VPLIV PREDHODNE OKSIDACIJE NA PREVLEKO Al–Si ODPORNO NA KOROZIJO V VRO^EM

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In this study, effects of pre-oxidation on the hot corrosion behaviour of Al–Si coatings in a Na2SO4 and 25 *w*/% NaCl mixture at 900 °C were investigated. The results showed that after 120 h of corrosion, a large amount of β -NiAl residual was found with no vented external Q^2 and species such as S^2 and Cl⁻ from the molten salts from directly contacting the coating and reduced the rate of TiO₂ formation in the oxide layer, thus forming an Al₂O₃-based oxide film and reducing the growth rate of the oxide film on the coating surface. Cracking and peeling of the oxide film, caused by the internal mal expansion coefficient between the oxides in the oxide layer, are suppressed; thus, the hot corrosion resistance of the coating is significantly improved.

Keywords: superalloy, Al–Si coating, pre-oxidation, hot corrosion

Namen raziskave je ocena vpliva predhodne oksidacije prevleke na osnovi Al–Si na njeno obnašanje med vročo oksidacijo v
raztaljeni mešanici soli Na₂SO₄ in 25 masnih % NaCl pri 900 °C. Rezultati raziskave so pokazali, prevleke Al–Si. Zaradi predhodne oksidacije prevleke je nastali oksidni film preprečil neposredni stik zunanjih ionov O^{2–} in tudi drugih, kot sta S^{2–} and Cl[–] v raztaljeni soli. Tako je prišlo tudi do zmanjšane hitrosti tvorbe TiO₂ v oksidni plasti. Prišlo je do tvorbe oksidnega filma na osnovi Al₂O₃ in zmanjšanja hitrosti rasti oksidnega filma na površini prevleke. Pokanje in lupljenje oksidnega filma zaradi notranjih napetosti so pripisali notranjim napetostim nastalim zaradi razlike v koeficientih termičnega raztezka med oksidi v oksidnem filmu. Avtorji so ugotovili, da je bila zaradi postopka pred oksidacije odpornost proti koroziji v vročem obravnavane prevleke Al-Si močno izboljšana.

Ključne besede: superzlitina, prevleka Al-Si, predhodna oksidacija, korozija v vročem

1 INTRODUCTION

DZ417G, a nickel-based superalloy, is mainly applied to turbine blades of aircraft engines, and its working temperature is below 980 °C. In a high-temperature environment, the fuel in the engine produces large amounts of products including S, Na, V, Cl and other elements during the combustion process. These products react with salts in the marine environment to generate the corresponding sulphate or chloride salts, and eutectics containing mixed salts may cause accelerated or destructive corrosion of materials, $\frac{1}{x}$ having a significant impact on the performance and service life of the engine blades. Hot corrosion is divided into two main categories: high-temperature hot corrosion (type I) at 850–1000 °C and low-temperature hot corrosion (type II) below 800 °C. The corrosion rate can be reduced by forming an Al_2O_3 or Cr_2O_3 oxide film on the substrate surface; however, the high-temperature hot corrosion resistance of the substrate is affected. Various studies have concluded that high-temperature protective coatings can effectively protect nickel-based superalloys from high-temperature hot

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corrosion for a long time. Among them, Al–Si coatings provide the advantages of a simple process, low cost and unique corrosion resistance for types I and II.²⁻⁴ After an Al–Si coating is subjected to hot corrosion, there is no internal vulcanisation phenomenon in the matrix, but the internal corrosion of the coating is a serious issue. However, studies have shown that pre-oxidation can reduce the internal vulcanisation rate in the coatings.

After a pre-oxidation treatment at a certain temperature and time, a protective oxide film is formed on the material surface, preventing corrosive substances from directly contacting the coating, thus slowing down the subsequent corrosion rate. Yang et al.⁵ studied the changes in the microstructures and properties of Pt–Al coating samples after the pre-oxidation treatment and found that a continuous and dense Al_2O_3 film formed on the coating surface after the treatment. After hot corrosion, the oxide film on the surface of the pre-oxidised Pt–Al coating was thinner and more complete, and the structure of the oxide film formed at the same time fluctuated greatly, indicating that the oxide grew toward the inner layer of the coating during the dissolution pro $cess^{6,7}$

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To date, studies on the influence of pre-oxidation on the hot corrosion resistance of Al–Si coatings only investigated the functions of pre-oxidation; however, no systematic study was conducted on the impacts of the pre-oxidation process parameters on the hot corrosion resistance. To investigate the effects of the pre-oxidation process, this study pre-oxidised Al–Si coatings at 950 °C for 7 h, and a hot corrosion experiment was performed to analyse the effects of pre-oxidation on the hot corrosion behaviour of Al–Si coatings and the corresponding mechanisms.

