High Temperature Oxidation Behavior of Nano-Alumina–Modified NiAl Coating

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In this article, the nickel aluminate coating prepared by the chemical vapor deposition method has adhered deliberately with nano-alumina film on the surface by the electrophoresis method. The analysis results of oxidation behavior in the air at 1,000°C reported that the nano-alumina particles adhering to the nickel aluminate coating surface appear to be effective in facilitating the θ-Al2O3 to α-Al2O3 phase transformation. The fast θ to α phase transformation helps reduce the oxidation rate of the nickel–aluminum coating and prevents the cracking and peeling in the oxide scale. The research addressed a novel method to improve the high-temperature oxidation resistance of nickel aluminate coatings.

Keywords: Nickel aluminate coating, alumina modified, high temperature, oxidation, phase transition

INTRODUCTION

Nickel aluminate coatings, such as NiAl and Ni2Al3, are generally applied to the surfaces of engine turbine components to lengthen their service life (Goward, 1970; Goward and Boone, 1971; Leng et al., 2020). The common preparation methods of nickel aluminate coatings include slurry, powder embedding, physical vapor deposition, and chemical vapor deposition (CVD) (Nicholls, 2003; Bouchaud et al., 2013; Mollard et al., 2013; Montero et al., 2013). Compared with the other methods, the coating prepared by the CVD method has a stronger bonding force with the substrate and is denser, so its high-temperature oxidation resistance is better (Warnes and Punola, 1997; Yavorska et al., 2011; Goral et al., 2020).

The surface of the nickel aluminate coating will form protective thermally grown oxide (TGO)-Al2O3 at high temperatures (Swadba et al., 2011). Several types of Al2O3 have been found on alloys forming aluminiides, such as γ-Al2O3, δ-Al2O3, θ-Al2O3, and α-Al2O3. α-Al2O3 is a good protective oxide scale because of its thermodynamic stability and slow growth rate (An et al., 2000). During the oxidation at a temperature above 1,100°C, the nickel aluminate coating quickly forms the thermodynamically stable α-Al2O3 scale, while the metastable alumina variants scales (normally γ and θ) are formed after a oxidation for a long time (Khana et al., 2019) at a temperature below 1,100°C, during which they always experience the phase transformation of metastable-to-stable alumina (Peng et al., 2003; Huang and Peng, 2016), such as θ-to-α. This phase transformation accompanies a volume shrinkage of about 10%, sometimes resulting in the generation of micro-cracks in the alumina scale (Peng et al., 2003; Veal et al., 2006; Wang and Zhou, 2012; Huang and Peng, 2016).

As a result, prompting the conversion of metastable-to-stable alumina is advantageous for improving the oxidation resistance of nickel aluminate coatings at high temperatures (Khana et al., 2019). Moreover there is a well-known fact that a rising temperature causes a faster phase transformation of the metastable-to-stable alumina, dopants or template effects have been found
to change this process (Tolpygo and Clarke, 2000; Huang et al., 2019). For instance, Pint and his co-workers (Pint et al., 1997; Huang et al., 2019) found that Y2O3, La2O3, ZrO2, and HfO2 dispersions in β-NiAl tend to retard the θ-to-α phase transformation, whereas TiO2 dispersion appears to promote this process. Lately, Peng and his co-workers (Xu et al., 2010; Huang and Peng, 2016; Khana et al., 2019) reported the promoted formation of an α-Al2O3 scale on nickel aluminate surface. On these bases, we have a concept that spreading oxide particles (e.g., Cr2O3) with the same crystallographic structure as α-Al2O3 to aluminate the growth of an α-Al2O3 scale at a high temperature (Yin and Zhen, 2015). Valet et al. (Liu et al., 2007; Huang and Peng, 2016; Khana et al., 2019) found that the remaining fine α-Al2O3 grists, used for the polishing surface of the intermetallic sample, appeared to promote the nucleation of α-Al2O3.

In this article, nano-alumina particles were deposited on the surface of aluminate coatings by electrophoresis, and their effects on the phase transition and oxidation kinetics of the nickel aluminate coatings were investigated. It is aimed to better understand the phase transitions of the oxidative properties of nickel aluminides, and find a more effective way to improve the oxidation resistance of these nickel aluminate oxides by promoting the growth of α-Al2O3.

