Microstructure and properties of ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating on stainless steel prepared by powder sintering one-step forming process

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Abstract

ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating on stainless steel was prepared by powder sintering one-step forming process. The surface-interface morphologies, chemical element distribution and phase compositions of the coatings before and after thermal oxidation or hot corrosion were analyzed using scanning electron microscope (SEM), energy dispersive spectrometer (EDS) and X-ray diffractometer (XRD). The microstructure of the coating was Ni$_3$Al matrix with dispersed ZrO$_2$ particles, which was pure and compacted. The Ni$_3$Al intermediate layer compactly formed between stainless steel substrate and ZrO$_2$/Ni$_3$Al composite coating, with good metallurgical bonding interfaces. The ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating exhibited a relatively high outmost microhardness of about 500 HV, and then gradually decreased from the coating to the substrate. When thermal oxidized or hot corroded at 1050°C, a mixed oxide layer of NiO, Al$_2$O$_3$ and NiAl$_2$O$_4$ formed on the coating surface, which effectively acted as a diffusion barrier for oxygen and corrosive substances, and thus demonstrated the good protective effect of ZrO$_2$/Ni$_3$Al coating.

1. Introduction

Intermetallic compounds have a series of excellent properties and are the most attractive new generation of high-temperature structural materials and surface coating materials [1–3]. At present, Ni-Al phases are the most studied intermetallics, among which Ni$_3$Al intermetallic is reported to have the advantages of high temperature strength, high temperature creep resistance, corrosion resistance and oxidation resistance. Besides, within a certain temperature range, the yield strength of Ni$_3$Al increases with the increase of temperature [4–8]. However, as a practical structural material, Ni$_3$Al has the disadvantages of low hardness, poor fracture toughness and insufficient high temperature strength. In such cases, it is an important development direction at present to combine Ni$_3$Al intermetallic with ceramics to produce composites with the advantages of both materials.

If a Ni$_3$Al coating is fabricated on steel surface, the oxidation resistance and corrosion resistance of the steel will be significantly improved, but the strength and wear performance, especially at high temperature, are still insufficient. Therefore, the coating properties are usually further improved by adding ceramic reinforced particles [9–13]. ZrO$_2$ ceramic particle is an ideal reinforced material for Ni$_3$Al coating. Compared with other reinforced materials, the thermal expansion coefficient of ZrO$_2$ is close to that of Ni-Al phases, and phase transformation can be induced by the action of stress in order to improve the toughness of the material. ZrO$_2$/Ni-Al composites not only present high temperature creep resistance, corrosion resistance, oxidation resistance, but also have higher specific strength, specific stiffness and high temperature wear resistance [14–18]. Therefore, ceramic reinforced intermetallic compounds have better properties and wider application fields than single intermetallics, thus holding a potential and broad prospect as coating materials.
Powder sintering technology has been widely used to prepare ceramic particle reinforced metal matrix composites, but it is rarely used to prepare composite coatings on metal substrates [19–22]. In present research, by using powder sintering one-step forming process, the in situ reaction synthesis of ZrO₂/Ni₃Al-Ni₃Al double-layer coating was synchronized with the powder metallurgy preparation of the steel matrix, which greatly simplified the production procedure of the coating. In addition, the heat and pressure in the sintering process promoted the denseness of the interface diffusion layer, which contributed to form a good metallurgical interface bonding. By a detailed analysis of microstructures for coatings, interfaces and cross sectional microhardness distributions, ZrO₂/Ni₃Al-Ni₃Al double-layer coating was comparatively studied with ZrO₂/Ni₃Al single-layer coating. The thermal oxidation behavior in air environment as well as the hot corrosion behavior in molten salt of Na₂SO₄ + 25% K₂SO₄ for ZrO₂/Ni₃Al-Ni₃Al double-layer coating were discussed. The study could provide a new idea for preparation of coatings on metal surface in the future, which will effectively promote the wide application of ceramic reinforced intermetallics coatings.

