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The oxidation performance for Zr-doped nickel aluminide coating by composite electrodepositing and pack cementation

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ABSTRACT

Ni-Zr composite coatings were prepared by composite electrodepositing with the Zr particle loadings from 0.1 g/ L Zr to 0.7 g/L Zr. Then the Ni-Zr coatings were aluminized by the pack cementation and heat-treatment to obtain Zr-doped β -NiAl coatings. The effects of Zr on the coating microstructure and oxidation performance were investigated. The spallation resistance for Zr-doped NiAl coatings was better than the coating without Zr. The coating of 0.5 g/L Zr loading exhibited the best spallation resistance duo to a lower growth rate of the oxide scale and the formation of a mechanically interlocking oxide pegs structure.

1. Introduction

Although NiAl-based coatings especially Pt modified β -NiAl coatings have been widely used as an oxidation resistant coating or a bond coat in thermal barrier coatings due to the excellent oxidation resistance [1,2], the cost of the Pt is too high for industrial applications. Researchers found that doping small amounts of reactive elements (RE), e.g. zirconium (Zr), to β -NiAl bulks and coatings not only has beneficial effects on the oxidation behavior, but also drastically saves the cost compared with that of Pt modified β -NiAl coatings [3,4].

To apply RE-doped β -NiAl coating on the nickel-base superalloy, the chemical vapor deposition, the physical vapor deposition and pack cementation techniques were developed [5-8]. Among those methods, pack cementation method has been widely employed because of its low cost and easy production. However, pack cementation is a diffusion process and the alloy elements from the substrate would migrate to the coating, which can affect the oxidation performance [9,10], and make the discernment of the roles of RE from the effects of the other alloy elements difficult. In addition, the content of RE has a significant influence on the oxidation performance, but the controlling of the content and distribution of Zr in the coating is difficult. Composite electroplating, due to the low-cost of instrumentations as well as the possibility to control the content of the doping particles, has attracted much attention in recent years [11–14]. When a certain RE particlesdoped Ni coating is electroplated, subsequent pack cementation aluminizing is applied. This combined process can control both of the content and distribution of the RE dopants. Moreover, it can obtain the

RE-doped aluminide coating on any type of substrate without depending on the superalloy composition.

In the published literature, a lot of efforts have been made during the last decades to study the impact of RE, such as Zr, Hf, Y, et al. (or their oxides) which present in the alloys or coatings on the oxidation resistance [3,4,6-8,15,16]. A number of theories and mechanisms have been proposed to explain the effect of RE on the oxidation resistance, such as the oxide scale spallation, the growth rate of the oxide scale, the transformation of the θ -Al₂O₃ to α -Al₂O₃ in the transient oxidation stage, and the oxide scale stress [17-20]. However, the exact mechanisms behind these observations are still in debate, which depend either on differences in the composition, the microstructure of alloys and the coatings investigated, or on the differences in the concentration and techniques of RE doping. For the electrodeposited composite coatings, the small RE (or RE oxide) particles dispersed exhibited better high temperature oxidation resistance compared to RE-free coatings, which have been reported recently. For example, Zhou et al. [21] showed that the Y₂O₃-dispersed chromizing coating by electrodepositing and pack cementation had a superior oxidation resistance. Xu et al. [11] found that CeO_2 -dispersed δ -Ni₂Al₃ coating significantly increased the scale spalling resistance during cyclic oxidation. However, to the author's knowledge, there is no report about the oxidation behavior of the Zr particles dispersed β-NiAl coatings prepared by electrodepositing and pack cementation.

Therefore, the Zr-doped β -NiAl coatings were prepared by the composite electrodepositing and pack cementation in this paper. The effects of Zr on the aluminide coating microstructure, the oxidation

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Table 1

The compositions and parameters of the nickel-plating.