THE EXPERIMENT

The pure nickel samples with dimensions of 10 mm × 10 mm × 2 mm were cut from an electrolyte pure nickel plate. After being abraded to a final 2,000 grit SiC paper, they were ultrasonically cleaned in acetone. By using the CVD method, the Ni2Al3 coating was developed on the pure nickel surface after alumining at 750°C × 200 torr for 3 h. The preparation process is as follows: high-purity Cl2 was injected into the reaction chamber containing high-purity aluminum particles (purity: 99.99%, size: 3 mm) to generate gas-phase halide AlClg. AlClg was carried into the deposition chamber by Ar, and then was reduced to activity [Al] by H2. The [Al] deposited on the pure nickel, and reacted with nickel to form a nickel aluminum coating. After alumining for 3 h, the samples were cooled to room temperature with the furnace. The main equations involved in the reaction are as follows:

\[2Al + Cl2(g) \rightarrow 2AlCl(g)\]  (1)
\[Al + Cl2(g) \rightarrow AlCl(g)\]  (2)
\[2Al + 3Cl2(g) \rightarrow 2AlCl3(g)\]  (3)
\[AlCl(g) + AlCl2(g) + AlCl3(g) + 3H2(g) \rightarrow 3[Al] + 6HCl(g)\]  (4)

The nano-Al2O3 film was made to adhere to the nickel aluminum coating surface by electrophoresis technology. High temperature oxidation experiments were performed using the SETSYS evolution thermo-gravimetric analyzer (for in situ recording oxidation curves) in the air at 1,000°C for 50 h. A nickel layer was coated on the oxide scale surface using electroplating in the acidic electroless nickel plating mixed solution (containing 25 g/L C6H5O4, 6.25 g/L Na2CO3, 43 g/L NiSO4·6H2O, and 25 g/L NaH2PO4·H2O) at 98°C for 5 h. Scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD) were used to investigate the surface and cross-section morphologies, structure, and the phases of various nickel aluminide coatings.

RESULTS AND DISCUSSION

The cross-sectional morphology (Figure 1A) shows that a single layer of nickel aluminate coating (~30 μm thickness) was deposited on pure nickel after alumining. The coating was dense and well adherent to the pure Ni substrate. The Ni content and Al content of the coating were 39.3 at.% and 58.9 at.%, respectively. The XRD result (Figure 1C) characterized them as deposited coating as a β-Ni2Al3 phase. Backscattered SEM images of the polished cross-section of a nano-Al2O3 film sample are shown in Figure 1B. The as-electrophoresis Al2O3 film was continuous with a thickness of around 3 μm. However, the Al2O3 film was partially detached from the coating due to grinding and polishing during the preparation process.