2. Experimental details

2.1. Preparation of coating

316L stainless steel powder (purity ≥99%, 200 mesh) was used as raw material of the experimental substrate. Ni powder (purity ≥99%, 200 mesh), Al powder (purity ≥99%, 300 mesh) and ZrO₂ powder (purity ≥99%, 300 mesh) were used as raw materials of intermediate layer and coating. 3Ni-Al system was selected as intermediate layer with a mol ratio of 3:1 for Ni and Al. The coating was made of 18% ZrO₂-3Ni-Al system, that was, ZrO₂ with the mass fraction of 18% was added into the 3Ni-Al system. The powders were weighed with JD200-2 electronic balance (accuracy 0.01 mg) according to the set ratio. Then, ZrO₂ as dispersed phase were pre-mixed with Ni and Al powders in a planetary ball mill Type QM-3SP2 for 8 h at 200 rpm to achieve a uniform mixing effect.

316L stainless steel powder, 3Ni-Al powder and 18% ZrO₂-3Ni-Al powder were successively put into the VYS-100 vacuum hot pressing sintering furnace. The sintering parameters were selected as follows: the sintering temperature of 1050 °C, the pressure of 40 MPa, and the holding time of 3 h. The samples with a size of 10 mm × 10 mm × 3 mm were cut off from the sintered products using an electric spark wire cutting machine, as shown in Figure 1. They were cleaned with ultrasonic cleaning instrument and then dried in a vacuum drying oven.

2.2. Thermal oxidation experiments

The thermal oxidation experiments were carried out in the air environment, with the experimental temperature of 1050 °C and the holding time of 10 h to 100 h in a step of 10 h. ZS-1021 type antioxidant coating was applied on all surfaces of the sample except ZrO₂/Ni₃Al coating surface (figure 1) to ensure that only this surface was oxidized. Then the sample was kept in a corundum crucible and the crucible containing the sample was put into the furnace which was preheated to a temperature of 1050 °C. After oxidation for a certain time, the crucible along with the specimen was taken out and cooled at the ambient temperature for 30 min. The weight of the crucible along with the sample before and after oxidation was measured by electronic analytical balance (with an

![Figure 1. Schematic diagrams for samples (a) ZrO₂/Ni₃Al single-layer coating, (b) ZrO₂/Ni₃Al-Ni₃Al double-layer coating.](image-url)
accuracy of 0.01 mg) to evaluate the weight gain of thermal oxidation. The exfoliated oxides falling into the crucible were included in the oxidation weight gain, and the average value of no less than three samples was taken as the weight gain value.

2.3. Hot corrosion experiments
The hot corrosion experiments were carried out in molten salt ($\text{Na}_2\text{SO}_4 + 25\% \text{ K}_2\text{SO}_4$), with the experimental temperature of 1050 °C and the holding time of 10 h to 100 h in a step of 10 h. ZS-1021 type antioxidant coating was applied on all surfaces of the sample except ZrO$_2$/Ni$_3$Al coating surface (figure 1) and the weight of the sample coved with antioxidant coating was measured. Before entering the furnace, an initial coating of $\text{Na}_2\text{SO}_4 + 25\% \text{ K}_2\text{SO}_4$ paste was applied on ZrO$_2$/Ni$_3$Al coating surface so as to have approximately 2 mg of the paste per cm$^2$ of the coating surface area. Then the sample was kept in a corundum crucible and put into the furnace.

After corrosion for a certain time, the sample was taken out and cooled to room temperature. Then it was put into distilled water heated to boiling, and cooked for 0.5 h to completely dissolve the residual salt. The sample was dried in a drying oven and weighed with an electronic balance. The weighed sample was coated with new salt and the process above was repeated. The average value of no less than three samples was taken as the weight gain value for corrosion.

2.4. Microstructural characterization and mechanical properties
D/Max-2500/PC type X-ray diffractometer, JSM-7001F type scanning electron microscope and Inca Energy 350 type energy dispersion spectrometer were used to analyze the microstructure and phase composition of the coatings and oxide layers after thermal oxidation and hot corrosion. HVS-1000 type digital Vickers hardness tester was used to analyze the microhardness variation for cross section of the coatings. The applied load was 1.96 N (200 gf), and the loading time was 15 s.

