oxidation.

4. CONCLUSIONS

1. MCrAlY coating on the surface of nickel base superalloy, as an effective way to improve the oxidation resistance of Ni-base superalloy. NiCrAlY coating was successfully prepared on GH4133 nickel base superalloy by arc ion plating, The surface is smooth, and many small particles are distributed, with no micropores, pits and other defects.
2. After heat treatment of MCrAlY coating, the main phase γ'-Ni_3Al did not change, but γ'-Ni_3Al reinforced phase was added. The high temperature oxidation resistance of MCrAlY coating was improved obviously.
3. The high temperature oxidation resistance of the coating was tested: 1) at the initial stage of oxidation, a uniform and dense protective layer of oxide film Cr_2O_3 was formed on the surface of the coating, which prevented the oxygen from diffusing into the inner of the coating and enhanced the oxidation resistance of the coating; 2) during the oxidation process, the element diffusion phenomenon occurred at the interface between the coating and the substrate; 3) when the oxide layer Cr_2O_3 produced crack or spalling, the inner substrate could form a new oxide layer after contact with the oxygen, which reduced the oxidation rate and continued to play the role of high temperature oxidation resistance.
4. The preparation and properties of NiCrAlY coating on Ni-base superalloy were evaluated. It provides a theoretical reference for the design of MCrAlY coating on Ni-base superalloy with higher properties. The research results are of great significance to the development of MCrAlY coatings for improving the oxidation resistance of Ni-base superalloy. At the same time, it also enriches the application of MCrAlY coating in the field of oxidation resistance of Ni-base superalloy.

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