2 EXPERIMENTAL PART

Prior to the hot corrosion experiment, an Al–Si coating was pre-oxidised at 950 °C for 7 h, as shown in **Figure 1**. Two groups of control experiments were conducted with three parallel samples in each group. A prepared mixed-salt solution containing $Na₂SO₄$ and 25 *w*/% NaCl was applied on the sample surfaces with a brush to form a salt film, and each sample was dried until the salt with a load of approximately (1.0 ± 0.5) mg/cm² was deposited on the surface. The specimens were put into a crucible and subjected to a hot corrosion experiment in an SXL-1400C high-temperature furnace at 900 °C. The specimens were removed, weighed and recorded after (24, 48, 72, 96 and 120) h. After weighing, the specimens were returned to the furnace, and the above processes were repeated until the corrosion time reached 120 h. The samples obtained after 24 h and 120 h of hot corrosion were taken out as intermediate samples, and Origin was used to plot the corrosion kinetics curve based on the corrosion weighing data. A ZeissUltra55 scanning electron microscope (SEM) and SmartLab9KW X-ray diffractometer (XRD) were used to observe the surface corrosion morphologies and crystal structures, respectively, at the early corrosion stage (24 h) and after 120 h of corrosion. The elemental distribution in the cross-sections of the two sample groups was conducted using a field emission electron probe (JEOL-JXA-8530F).

Figure 1: Experimental process of hot corrosion of pre-oxidised Al–Si coatings

3 RESULTS

3.1 Hot corrosion products and microstructures after 24 h of corrosion

Figure 2 shows XRD patterns of the pre-oxidised and un-pre-oxidised Al–Si coatings after hot corrosion at 900 °C for 24 h. The main corrosion products of both groups were α -Al₂O₃ and β -NiAl, with a few low-intensity peaks corresponding to Cr_2O_3 . The corrosion products on the surface of the Al–Si coating without the pre-oxidation treatment contained strong γ' -Ni₃Al diffraction peaks and small amounts of CrS. However, the intensity of the α -Al₂O₃ diffraction peaks on the surface of the pre-oxidised Al–Si coating was relatively high, and only a few low-intensity peaks corresponding to γ '-Ni₃Al could be observed.

The surface and macroscopic morphologies of the pre-oxidised and un-pre-oxidised Al–Si coatings after corrosion in the Na2SO4 and 25 *w*/% NaCl mixture at 900 °C for 24 h are shown in **Figure 3**. The oxide films on the surfaces of both groups are relatively lamellar, with small granular oxides. The granular oxides are mainly composed of Al, O and Ti (from Al_2O_3 and TiO₂), with $TiO₂$ growing on $Al₂O₃$. On the other hand, the samples without the pre-oxidation treatment (**Figure 3b**) have more granular oxides on the surface. TiO₂ can accelerate the growth of oxide films, and the Ti in the oxide film promotes the transformation of metastable θ -Al₂O₃ to steady-state α -Al₂O₃, giving rise to a surface oxide film mainly composed of stable α -Al₂O₃ and its improved high-temperature oxidation resistance.8,9 However, this transformation increases the hot-compression stress, and thus the coating may flake or crack.¹⁰ The surface of the pre-oxidised Al–Si coating (**Figure 3b**) is relatively flat after 24 h of corrosion. The macroscopic morphologies after 24 h of corrosion in **Figures 3b** and **3c** show that the surfaces of the two sample groups are relatively intact. As displayed in **Figure 3c**, the surface of the coating without the pre-oxidation treatment shows

Figure 2: XRD patterns of pre-oxidised and un-pre-oxidised Al–Si coatings corroded in mixed salts at 900 °C for 24 h

50 Materiali in tehnologije / Materials and technology 57 (2023) 1, 49–56

Figure 3: Surface and macroscopic morphologies of pre-oxidised and un-pre-oxidised Al–Si coatings etched in the mixed salts of Na₂SO₄ and 25 *w/%* NaCl at 900 °C for 24 h: surface morphology for a) un-pre-oxidised coating, b) pre-oxidised coating; and macroscopic morphology for c) un-pre-oxidised coating, d) pre-oxidised coating

slight cracking, which is mainly ascribed to the uneven oxidation caused by stress relaxation during the growth of the oxide films or the uneven thickness of the oxide films caused by the growth of the new oxide.11 In **Figure 3d**, no corrosion trace or cracking on the surface of the pre-oxidised Al–Si coating can be observed.

