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Synergistic effect of reactive element co-doping in two-phase ($\gamma' + \beta$) Ni–Al alloys

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ABSTRACT

To propose reactive element (RE) co-doping strategy, the oxidation behaviour of Dy, Hf, Y, Zr single- and co-doped Ni–30Al alloys was comparatively studied. All the alloys containing Dy or Y exhibited improved scale adhesion due to the pinning effect of the pegs at the scale/alloy interfaces and the reduced residual stress by the flaky oxides formed on the alloy surfaces. Compared to the corresponding RE single-doping, Dy/Hf, Dy/Zr, Hf/Y and Y/Zr co-doping further reduced the scale growth rate, revealing a synergistic effect. The role of reactive element co-segregation in the formation of synergistic effect was analyzed.

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1. Introduction

For years, reactive element (RE) modified β -NiAl has drawn much attention [1–5] due to its good high-temperature oxidation resistance, especially above 1200 °C. The investigations have shown that REs (e.g., Hf, Zr, Y and Dy) doped in β -NiAl can not only improve the adhesion of the scale/alloy interface, but also lower the oxide scale growth rate, and the effectiveness on these two aspects is in the order of Hf and Zr, followed by Dy and Y, respectively [5]. Based on the results collected above, research efforts were initiated to develop nickel aluminide based coatings, hoping to replace the conventional diffusion aluminide and MCrAlY overlay coatings. Meanwhile, the reactive element effect (REE) started to become a special focus of attention again [6,7].

REE was first found by Pfeil [8] in the patent in 1937. Since that date, a lot of relevant reports have been published [1,2,5,9–14]. However, although great progress has been made up to now, the mechanical explanation is still in debate [15]. Because the role of reactive element strongly depends on the doping methods, the matrix alloys and the service conditions, which makes the study complicated. Moreover, different REs exhibit different effects on

high-temperature oxidation and the doping level also affects the REE. Undoubtedly, the over doping of REs leads to severe internal oxidation. In viewing of this, Pint proposed a strategy named “co-doping” [16] to exert each advantage of REs and to minimize the total doping level.

In fact, many commercial alumina-forming alloys like René N5 and Haynes alloy 214 have contained more than one reactive element dopant, and the resulting performance reveals a great improvement [16]. Some research on the co-doping has also been carried out. It is reported that compared to the Y-doping, the Hf/Y co-doping produced a better benefit on the scale adhesion of NiCrAl and FeCrAl alloys [17–19]. Recently, an interesting work by Guo et al. [20] showed that Hf and Zr as well as Y and La preferred to co-segregate at grain boundaries and thus produced a “synergistic effect” which further lowered oxide scale growth rate of β -NiAl as compared to the corresponding RE single-doping. Obviously, it has become increasingly clear from the applications and research that co-doping leads to the best overall oxidation performance thus opens the possibility for future improvements. Despite all this, the co-doping effect of REs on the cyclic oxidation behaviour is not yet clarified.

Moreover, as mentioned previously, studies of RE modified single phase β -NiAl have been widely reported, but the effect of REs in two-phase ($\gamma' + \beta$) Ni–Al is seldom investigated. However, phase transformation from β -NiAl to γ' -Ni₃Al or even to γ would happen inevitably in the β -NiAl based coatings during service at high tem-

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peratures [21–24]. As a result, the operating environment for the REs would be changed and the REE would be more complicated. On the other hand, the ductile γ' phases can result in remarkable enhancement of high-temperature strength and room temperature ductility for β -NiAl [25]. So, there is great significance in exploring the REE in two-phase ($\gamma' + \beta$) Ni-Al alloy.

In the present paper, Ni-30Al with γ' and β two phases was used as base alloy. One or two REs including Dy, Hf, Y and Zr were doped into the alloy. Oxidation behaviour of these alloys was studied at 1100 °C, aiming to systematically compare the effects of REs in affecting high-temperature oxidation of two-phase ($\gamma' + \beta$) alloy. Structures, compositions and oxidation resistance of the tested samples were evaluated in this paper and the role of REs used for single- and co-doping was preliminary discussed.

2. Experimental procedures

Designed compositions (all in at.%) of the alloys used in this study were Ni-30Al-0.1A (A = Dy, Hf, Y or Zr) for the single-doped ones and Ni-30Al-0.05A-0.05B (A, B = Dy, Hf, Y or Zr, and A \neq B) for the co-doped ones. As can be seen, to ensure the same doping level, the total content of REs for each alloy was 0.1 at.%.

The alloys were produced by arc melting and casting in argon atmosphere using high purity Ni ($\geq 99.9\%$), Al ($\geq 99.99\%$), Dy ($\geq 99.9\%$), Hf ($\geq 99.9\%$), Y ($\geq 99.95\%$) and Zr ($\geq 99.96\%$) as raw materials. After casting, the alloy ingots were heat treated in the vacuum at 1200 °C for 20 h. The actual compositions of the as-annealed alloys are listed in Table 1. Note that to ensure the detect precision, the contents of Al and REs were identified by chemical titration and inductively coupled-plasma (ICP) analysis, respectively, while C and S contents were determined by high frequency combustion-infrared carbon-sulphur spectrometry. As seen in the table, the compositions are very close to those designed and the slight differences will not lead to significant effect on the oxidation behaviour of the alloys.