3. Results and discussion
3.1. XRD analysis
Figure 2 illustrates the XRD patterns of ZrO$_2$/Ni$_3$Al coating and Ni$_3$Al intermediate layer. According to Ni-Al binary phase diagram, Ni$_3$Al, NiAl, Ni$_2$Al$_3$ and other compounds will probably be formed by the reaction between Ni and Al in the sintering process [23]. With the increase of sintering temperature, NiAl and Ni$_2$Al$_3$ will gradually decrease and finally form pure Ni$_3$Al phase [24]. XRD results showed that the coating was composed of Ni$_3$Al and ZrO$_2$ phases, and the intermediate layer contained single Ni$_3$Al phase without other impurities generated. It could be seen that Ni and Al powders fully reacted to form pure Ni$_3$Al phase under the action of sintering temperature and pressure of the experiment.

3.2. SEM morphology
Figure 3 shows SEM morphology of ZrO$_2$/Ni$_3$Al single-layer coating and ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating on stainless steel, respectively. In figure 3(a), the left was ZrO$_2$/Ni$_3$Al coating with a thickness of about 0.8 mm, and the right was stainless steel substrate. A large number of cracks and pores can be observed in the...
interface area between ZrO2/Ni3Al coating and stainless steel substrate (labeled ‘interface 1’). The presence of ZrO2 particle phase changed the two-phase bonding of Ni3Al coating and stainless steel into three-phase bonding, resulting in a non-compact interface (figure 3(b)). From the morphology of the coating surface as shown in figure 3(c), it could be observed that fine ZrO2 particles were evenly dispersed on Ni3Al matrix.

In figure 3(d), the outermost layer was ZrO2/Ni3Al coating (about 0.8 mm thickness), the middle layer was Ni3Al intermediate layer (about 0.8 mm thickness), and the innermost layer was stainless steel substrate. Figures 3(e) and (f) shows the interface between ZrO2/Ni3Al coating and Ni3Al intermediate layer (labeled ‘interface 2’) and the interface between Ni3Al intermediate layer and stainless steel (labeled ‘interface 3’), respectively. It could be noticed that a good metallurgical bonding was obtained at ‘interface 2’ as well as at ‘interface 3’. The more excellent interfacial bonding was attributed to two factors: (1) Ni3Al intermetallic was used as the intermediate layer to improve poor wettability and reduce interfacial bonding stress between ZrO2/Ni3Al coating and steel substrate. (2) The in situ reaction synthesis of the coating was synchronized with the powder metallurgy preparation of the steel matrix, and consequently the heat and pressure in the sintering process promoted the denseness of interface diffusion layer.

3.3. Microhardness analysis
Figure 4 shows the microhardness distribution on the cross section of ZrO2/Ni3Al single-layer coating and ZrO2/Ni3Al-Ni3Al double-layer coating on stainless steel. As shown in figure 4(a), the coating layer exhibited a high value of microhardness, and dramatically decreased from outside in. Due to the presence of ZrO2 hard particles, the hardness of ZrO2/Ni3Al coating was significantly higher than that of stainless steel, which was about 500 HV. Once subjected to a large external force, steel substrate would undergo plastic deformation, and thus ZrO2/Ni3Al coating was prone to spall due to the great hardness difference between the coating and the substrate. As can be seen from figure 4(b), for ZrO2/Ni3Al-Ni3Al double-layer coating, the outmost microhardness was close to that of ZrO2/Ni3Al single-layer coating. Moreover, because of the existence of Ni3Al phase, the microhardness gradually decreased from the coating to the intermediate layer, then to steel substrate, presenting a flat gradient variation trend. The microhardness of Ni3Al intermediate layer was higher than that of steel matrix, about 400 HV, which provided a better support for ZrO2/Ni3Al coating, thus furtherly improved the overall performance of the coating.