Figure 4 shows cross-sectional morphologies of the two sample groups after 24 h of corrosion. A continuous oxide film, mainly composed of Al_2O_3 , is in the outermost layer of both coatings; however, significant morphological differences appear in the interdiffusion zone. The interdiffusion zone of the Al–Si coating without the pre-oxidation treatment is mainly composed of β -NiAl and a small amount of γ' -Ni₃Al. In addition, this interdiffusion zone is not continuous and has an obvious black-tissue formation. According to the elemental distribution in the section shown in **Figure 5**, the black tissue mainly contains the sulphide of Cr, namely, CrS. On the coating sample with the pre-oxidation treatment, the interdiffusion zone exhibits an island structure with more β -NiAl and less γ '-Ni₃Al and it is more continuous with no obvious vulcanization in the coating. More importantly, after 24 h of corrosion, the pre-oxidised coating formed a Si- and Cr-rich layer at the interface with the matrix. Part of the Cr formed M_6C by combining with C and other elements, while Si promoted the formation of M_6C . The Si-rich M_6C formed a precipitated band, which greatly reduced the Al diffusion in the coating in the substrate direction and reduced the Ni diffusion in the coating direction.12,13 In addition, the precipitated zone can

Figure 4: Cross-sectional morphologies of Al–Si coatings corroded in mixed salts at 900 °C for 24 h: a) un-pre-oxidised coating and b) pre-oxidised coating

Figure 5: Cross-sectional elemental distribution of Al–Si coatings corroded in mixed salts at 900 °C for 24 h: a) un-pre-oxidised coating and b) pre-oxidised coating

also easily combine with the refractory particles (Mo and V) in the matrix, ensuring that the mechanical properties of the matrix are not affected when the coated sample is oxidised at high temperatures. Si can also slow down the rate of the oxide dissolution in an alkaline solution during the molten salt corrosion and reduce the rate of hot corrosion.14–17

3.2 Hot corrosion products and microstructures after 120 h of corrosion

Figure 6 shows XRD patterns of the two sample groups after 120 h of hot corrosion. α -Al₂O₃ remained dominant on the surfaces of the two groups; however, γ '-Ni₃Al diffraction peaks with high intensities appeared in both groups, and some $TiO₂$ remained. The diffraction peak from $NiAl₂O₄$ with a relatively weak intensity was observed in the un-pre-oxidised Al–Si coating, indicating that the oxide films in the coating were partially degraded after 120 h of corrosion. Compared with the un-pre-oxidised coating, the pre-oxidised Al–Si coating presented more α -Al₂O₃ and β -NiAl with fewer γ '-Ni₃Al diffraction peaks. Generally, the appearance of γ' -Ni₃Al indicates that the coating is degraded; however, the presence of β -NiAl can hinder further degradation of the coating. A continuous Al_2O_3 film was maintained on the coating surface, and the oxide-film formation and spalling rates reached a dynamic balance, which was beneficial for improving the high-temperature corrosion resistance of the Al–Si coating.

Figure 7 shows microstructures and macroscopic morphologies of the two groups of Al–Si coatings after 120 h of corrosion. As shown in **Figure 7c**, the surface of the Al–Si coating without the pre-oxidation treatment exhibits an obvious peeling phenomenon after 120 h of corrosion, especially at the boundary. Moreover, for the

surface microstructure (**Figure 7a**), various round or block oxides can be observed after the corrosion, mainly containing TiO₂ by analysis. As displayed in **Figure 7c**, $TiO₂$ damages the oxide film on the Al–Si coating surface, which is because $TiO₂$ nucleates, grows on $Al₂O₃$ and aggregates on the sample surface, damaging the stability of the oxide films. At present, there are two theories regarding the effects of Ti on promoting the spalling and cracking of oxide films:18–20

- The rapid diffusion of Ti from the matrix to the coating surface leads to the formation of $TiO₂$ on the $Al₂O₃$ film and improves the growth rate of the oxide film.
- Ti promotes the formation of Ti-rich oxide bulges on the surface of metal or oxide films, complicating the stress distribution during the oxide-film growth and leading to the formation of cracks in the films.