Figure 2 shows the dependence on the time of mass gain and the square of mass gain of naked and Al2O3 film-covered Ni2Al3 coatings oxidated at 1,000°C for 50 h. The mass gain of Al2O3 film-covered Ni2Al3 coating is less than that of the naked one as shown in Figure 2A. In the early stages of oxidation, the surface of the two types of coatings quickly formed an aluminum oxide scale, and the oxidation weight gain rate was high. Due to the presence of alumina, the contact surface between oxygen and the coating gradually decreased, and the alumina scale gradually stabilized, so the oxidation weight gain curve tended to be flat. As shown in Figure 2B, during the 50-h oxidation, the oxidation processes of both coatings can be divided into two stages: In the initial stage (<3 h), the parabolic oxidation rate constant (KpI) of the Al2O3 film-covered Ni2Al3 coating is \(3.2 \times 10^{-13} \text{ g}^2/\text{cm}^4 \text{s}\) lower than that of the naked coating with parabolic oxidation rate constant Kp of \(~4.4 \times 10^{-13} \text{ g}^2/\text{cm}^4 \text{s}\). In the steady stage (>3 h), the parabolic oxidation rate constant (KpII) of the Al2O3 film-covered Ni2Al3 coating is \(1.2 \times 10^{-13} \text{ g}^2/\text{cm}^4 \text{s}\), also lower than that of the naked coating with parabolic oxidation rate constant KpII of \(~2.4 \times 10^{-13} \text{ g}^2/\text{cm}^4 \text{s}\).
alumina. According to the above morphologies and the characteristics of the alumina phase structure (Swadba et al., 2011), it can be judged that $\theta$-$\text{Al}_2\text{O}_3$ is rapidly formed in the early stage of oxidation, and then the $\theta$ phase is partially converted into the $\alpha$ phase. The growth of $\alpha$-$\text{Al}_2\text{O}_3$ will inhibit the growth of the $\theta$ phase. The lateral growth direction of $\alpha$-$\text{Al}_2\text{O}_3$ resulted in surface unevenness. The conversion of $\theta$ phase to $\alpha$ phase will cause the microcracks or even local spalling because of volume shrinkage (the transformation from metastable alumina to $\alpha$-$\text{Al}_2\text{O}_3$ is a reconstructive phase transition, which is a first-order phase transition and the process is irreversible). The spalling area will quickly generate $\theta$-$\text{Al}_2\text{O}_3$, so both $\theta$-$\text{Al}_2\text{O}_3$ and $\alpha$-$\text{Al}_2\text{O}_3$ phases can be seen on the surface after high-temperature oxidation for 50 h. For the $\text{Al}_2\text{O}_3$ film–covered Ni$_2$Al$_3$ coating, an $\alpha$-$\text{Al}_2\text{O}_3$ scale was formed rapidly in the early stage of oxidation. With the prolongation of oxidation time, the oxide scale gradually thickens and becomes flat (Figures 4B,D,F,H). At 5 h of oxidation, the scale appeared to slightly peel. After 50 h of oxidation, a dense and stable alumina scale has been formed. Therefore, there is no phase change in the oxide scale of the $\text{Al}_2\text{O}_3$ film–covered coating during the whole oxidation process. It can be seen in Figure 5A that the coating degradation completely conforms to the mechanism of the nickel–aluminum coating: $\delta$-$\text{Ni}_3\text{Al}_2$→$\beta$-$\text{NiAl}$→$\gamma'$-$\text{Ni}_3\text{Al}$. At the beginning of oxidation, the generated alumina is too thin and its X-ray diffraction peak intensity is weak, so the original diffraction peak pattern is partially enlarged as shown in Figures 5B',C'. From it is known that the $\alpha$ phase is formed on the surface of the electrophoretic alumina film sample from the beginning of oxidation. Based on the XRD, SEM, and TGA analysis, the early oxidation progress of aluminide in the absence and presence of the surface $\text{Al}_2\text{O}_3$ film is shown in Figure 6. At the beginning of oxidation (Stage I), $\theta$-$\text{Al}_2\text{O}_3$ is rapidly formed on the naked aluminide surface, while $\alpha$-$\text{Al}_2\text{O}_3$ is formed on the surface of the aluminide coating with electrophoretic $\text{Al}_2\text{O}_3$ particles (An et al., 2000). Then both increase to form an alumina scale (Stage II); the alumina is much denser as shown in Figures 4C,D. In stage III, generated $\alpha$-$\text{Al}_2\text{O}_3$ grows inward and outward at the same time, during which the most electrophoresed $\text{Al}_2\text{O}_3$ film on the surface is pushed outward, and a small part is wrapped by the outwardly growing $\alpha$-$\text{Al}_2\text{O}_3$ (Huang and Peng, 2016). At this time, the exposed $\theta$-$\text{Al}_2\text{O}_3$ of the naked sample transforms into $\alpha$-$\text{Al}_2\text{O}_3$ (Figure 4E). With $\alpha$-$\text{Al}_2\text{O}_3$ gradually increasing, an $\alpha$-$\text{Al}_2\text{O}_3$ scale was formed at the interface between NiAl coating and $\theta$-$\text{Al}_2\text{O}_3$ (Stage IV). During this phase transition, volume shrinkage will occur, resulting in cracks or even peeling of the scale (Tolpygo and Clarke, 2000), which are visible on the surface topography (Figure 4).

The nickel aluminide thermally grows $\theta$-$\text{Al}_2\text{O}_3$ before $\alpha$-$\text{Al}_2\text{O}_3$ because the former has lower surface energies and thus a much
lower nucleation barrier $\Delta G^*$. This is the direct origin of why the metastable alumina phases rather than the most stable ones usually form during the initial oxidation of $\beta$-NiAl (Huang et al., 2019). Therefore, during the subsequent oxidation, the undesirable metastable-to-stable phase transformation of alumina becomes unavoidable. The direct $\alpha$-$\text{Al}_2\text{O}_3$ formation without metastable-to-stable alumina phase transformation on typical oxide particles (e.g., $\alpha$-$\text{Al}_2\text{O}_3$, $\text{Cr}_2\text{O}_3$, and $\text{Fe}_2\text{O}_3$) means that the template effect of the oxides isostructural to $\alpha$-$\text{Al}_2\text{O}_3$ intrinsically reduces $\Delta G^*$ of $\alpha$-$\text{Al}_2\text{O}_3$ down to such a level in favor of its thermal nucleation (Pint et al., 1997; Wang and Zhou, 2012; Yin and Zhen, 2015; Huang et al., 2019). The template effect has been widely studied by revealing some oxides (e.g., $\text{Cr}_2\text{O}_3$ and $\text{Fe}_2\text{O}_3$), which are isostructural to $\alpha$-$\text{Al}_2\text{O}_3$, and may serve as the nucleation sites facilitating the growth of the $\alpha$-$\text{Al}_2\text{O}_3$ (Pint et al., 1997; Wang and Zhou, 2012; Yin and Zhen, 2015; Huang et al., 2019). This interpretation coincides well with a promoted $\theta$-to-$\alpha$ alumina phase transformation on $\beta$-NiAl when it has been embedded with the fine particles of $\alpha$-$\text{Al}_2\text{O}_3$ (Veal et al., 2006). The electrophoretic $\alpha$-$\text{Al}_2\text{O}_3$ particles have the same HCP crystal structure as the $\alpha$-$\text{Al}_2\text{O}_3$ grown from the coating and can act as an epitaxial growth platform for $\alpha$-$\text{Al}_2\text{O}_3$ during oxidation, reducing the nucleation energy of the latter. Therefore, when the surface is completely covered with alumina nanoparticles, the $\theta$-to-$\alpha$ phase transition can be skipped when the coating is oxidized at 1,000°C, and $\alpha$-$\text{Al}_2\text{O}_3$ is directly generated.