3.4. Thermal oxidation behavior
The kinetic curve of ZrO2/Ni3Al-Ni3Al double-layer coating thermal oxidized at 1050 °C is shown in figure 5. The exfoliated oxides falling into the crucible were included in the oxidation weight gain. At the initial stage of oxidation, almost no exfoliated oxides were observed in the crucible. The weight gain of the sample was small, and increased slowly with the increasing of holding time. As the holding time increased from 30 h to 40 h, a lot of exfoliated oxides were found in the crucible and there was a significant increase in oxidation weight gain, indicating that the oxidation was accelerated by the spalling of oxide layer. Once the holding time exceeded 40 h,
trace amounts of exfoliated oxides were observed and the oxidation weight gain of the sample increased extremely slightly. When the holding time was in the range of 40 h to 100 h, the kinetic curve appeared to be approximately horizontal. That was to say, the oxidative weight gain of the sample was almost negligible. At 1050 °C, ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating demonstrate an approximately parabolic shape kinetic curve with the increasing of thermal oxidation time, displaying excellent thermal oxidation resistance.

The XRD patterns of ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating thermal oxidized at 1050 °C during different times were illustrated in figure 6. It could be seen that after thermal oxidation for 10 h, the coating presented an obvious oxidation phenomenon of forming mixed oxides of NiO, NiAl$_2$O$_4$ and Al$_2$O$_3$. It was found that NiO was the major oxide with median to strong signals detected in the XRD spectra for all samples, which was accompanied by NiAl$_2$O$_4$ and Al$_2$O$_3$ with weak signals. As the thermal oxidation time increased from 10 h to 30 h, the diffraction peak of NiO became stronger, indicating that the content of NiO in mixed oxides increased. When the thermal oxidation time increased above 30 h and further increased, the XRD patterns presented no obviously change, which indicated that the phase composition of the oxide layer tended to remain constant.

Combined with the analysis results of the thermal oxidation kinetic curve in figure 5, it can be concluded that in the initial stage of thermal oxidation, oxides content in the oxide layer increased significantly with the oxidation progressing. As the thermal oxidation time increased from 30 h to 40 h, the thickness of the oxide layer increased to a relatively high value, and then delaminated, forming exfoliated oxides. Due to the peeling of oxides, the fresh surface was exposed and the oxidation reaction was accelerated, thus the oxidation weight gain increased significantly. When the thermal oxidation time exceeded 40 h, the phase composition and thickness of the oxide layer tended to be constant, which adhered closely to the coating surface, hence the oxidation weight gain increased extremely slightly.

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**Figure 4.** The microhardness distribution on the cross section of the coatings (a) ZrO$_2$/Ni$_3$Al single-layer coating, (b) ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating.

**Figure 5.** The kinetic curve of ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating thermal oxidized at 1050 °C.
Figure 7 shows SEM morphology of ZrO2/Ni3Al-Ni3Al double-layer coating thermal oxidized at 1050 °C during different times. It was noticed that after thermal oxidation for 10 h, obvious oxidation occurred on coating surface, forming a layer of uneven oxide film (figure 7(a)). After 30 h of thermal oxidation, the oxide layer formed on the coating surface became uniform and continuous (figure 7(b)). XRD results proved that NiO was the predominant phase of the oxide layer. As the thermal oxidation time increased, the oxide layer became more compacted, which prevented the further diffusion of oxygen and reduced the oxidation rate. Therefore, the oxidation weight gain increased extremely slightly after thermal oxidation for 40 h. The NiO-dominated oxides were attached to the coating surface, and their grains were closely arranged in the shape of irregular fine polyhedron (figure 7(c)). With the extension of thermal oxidation time, the grain size became larger (figure 7(d)).

X-ray mappings for ZrO2/Ni3Al-Ni3Al double-layer coating thermal oxidized at 1050 °C after 100 h are illustrated in figure 8. According to the cross sectional morphology of the coating after thermal oxidation for 100
h as shown in figure 8(a), the oxide layer could be divided into two obvious regions: the top layer and the subsurface layer. The top layer presented a relatively high concentration of Ni and a relatively low concentration of Al. There was a continuous Al-rich band formed in the subsurface layer, while Ni was present in relatively small quantities in this band (figures 8(b) and (c)). A certain amount of Zr was uniformly distributed throughout the scale (figure 8(d)). Combined with XRD spectrums and EDS analysis results, the main phase of the top oxide layer with a thickness of about 200 μm was NiO, which seemed to be compacted. However, the predominant phase of the subsurface oxide layer with a thickness of about 100 μm was Al2O3. When the nickel aluminum alloy was kept in an oxygen containing environment at high temperature, the following reactions occurred:

\[
\begin{align*}
2\text{Ni} + \text{O}_2 (g) & = 2\text{NiO} (s) \\
2\text{Al} + 3\text{O}_2 (g) & = \text{Al}_2\text{O}_3 (s) \\
\text{Ni} + 2\text{Al} + 2\text{O}_2 (g) & = \text{NiAl}_2\text{O}_4 (s)
\end{align*}
\]