Figure 6: XRD patterns of the pre-oxidised and un-pre-oxidised Al–Si coatings corroded in the mixed salts of Na2SO4 and 25 *w*/% NaCl at 900 °C for 120 h

52 Materiali in tehnologije / Materials and technology 57 (2023) 1, 49–56

Table 1: Average compositions of pre-oxidised and un-pre-oxidised Al–Si coatings after 120 h of corrosion in atomic % (*x*/%)

Figure 7: Al–Si coatings corroded in the mixed salts of Na₂SO₄ and 25 *w/%* NaCl at 900 °C for 120 h with surface morphologies for: a) un-pre-oxidised coating, b) pre-oxidised coating; and macroscopic morphology for c) un-pre-oxidised coating, d) pre-oxidised coating

However, the Ti content on the surface of the pre-oxidised Al–Si coating was lower (as shown in **Table 1**), and less $TiO₂$ was generated on the surface after hot corrosion. After 120 h of hot corrosion, the surface was intact with no obvious spalling or cracking; only the edges and corners exhibited slight corrosion spalling, which was mainly caused by the growth stress in the oxide film

and had little impact on the overall heat-resistance corrosion performance of the sample.

Figures 8 and **9** show the cross-sectional morphologies and elemental distribution for the two sample groups after 120 h of corrosion. As shown in **Figure 8a**, a gap with a saw-tooth shape under the oxide layer is observed on the surface of the Al–Si coating without the pre-oxidation treatment, and the elemental distribution

Figure 8: Cross-sectional morphologies of the Al–Si coatings corroded by the mixed salts of Na₂SO₄ and 25 *w*/% NaCl at 900 °C for 120 h: a) un-pre-oxidised coating and b) pre-oxidised coating

Materiali in tehnologije / Materials and technology 57 (2023) 1, 49–56 53

Figure 9: Elemental distribution of the cross-sections of the Al–Si coatings corroded by the mixed salts of Na₂SO₄ and 25 *w*/% NaCl at 900 °C for 120 h: a) un-pre-oxidised coating; and b) pre-oxidised coating

indicates that it is a Ti-rich diffusion layer. The black area mainly contains the sulphide of Cr, namely, CrS, indicating that the inner coating has been corroded by the molten salts, leading to coating degradation. As displayed in **Figure 8b**, no void band can be found on the surface of the pre-oxidised coating after 120 h of corrosion, and no vulcanisation inside the coating is observed. The overall coating is relatively intact. The outer layer of the coating is an Al-rich oxide layer, and the interdiffusion zone is composed of β -NiAl and a small amount of γ' -Ni₃Al, with Si- and Cr-rich diffusion bands at the interface between the coating and the matrix.

4 DISCUSSION

4.1 Corrosion kinetics analysis

The hot corrosion kinetic curves for the pre-oxidised and un-pre-oxidised Al–Si coatings in the $Na₂SO₄$ and 25 *w*/% NaCl mixture at 900 °C are shown in **Figure 10**. The trends of the mass gain in the two sample groups are almost identical. The corrosion mass gain for the samples with the pre-oxidation treatment is significantly lower than that for the samples without the treatment. In the first 48 h, the corrosion mass gain rate for the Al–Si coating without the pre-oxidation treatment is faster because a metastable θ -Al₂O₃ film is mainly formed on the coating surface, which has a weak hot corrosion resistance. After 48 h, the corrosion mass gain rate for the pre-oxidised Al–Si coating decreases and tends to be constant; however, the rate remains faster for the samples without pre-oxidation. The growth stress in the pre-oxidised samples increase owing to the damage by the $S²$ and Cl– from the molten salts at the early stage of corrosion, damaging the integrity of the oxide films and making them loose and porous.

4.2 Mechanistic analysis of hot corrosion at high temperatures

Owing to the combination of turbine gases (V, Cl and S) and elements (Na) in air, salt mixtures with low melting points are formed. The melting point of pure $Na₂SO₄$ is 884 \degree C, and NaCl can reduce the Na₂SO₄ melting point and finally form a eutectic system with a melting point^{21, 22} of 620 °C.^{21, 22} The experimental temperature is 900 °C, significantly promoting the oxidation process of $Na₂SO₄$ and NaCl.²³ In the early stage of hot corrosion, the main corrosion product on the outer surface of the coating is Al_2O_3 owing to the high Al content, high oxygen partial pressure at the initial stage and fast oxidation rates. The specific reaction is given by Equation (1):

$$
4/3 \text{ Al} + \text{O}_2 = 2/3 \text{ Al}_2\text{O}_3 \tag{1}
$$

In the molten state, the $Na₂SO₄$ in the mixed salts penetrates the Al_2O_3 film formed on the coating surface

