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Preparation and enhanced oxidation performance of a Hf-doped single-phase Pt-modified aluminide coating

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ABSTRACT

A Hf-doped β -(Ni,Pt)Al coating was prepared by co-deposition of a Pt-Hf composite plating and successive aluminisation. Then, a distinct Hf-rich belt was formed internally between the outer additive (Ni,Pt)Al coating and interdiffusion zone. An isothermal oxidation test at 1100 °C revealed a relatively lower oxidation rate constant and decreased oxide scale rumpling tendency for the Hf-doped coating during which the Hf-rich belt partly acted as an effective diffusion barrier. The unique addition of Hf into a β -(Ni,Pt)Al coating can delay the transitional oxidation period from transient alumina to stable one and postpone the degradation from β to γ .

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1. Introduction

For modern gas-turbine engines, the Turbine Entry Temperature (TET) has been increased continuously to meet the strategy of both increasing the power-output efficiency and reducing greenhouse emissions (e.g., CO₂, SO₂ and NO_x etc.). Coupled to a sophisticated cooling system, the development of advanced thermal barrier coating systems (TBCs) could offer a temperature reduction of more than 150 °C at the superalloy blade surface, ensuring that the gas-turbine engine operates above the melting-point of a nickel-based substrate alloy [1]. In the case of aircraft engines, the most widely utilised diffusion coatings are based on Pt-modified NiAl [2], for which the resistances against high temperature oxidation as well as hot corrosion have been greatly enhanced by the addition of platinum [3–6]. The beneficial merits of using Pt in aluminide coatings mainly consist of promoting the formation and adhesion of α -Al₂O₃ scale, mitigating the deleterious effects of S, reducing the activity of Al, among others [7–10].

To further enhance the scale adhesion and eliminate underlying voids, reactive elements (RE), such as Ce, Hf and Y, were often added to NiAl series bond coats to produce a number of favourable effects

for improving the oxidation performance. It is widely accepted that Hf possesses relatively high chemical reactivity, which assists in gathering sulphur and segregating below the thermally grown oxide (TGO), which prevents sulphur segregation [11]; forming oxide protrusions in the substrate that obstruct crack propagation [12]; and most importantly, decreasing the oxide growth rate by reducing Al diffusivity in Al₂O₃ [13]. It has been revealed that Hf-doped cast alloys of β -NiAl and Ni-Pt aluminides could decrease the parabolic rate constant by a factor of ten [14]; Hf was believed to be more efficient than Pt in reducing the oxidation rate [15].

Due to the attractive features of Hf addition, many research trials have been reported to incorporate this promising element into Ni-based aluminide systems via cast-alloying [15–17], ion plasma deposition [18–20], electron beam physical vapour deposition (EB-PVD) [21,22], laser cladding [23], direct current (DC) magnetron sputtering [24], pack-cementation [2,25] and vapour phase co-deposition with Al [26–29]. Thus, until now, either a PVD or CVD facility was required to load Hf into a practical NiAl based bond coat. However, the aforementioned PVD approaches demand costly vacuum systems and are inapplicable to deposit multifaceted components. Precise alloying and incomplete target utilisation may increase the additive costs for PVD techniques. In a CVD process, it is quite difficult to control the Hf level or make it evenly distributed within the coating interior [26,30]. A typical outcome is that Hf-rich particles are often segregated along grain boundaries [31], which is

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Table 1
Bath composition and electroplating parameters for depositing Pt and Pt-Hf coatings.

Compositions/Parameters	Pt coating	Pt-Hf composite coating
K ₂ [Pt(NO ₂) ₂ SO ₄]	23.64 g L ⁻¹	23.64 g L ⁻¹
Hf particles (0.1–10 μm)	/	8–10 g L ⁻¹
Magnetic stirring	/	15 r min ⁻¹
pH value	1–2	1.5
Current density	1 A dm ⁻²	0.8 A dm ⁻²
Temperature	65 °C	55 °C
Deposition rate	2 μm h ⁻¹	1.5 μm h ⁻¹

believed to be deleterious for the integrity of the posterior alumina scale formed on the surface.

In addition, it is of great importance to control the amount of Hf incorporated in the Ni-based aluminide coating. Practically, the content of Hf needs to be regulated at an optimal range to exhibit the reactive element effect (REE) [32]. If the Hf content is too low, little or no benefit will be observed, whereas overly high Hf levels will accelerate the scale growth by internal oxidation, forming large HfO₂ pegs, which further rapidly transport oxygen [33].

Unlike the aforementioned PVD and CVD methods, in the current work, the incorporation of Hf was achieved by the co-deposition of Pt using a simple electroplating approach. This is a cost-saving method and is more adaptable to parts with complex shapes for industrial-scale production. With sequential treatments of vacuum annealing and aluminisation, a distinct structure of an outer (Ni,Pt)Al coating plus an internal Hf-rich belt was obtained. The Hf-doped (Ni,Pt)Al coating was confirmed to be more advantageous than the normal (Ni,Pt)Al in the isothermal oxidation test at 1100 °C in several respects: a lower oxidation rate constant k_p , lower tendency of scale rumpling, decreased coating degradation rate from β to γ' , and partly serving as a diffusion barrier. These promising effects, including the oxidation behaviour in the early stage, are discussed in comparison with a Hf-free (Ni,Pt)Al coating.