The underlined elements in the above equations represented elements presented in the coating. During thermal oxidation, the formation of NiO, Al2O3 and NiAl2O4 obviously competed with one another. At high temperature, the Al in the coating diffused outward to form Al2O3 and NiAl2O4, while the mutual diffusion of Ni and Al occurred on the inner side, which led to the continuous reduction of Al content in the coating and probably caused the degradation of coating performance. Ismail and Yaacob [14] also found the existence of NiO, Al2O3, and NiAl2O4 when studying the thermal oxidation of Ni3Al/ZrO2 nanocomposite in 1% SO2/air gas mixture at 800 °C and 1000 °C. Because the thermodynamic solubility of NiAl2O4 spinel in molten salt was less than NiO, NiAl2O4 spinel might possessed better thermal oxidation resistance than NiO. In addition, it could be seen from figures 8(c) and (e) that almost no oxygen was detected below Al2O3 layer. The oxide layer formed on the coating surface prevented the further diffusion of oxygen, so that the formation and growth of oxides occurred only in a small range under the coating surface (about 300 μm depth), and consequently there was a moderate increase in oxidation weight gain.

In the process of high temperature thermal oxidation, ZrO2/Ni3Al-Ni3Al double-layer coating maintained good adhesion to the stainless steel substrate and showed approximately parabolic oxidation behavior. The oxide layer formed on the coating surface remained intact without serious fracture and spalling, which greatly improved thermal oxidation resistance of the sample.

### 3.5. Hot corrosion behavior

Figure 9 shows the kinetic curve of ZrO2/Ni3Al-Ni3Al double-layer coating hot corroded in Na2SO4 + 25% K2SO4 mixed salt at 1050 °C. To evaluate the spalling degree of corrosion products, the exfoliated oxides were not included in the corrosion weight gain. When the holding time was in the range of 10 to 70 h, because of a lot

Figure 8. X-ray mappings for the cross section of ZrO2/Ni3Al-Ni3Al double-layer coating oxidized at 1050 °C after 100 h.
of exfoliated oxides were removed along with the residual salt after corrosion, the corrosion weight gain was negative, and changed slightly with the increase of holding time. Once the holding time increased above 70 h, less exfoliated oxides were observed in the residual salt after corrosion, therefore, the corrosion weight gain began to increase obviously. Even so, as the holding time increased to 100 h, the corrosion weight gain kept a very small value, only about 1 mg cm$^{-2}$, which was much less than that of thermal oxidation in atmospheric environment.

XRD patterns for ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating hot corroded at 1050°C during different times are given in figure 10. It was noticed that the coating surface appeared obvious oxidation after hot corrosion for 10 h, generating the mixed oxides which contained main phase of NiO and minor phases of NiAl$_2$O$_4$ and Al$_2$O$_3$. With the increasing of hot corrosion time, the diffraction peak of NiO was further enhanced. The XRD patterns for hot corrosion samples were similar to those of thermal oxidation samples, indicating that the hot corrosion products were similar to the thermal oxidation products.

Figure 11 illustrates SEM morphology and EDS analysis for ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating hot corroded at 1050°C during different times. According to figure 11(a), visible oxidation of the coating occurred after hot corroded in Na$_2$SO$_4$ + 25%K$_2$SO$_4$ mixed salt for 10 h. Based on XRD analysis results, a relatively continuous oxide layer, composed of NiO, NiAl$_2$O$_4$ and Al$_2$O$_3$ oxides, was formed on the coating surface. The oxide layer was rough and not as smooth as that formed by thermal oxidation in atmospheric environment.