Rectangular coupons with dimensions of $12 \times 10 \times 3 \text{ mm}^3$ were cut from the ingots. All the surfaces of the coupons were carefully ground by SiC abrasive papers from 400-grit to 800-grit. After that the dimensions of the coupons were measured by a vernier calliper with a precision of 0.02 mm. And then, they were ultrasonically cleaned in acetone and alcohol.

Cyclic oxidation tests at 1100 °C were performed in air in an open-ended tube furnace. Each specimen was placed in a constant-weight alumina crucible. To capture the spalled oxides, a crucible cover was also used. During every cycle the specimens were oxidized in the furnace for 5 h at 1100 °C and then were moved out and cooled to room temperature in 10 min, after which the mass change of the specimen and the total mass gain (specimen with crucible and cover) were both measured by an electronic balance (Sartorius CPA 225D, Germany) with a precision of 0.01 mg. In addition, it should be noted that three specimens for each alloy were simultaneously tested and the average value was obtained from the measurements made on the three specimens. Afterwards the average values were adopted to determine the oxidation kinetic curve for each alloy.

Field emission-scanning electron microscope (FE-SEM, Quanta 200F) combined with energy dispersive X-ray (EDX) spectrum and electron probe micro-analyzer (EPMA, JXA-8100) were utilized to examine the microstructure and chemical compositions of the specimens. And the different phases were identified by X-ray diffraction (XRD, D/max2200PC) using Cu $K\alpha$ radiation ($\lambda = 0.154056 \text{ nm}$). To analyze the more detailed microstructures of the oxide scales, a transmission electron microscope (TEM, FEI-F20) equipped with EDX and high-angle annular dark field (HAADF) detectors was used. The cross-sectional TEM samples were pre-

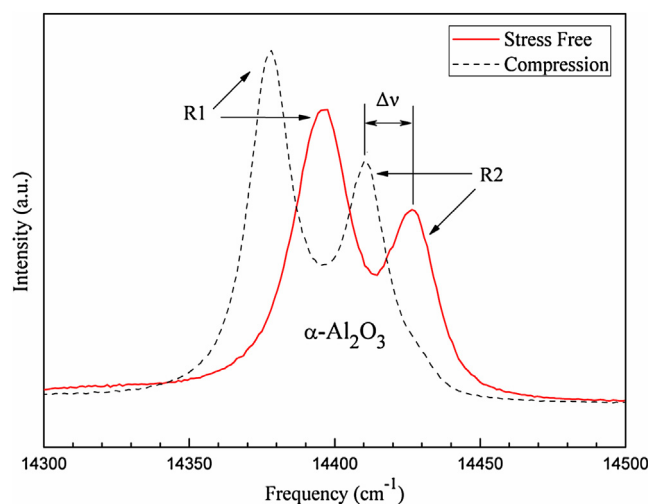


Fig. 1. The luminescence spectra showing the characteristic doublet (Peak R1 and R2) of α -Al₂O₃ and its frequency shift $\Delta\nu$ caused by stresses in the oxide scale. Note that the stress-free reference spectrum is obtained by PSLs from a stress-free single crystal sapphire.

pared by focused ion beam (FIB) thinning and a Pt film was applied to protect the outer surface of the oxide scale.

The stresses in the scales with different morphologies were measured by photo-stimulated luminescence spectroscopy (PSLS). The Cr³⁺ luminescence spectra were acquired from the scale surfaces using Raman spectroscopy (LabRAM HR Evolution, HORIBA Jobin Yvon, France). More than ten random positions were measured for each kind of scales on the same specimen, and the positions were far away from the edges as well as the spalled regions. In this way, stress relaxation in the surrounding areas resulting from spalled regions and specimen edges can be eliminated. Residual stresses can be calculated by the following equation [24,26–28]:

$$\Delta\nu = 5.07\bar{\sigma} \quad (1)$$

where $\Delta\nu$ (cm⁻¹) is the frequency shift of R2 peak (illustrated in Fig. 1), and $\bar{\sigma}$ (GPa) is the biaxial stress in the scale. Finally, the average value was obtained from the ten random measurements mentioned above for each specimen.

3. Results and discussion

3.1. Single-doping

The comparison of the total mass gains and specimen mass changes of the Ni-30Al and Ni-30Al-0.1A (A = Dy, Hf, Y or Zr) alloys after 200 h cyclic oxidation at 1100 °C is shown in Table 2. The difference between the total mass gain and the specimen mass change represents the mass loss, which can be used to estimate the scale spallation. As can be seen, after 200 h oxidation, the mass changes of Ni-30Al, Ni-30Al-0.1Hf and Ni-30Al-0.1Zr alloys are all negative and the mass losses for them are 6.21, 2.81 and 1.97 mg/cm² respectively, indicating that severe scale spallation occurred during oxidation. By contrast, almost no spallation was observed on the surfaces of Dy- and Y-doped alloys, leading to slight mass loss shown in the table. Based on this, it can be concluded that the doping of Dy and Y can significantly improve the scale adhesion of Ni-30Al alloy while Hf and Zr both have no obvious effect on this aspect.

The main reason for this result has been discussed in our previous work [29]. As shown in Fig. 2a and b, the undoped and RE single-doped Ni-30Al alloys are all composed of two main phases,

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