2. Experimental

A nickel-based single crystal (SC) superalloy with a composition of Ni-12Cr-9Co-3.7W-1.9Mo-5Ta-3.6Al-4Ti (minor C, in wt.%) was used as the substrate material. Disc-shaped specimens of 2 mm in thickness and 15 mm in diameter were sliced from [001]-orientated crystal rods using a spark-discharging machine. The bare samples were ground with a final 400# SiC paper finish and then humidly blasted with 300 mesh alumina grit under an air-pressure of 0.3 MPa. The sample surface was degreased by boiling in a NaOH aqueous solution of 50 g L⁻¹ for 10 min, followed by ultrasonic cleaning in acetone and ethanol for 15 min. After that, SC samples with fresh surfaces were ready for electroplating with the Pt-Hf composite coating and conventional Pt coating.

Hafnium particles of a 99.5 wt.% purity (500 mesh, commercially available) were loaded into an agate container filled with agate balls to perform wet grinding for about 1 week using deionised water by a planetary ball mill. Then the refined Hf particles with diameters of 0.1–10 μm were dispersed in an acid Pt-plating solution, which mainly consists of K₂[Pt(NO₂)₂SO₄]. The preparation of K₂[Pt(NO₂)₂SO₄] solution can be referred to our previous publication [34]. To obtain an optimal Hf content after aluminisation, the addition of hafnium particles in the Pt-plating solution was controlled at 8–10 g L⁻¹. A counter-rotating magnetic stirrer was used at a speed of 15 r min⁻¹ to ensure that the Hf particles were uniformly suspended in the solution. Prior to electroplating, diluted H₂SO₄ was used to adjust pH value of the solutions. Details of the bath composition and electroplating parameters for depositing the Pt and Pt-Hf coatings are summarised in Table 1. For appropriate

comparison, the thickness of both Pt platings with and without the Hf dopant was 5 μm.

After electroplating, an annealing treatment to reduce residual stress and dilute the Pt content at the surface was conducted at 600 °C for 2 h and at 1050 °C for 1 h in vacuum ($<6 \times 10^{-3}$ Pa). The low-activity 'above-pack' aluminisation treatment was conducted at 1060 °C for 6 h in a vertical furnace filled with an argon atmosphere, in which the heating rate was controlled below 8 °C min⁻¹. The aluminising pack in the current study was composed of a 96 wt.% commercially available Fe-50Al (wt.%) powder and 4 wt.% NH₄Cl activator (analytically pure). Prior to aluminisation, a dehydration treatment for the aluminising pack was conducted at 120 °C for 24 h since the hot humid atmosphere was harmful to the adsorption of aluminium chlorides [35]. Finally, Hf-free and Hf-doped single phase β -(Ni,Pt)Al coating samples with a thickness of ~50 μm were prepared.

The isothermal oxidation test was performed in a muffle furnace in air at 1100 °C for 300 h. An electronic balance (Sartorius BP211D) with a sensitivity of 10⁻⁵ g was utilised to measure the average mass gain from three parallel samples at the oxidation intervals. The weights of the samples were recorded with an alumina crucible, ensuring the engagement of spalled oxide crumbs, and the crucibles were pre-heated at 1200 °C until no further weight change was observed. To preserve the oxide scales, the oxidised samples were electroplated with a thin layer of nickel and mounted in resin before cross-sectional observation.

X-ray diffraction (XRD, X' Pert PRO, Cu K α radiation at 40 KV, PANalytical, Almelo, Holland) was used to examine the phase constitution of the coatings. A field-emission scanning electron microscope (SEM, Inspect F 50, FEI Co., Hillsboro, OR) was used to observe the surface (secondary electron mode) and cross-sectional (back-scattered electron mode) morphologies of the coating samples. An electron probe micro-analyser (EPMA-1610, Shimadzu, J.P.) was employed to quantitatively measure the coating composition and plot the element mapping profiles to observe the distribution of the main elements in the coatings after oxidation.

3. Results

3.1. Preparation of the Hf-doped β -(Ni,Pt)Al coating

Fig. 1 shows the surface and cross-sectional morphologies of the Pt-Hf composite plating. As indicated in Fig. 1a, tiny and well-dispersed hafnium particles (relatively bright due to the discharging effect at the tips) were uniformly incorporated into the Pt plating after the co-deposition of electroplating. The inserted higher magnification image in Fig. 1a shows that the hafnium particles were mostly below 3 μm in size and either nipped at the Pt grain boundaries or wrapped inside Pt grains. As shown in Fig. 1b, the Hf particles were evenly distributed throughout the entire Pt-plating (Layer I). The grey appearance of the Hf particles in contrast to the white Pt domain was mainly due to the smaller atomic number of Hf. Layer II was formed by blasting with wet alumina grit, where a severe deformation of the superalloy surface was achieved.

After vacuum annealing and above-pack aluminisation, the cross-sectional morphology of the Hf-doped (Ni,Pt)Al coating is shown in Fig. 2a. The coating uniformly consists of an outer additive layer (~28 μm) and an interdiffusion zone (IDZ, ~17 μm), between which the Hf-rich belt (~6 μm) emerged. No Hf-rich particles were found precipitated inside the outer (Ni,Pt)Al coating, which is different compared to the visible segregation of Hf-rich particles at the grain boundaries acquired by other CVD methods [31,36]. Below the IDZ, some topologically close-packed (TCP) precipitates were formed as a round second reaction zone (SRZ) since the aluminisation process assisted in squeezing out refractory elements from γ

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