![Figure 9](image1.png)

**Figure 9.** The kinetic curve of ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating hot corroded at 1050°C.

![Figure 10](image2.png)

**Figure 10.** XRD patterns for ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating hot corroded at 1050°C during different times.
After 30 h of hot corrosion, the oxide layer cracked, showing alternating traces of growth and peeling (figure 11(b)). After corrosion for 70 h, the oxide layer was irregular polyhedron or pyramid embedded on the surface of the coating (figure 11(c)). EDS analysis showed that the corrosion products were mixed oxides formed by Ni, Zr, Al and O, in which the content of Ni and O was as high as 91.90%, which corresponded to the very strong NiO diffraction peak in XRD (figure 11(e)). When corroded for 100 h, hot corrosion cracks occurred in the oxide layer (figure 11(d)).

When the hot corrosion time was in the range of 10 ~ 70 h, the peeling rate of oxide layer was greater than the growth rate, so the corrosion weight gain was negative. When the corrosion time was more than 70 h, the growth rate of oxide layer began to exceed than the peeling rate, thus the corrosion weight gain increased gradually. Because the growth and peeling of oxide layer during thermal corrosion was a dynamic change process, the weight gain of hot corrosion in Na2SO4 + 25% K2SO4 mixed salt was significantly less than that of thermal oxidation in atmospheric environment for the same holding time.
X-ray mappings for ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating hot corroded at 1050°C after 100 h are illustrated in figure 12. According to the cross sectional morphology of the coating after hot corrosion for 100 h as shown in figure 12(a), the oxide layer could be divided into two obvious regions: the top layer with high Ni content (thickness of about 200 μm) and the subsurface layer with high Al content (thickness of about 100 μm). Compared with the top layer, the Ni content in the subsurface layer was relatively less, while the Zr content was relatively more (figures 12(b) and (d)). Zr element was detected all over the scale due to the existence of ZrO$_2$ in the coating. Traces of S were faintly visible throughout the scale, which indicated that trace sulfate infiltrated into the sample through the cracks of corrosion products (figure 12(f)).

Combined with XRD spectrums and EDS analysis results, the main phase of the top layer (gray white) was NiO, however, in the subsurface layer (black), the amount of NiO decreased and Al$_2$O$_3$ greatly increased, which was similar to the oxide layer after thermal oxidation. In addition to NiO, NiAl$_2$O$_4$ and Al$_2$O$_3$ phases, trace sulfide could be deduced to exist in the oxide layer, but the X-ray with a lower detection limit of about 5% failed to detect it. Furthermore, almost no O was detected below Al$_2$O$_3$ layer as shown in figures 12(c) and (e), which indicated that the oxide layer formed on coating surface effectively acted as a diffusion barrier for corrosive substances and oxygen, and thus exhibited a good protective effect from corrosion.

ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating was successfully maintained the firm bonding with stainless steel substrate in hot corrosion process. Besides, from the X-ray mapping, it was evident that oxidation was limited to the range of about 300 μm under the coating surface and there was almost no vulcanization occurred inside the coating, further confirming the protective effect of the coating.

4. Conclusions

(1) The microstructure of the coating was Ni$_3$Al matrix with dispersed ZrO$_2$ particles, which was pure and compacted. The Ni$_3$Al intermediate layer compactly formed between stainless steel matrix and ZrO$_2$/Ni$_3$Al composite coating, with good metallurgical bonding interfaces.

(2) The outmost microhardness of ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating was similar to that of ZrO$_2$/Ni$_3$Al single-layer coating, which was about 500 HV, and presented a flat gradient variation from the coating to the substrate.

(3) When thermal oxidized at 1050 ºC, ZrO$_2$/Ni$_3$Al-Ni$_3$Al double-layer coating showed approximately parabolic oxidation behavior. A mixed oxide layer of NiO, Al$_2$O$_3$, and NiAl$_2$O$_4$ formed on the coating.
surface and remained intact without serious fracture and spalling, which greatly improved the thermal oxidation resistance of the sample.

(4) When hot corroded in the molten salt (Na$_2$SO$_4$ + 25% K$_2$SO$_4$) at 1050 °C, the hot corrosion oxide layer which shared a similar composition with thermal oxide layer, though grew and spalled alternately, effectively acted as a diffusion barrier for corrosive substances and oxygen, and thus exhibited a good protective effect from corrosion.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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