The $\text{Al}_2\text{O}_3$ particle pretreatment significantly decreases the oxidation rate, leading to a slower degradation rate of the nickel aluminide. As seen in the cross-sectional characteristics in Figure 7, after 50 h of oxidation, the phase structures of both types of coatings are NiAl + Ni$_3$Al phases, indicating that the unstable $\delta$-Ni$_2$Al$_3$ phase degenerates into the NiAl phase at a high temperature of 1,000°C, and then unceasingly degenerates into the Ni$_3$Al phase with oxidation time prolongation. In Figure 7, the thickness of the naked coating and the $\text{Al}_2\text{O}_3$ film-covered
coating after oxidation is thicker than that before oxidation, indicating that both coatings have a certain degree of Al elements internal-diffusion during the high-temperature oxidation process. It can be seen from Figures 7A,B that the alumina scale thickness of the naked coating is about 7.9 µm, which is twice that of the Al2O3-scale-covered coating (~3.5 µm).

**FIGURE 4** | Surface morphologies of the alumina scale formed on the δ-Ni2Al3 coating at 1,000°C for different oxidation times. (A), (C), (E), and (G): Ni2Al3; (B), (D), (F), and (H): Al2O3 film-covered Ni2Al3.
Figure 5: XRD patterns of (A) Ni₃Al coating at different times and (B) and (C) 30 min and 50 h naked and the Al₂O₃ film-covered Ni₃Al coating after oxidation in the air at 1,000°C and (B’) (C’) locally magnified.
This is consistent with the analysis result of the oxidation growth curve. During the oxidation process, aluminum ions moving from the inside of the coating to the coating surface meet with oxygen atoms to form an aluminum oxide scale, so there will be a γ-Ni₃Al phase formed due to the Al rapid consumption between the aluminum oxide film and the nickel–aluminum coating. Since the naked coating needs to consume more Al elements to form the aluminum oxide scale, the thickness of the γ-Ni₃Al phase coating is significantly thicker than that of the Al₂O₃ film-covered coating, which is near the aluminum oxide scale. The reason for the difference in the thickness of the two coatings is that electrophoretic alumina can promote the formation of α-Al₂O₃ on the coating surface, which can effectively prevent the oxygen element from entering the coating, reducing the consumption of the Ni₂Al₃ coating. Therefore, promoting the formation of stable-phase α-Al₂O₃ is an effective way to improve the high-temperature oxidation resistance and prolong the life of the coating. The large difference in coating consumption in the same sample is due to the consumption of aluminum elements again to form an aluminum oxide scale after the local scale peels off. With the prolongation of oxidation time, the cracks increase and cause local peeling of the aluminum oxide scale. The spalled
area will quickly deplete the nickel–aluminum coating to form a new aluminum oxide scale.

CONCLUSION

The Ni$_2$Al$_3$ coating with a thickness of 35 μm was prepared on the pure Ni matrix using a chemical vapor deposition method at 750°C × 200 T for 3 h, and then the Al$_2$O$_3$ film was made to adhere to the Ni$_2$Al$_3$ coating surface by electrophoresis technology. Because of the lower power required to form the critical nuclei, the θ-Al$_2$O$_3$ formed earlier on the naked Ni$_2$Al$_3$ coating and then undergo a θ-to-α Al$_2$O$_3$ transition process during the oxidation process at 1,000°C. The electrophoretic Al$_2$O$_3$ film adhering to Ni$_2$Al$_3$ coating can skip this phase transition because the Al$_2$O$_3$ film on the coating surface can provide a crystal template to promote the nucleation of α-Al$_2$O$_3$ at the beginning of oxidation. The rapid nucleation of α-Al$_2$O$_3$ significantly reduces the oxidation rate of NiAl coating and prevents microcracks and voids caused by phase transformation.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/supplementary material; further inquiries can be directed to the corresponding author.

AUTHOR CONTRIBUTIONS

YF carried out experiments; XS directed the experiment and analyzed experimental results; SD analyzed experimental results.

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FIGURE 7 | Cross-sectional morphologies of the Al$_2$O$_3$ scales on the (A) and (B) naked Ni$_2$Al$_3$ coating, and (C) and (D) the Al$_2$O$_3$ film-covered Ni$_2$Al$_3$ after 50 h of oxidation in the air at 1,000°C.
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