Figure 10: Hot corrosion kinetic curves of un-pre-oxidised and pre-oxidised Al–Si coatings in mixed salts at 900 °C

54 Materiali in tehnologije / Materials and technology 57 (2023) 1, 49–56

and reacts, as shown in Equation (2). Because of the reaction between Al_2O_3 and Na_2SO_4 , the protective oxide film, Al_2O_3 , is continuously dissolved during the hot corrosion process, leading to cracking and peeling of the oxide film.24

$$
Na_2SO_4 + Al_2O_3 = 2NaAlO_2 + SO_3
$$
 (2)

As the surface Al_2O_3 film is damaged by the molten salt, the β -NiAl in the coating, as a source storing Al, provides Al, which repairs the cracked or peeled off Al_2O_3 film by spreading to the outer layer of the coating, ensuring a relatively continuous and complete protective oxide film on the coating surface. With continuous corrosion, the oxides containing Al on the surface are dissolved and, hence, the Al element is constantly consumed, reducing the heat and corrosion resistance of the matrix and coating, thus promoting the oxidation and vulcanisation of the samples. Therefore, according to the above morphological characterisation and elemental distribution analyses, the coating samples without the pre-oxidation treatment exhibit internal oxidation and vulcanisation, which is mainly due to the fact that with an increase in the corrosion time, alloying elements in the matrix diffuse into the coating and interact with Na2SO4, resulting in the following reactions:

$$
Na_2SO_4 + 8/3 Al = Na_2O + Al_2O_3 + Al_2S_3
$$
 (3)

$$
Na_2SO_4 + 3Cr = Na_2O + Cr_2O_3 + CrS_3 \tag{4}
$$

4.3 Mechanism of pre-oxidation effects on hot corrosion resistance of Al–Si coatings

According to Vialas and Monceau,²⁰ the Bedworth ratios (PBRs) of Al_2O_3 and TiO₂ growing on β -NiAl are different. The PBR of Al_2O_3 on NiAl is 1.8, whereas that of $TiO₂$ on NiAl is 2.6. This significant difference causes the formation of $TiO₂$ on the oxide film, which can easily induce a large local compressive stress, leading to the tendency toward cracking and peeling of the coating.19 However, pre-oxidation can enhance the anti-corrosion properties of the Al–Si coating at high temperatures. After the pre-oxidation treatment, a relatively stable oxide film containing α -Al₂O₃ is formed on the coating surface. In addition, as provided in **Table 1**, the content of Ti on the coating surface is low. Therefore, the pre-oxidation treatment reduces the impacts of Ti on the oxide films on coating surfaces. Moreover, because the formation of the continuous α -Al₂O₃ on coating surfaces takes a certain time, $S²$ and Cl⁻ from the molten salts and O^{2–} from air enter the interior of the coating without pre-oxidation through diffusion during hot corrosion, leading to a reduction of the hot corrosion resistance of the Al–Si coating. In contrast, a continuous oxide film is formed on the coating surface due to the pre-oxidation treatment before hot corrosion, which prevents a direct contact of the coating with the external O^{2-} and the elements, such as S^{2–} and Cl[–] from the molten salts, and reduces its hot corrosion rate.

5 CONCLUSIONS

During hot corrosion in mixed salts at 900 °C, the corrosion weight of the pre-oxidised Al–Si coating was significantly reduced, and the corrosion rate was much slower than that of the un-pre-oxidised Al–Si coating.

After 24 h of hot corrosion, α -Al₂O₃ formed on the surfaces of both coating groups. However, the pre-oxidised Al–Si coating was more intact and compact after corrosion and contained more β -NiAl, ensuring the continuous formation of the Al_2O_3 film on the coating surface.

After 120 h of hot corrosion, large amounts of $TiO₂$ were generated on the un-pre-oxidised Al–Si coating surface, with obvious peeling at the boundaries; CrS and other compounds were found in the coating. In contrast, the surface of the pre-oxidised Al–Si coating remained intact and was mainly composed of α -Al₂O₃ with no obvious sulphides in the coating.

The oxide film generated by the pre-oxidation treatment can prevent $S²$ and $Cl⁻$ from the molten salts and O2– from air from directly contacting the coating surface, reduce the rate of the $TiO₂$ formation in the oxide layer, and form an oxide film dominated by Al_2O_3 , significantly improving the hot corrosion resistance of the coating.

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