Composition (g/L)	Parameters
NiSO ₄ 6H ₂ O: 150–300	pH: 4-4.5
NiCl ₂ 6H ₂ O: 10–30	Temperature: 55 °C
H ₃ BO ₃ : 20–40	Current density: 50–100 mA/cm ²
CH ₃ (CH ₂) ₁₀ CH ₂ OSO ₃ Na: 0.5	Time: 100 min
Zr: 0/0.1/0.3/0.5/0.7	Agitation: 300 rpm

same, $0.5 \,\mu\text{m}$ colloidal alumina was used to polish the surface of the annealed sample. The polished samples (after cleaned in acetone) were placed in alumina crucibles and cyclically oxidized at 1150 °C using a chamber furnace up to 60 h in a laboratory air atmosphere. Every 12 h, the crucibles were removed from the furnace, air-cooled outside the furnace at room temperature for approximately 15 min. At least five samples for each kind of coating were prepared for examining the oxidation performances. Samples were removed from the furnace at the specified intervals for spallation and stress measurements, and then



Fig. 1. The cross-section images of electrodeposited composite coatings with various Zr particle loadings in nickel-plating bath: (a) without Zr, (b) 0.1 g/L, (c) 0.3 g/L, (d) 0.5 g/L and (e) 0.7 g/L. (f) The Zr volume fraction in the composite coatings as a function of Zr particle loadings in nickel-plating bath.

spallation resistance, the growth rate of the oxide scale, the transformation of the θ -Al₂O₃ to α -Al₂O₃ in transient oxidation stage and the oxide scale stress were investigated. Moreover, the influence mechanisms of Zr on the growth rate of the oxide scale and the oxidation spallation resistance were discussed.

2. Experiment

2.1. Materials

Hastelloy X alloy was used as the substrate and its nominal composition was 46.8Ni-22Cr-18Fe-9Mo-1.5Co-0.6W-1Mn-1Si-0.1C (in wt.%). First, the substrates were electrodeposited by nickel-plating bath with Zr loadings of different concentration. The average particle size of Zr powder was $\sim 2 \,\mu m$. The detailed electrodepositing para-Table meters were shown in 1. The substrate of $20 \text{ mm} \times 25 \text{ mm} \times 4 \text{ mm}$ was used as cathode and a nickel sheet of 99.99% purity with a dimension of 20 mm \times 30 mm \times 2 mm as the anode. The substrate and the nickel sheet were ground up to 600# sandpaper, and the Hastelloy X substrate surface was pretreated by etching in the HCl solution prior to the electrodepositing. Second, the electrodeposited samples were pack cementation by the powder of 97 wt.% Al (75–150 $\mu m)$ and 3 wt.% $\rm NH_4Cl$ at 650 $^\circ C$ for 10 h under an argon atmosphere. According to the literature [11,22], the aluminide coatings developed on the pure Ni or Ni-base alloys at temperatures below 700 °C were mainly composed of δ-Ni₂Al₃. To obtain β-NiAl phase, the aluminized samples were annealed at 1050 °C for 2 h in a flowing argon atmosphere. Oxides formed on the surface of the annealed coatings although annealing was performed in argon atmosphere. In order to ensure the surface condition of the coating was the returned for further cyclic oxidation. Some samples were withdrawn from cycling, mounted in epoxy and polished to a mirror finish for observing the thickness of the oxide scale.

2.2. Characterization

The scanning electron microscopy (SEM, Inspect F50, FEI) in conjunction with energy dispersive spectroscope (EDS, Oxford Instruments) was used to determine the microstructures and chemical compositions of the samples. X-ray diffraction (XRD, Ultima IV, Rigaku) with CuK_{α} radiation was used for the phase identification. The volume fraction of Zr particles in the as-electrodeposited Ni coating, defined as the ratio between the Zr particles area and the total area of the coating, was examined by SEM. At least 10 cross-section images at 1000 times magnification were taken from each sample, which were then analyzed using an image processing software (ImageJ). The spallation of the oxide scale was examined by using an optical microscopy (BX51M, Olympus) and SEM. More than five images by SEM were taken from each sample to estimate the spallation degree of the oxide scale, which is defined as the ratio between the spalled area and the total area of the oxide scale in the image. The analysis was performed using the image processing software, Image J. The stress and θ -Al₂O₃ phase in the oxide scale were measured by photo-stimulated luminescence spectroscopy (PSLS). The PSLS spectra were collected using a LabRAM HR Evolution Raman microprobe system (Horiba, France) with a 50 \times objective lens and a 532 nm laser source at room temperature. For each sample, at least 25 positions were collected on the centre of the oxide scale surface and the average values were taken. All the spectra were fitted by the Lorentzian-Gaussian function using the Labspec 5 software to obtain the peak position and peak area.

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