



Cyclic oxidation of β -NiAl with various reactive element dopants at 1200 °C

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ABSTRACT

Cyclic oxidation of β -NiAl alloys doped with different reactive elements (REs) each of which had an optimized concentration was investigated. All of REs effectively suppressed growth of voids beneath oxide scales formed on the alloys. The La-doped alloy suffered from severe internal oxidation. The Hf or Zr-doped alloys exhibited lower oxidation rate, while the Dy-doped alloy showed less scale rumpling. The effect mechanisms associated with solid solubility and ion size of RE dopants on cyclic oxidation were discussed. Co-doping of REs, such as Dy and Hf or Zr, could be anticipated to provide better cyclic oxidation performance.

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1. Introduction

The high melting point, low density and good isothermal oxidation resistance of β -NiAl intermetallic compound has been widely documented over the past decades [1–3]. Recently, β -NiAl has been considered as a potential candidate for high-temperature protection of underlying superalloy substrates and suitable bond coat in thermal barrier coating (TBC) system due to these desirable properties [4–10]. However, the alumina scale formed on NiAl spalls readily during high-temperature cyclic oxidation, especially above 1200 °C.

To improve oxide scale adhesion to NiAl alloy or coating, RE additions of Hf, Zr, Y and La as well as their oxides dispersions in NiAl were systematically studied [11–15]. Numerous attempts have been made to clarify the beneficial RE effects on cyclic oxidation performance, but the mechanical explanation is still in debate. For alumina-forming alloys, a continuous flux of RE ions diffusing from the alloy to the scale/alloy interface and then to the scale grain boundaries is essential to realize the beneficial RE effects, which is recognized as dynamic segregation theory (DST) [16,17]. It reveals that the reduction in oxide scale growth rate by RE doping is due to the slow outward diffusion of RE ions inhibiting the outward diffusion of Al ions. Several hypotheses are also proposed concerning the enhancement on oxide scale adhesion by RE additions [18–24]. The formation of voids beneath the oxide scale

weakens the interfacial bonding and impurity elements such as sulfur in alloys segregates to the scale/alloy interface and promotes the interfacial void growth. RE ions are considered reacting and tying up sulfur due to their strong affinity for sulfur [18,19,25,26]. Apart from sulfur, other impurities such as C, O and P in alloys might be also detrimental to the oxidation performance [27–29]. Recent studies have indicated that mechanical interlocking of the oxide scale by “pegs” formed at the oxide/metal interface plays a dominant role in improving scale adhesion, while the excellent oxidation resistance of oxide dispersion strengthened alumina-forming alloys reveals that this pegging mechanism is sufficient but not necessary for optimizing the oxidation performance [14,17,23,24].

Dysprosium (Dy) is also a typical reactive element, but its benefit in improving oxidation resistance of alumina-formers has been just reported in recent years. The addition of Dy in NiAl significantly improved cyclic oxidation performance in mechanisms similar to other RE additions did [30–33]. However, no efforts are made to compare the capabilities of improving oxide scale adhesion between Dy and other reactive elements as quantifying the beneficial RE effects is rather difficult. In attempting to do this, identifying the differences in solubility, reactivity and diffusion kinetics between these reactive elements in Ni–Al system are necessary.

Early results suggested that similar mechanisms occurred when the RE was doped in NiAl as either an oxide dispersion or an alloy addition, but the addition of RE as an oxide dispersion which strengthens the alloy substrate might deteriorate oxide scale adhesion as a weaker substrate is better able to dissipate strain energy by deforming [14,17,34,35]. Besides, ion implantation is less

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Table 1
Chemical compositions of the as-annealed NiAl alloys determined by inductively coupled-plasma analysis (in at.%).

| | Al | Dy | Hf | Zr | Y | La | C (ppm) | O (ppm) | S (ppm) |
|---------|-------|------|------|------|------|------|---------|---------|---------|
| NiAl | 49.22 | – | – | – | – | – | 37 | 78 | <20 |
| NiAl–Dy | 49.22 | 0.05 | – | – | – | – | 120 | 36 | <20 |
| NiAl–Hf | 48.66 | – | 0.05 | – | – | – | 78 | 64 | <20 |
| NiAl–Zr | 49.75 | – | – | 0.09 | – | – | 62 | 33 | <20 |
| NiAl–Y | 49.80 | – | – | – | 0.06 | – | 73 | 22 | <20 |
| NiAl–La | 49.61 | – | – | – | – | 0.09 | 76 | 22 | <20 |

effective in improving the performance of alumina-formers than chromia-formers [36–38]. Therefore, arc-melting was chosen for specimen preparation in the present work.

In this work, a comparative research is carried out on different RE-doped NiAl alloys to investigate into the roles of different reactive elements in affecting the oxide scale growth rate and scale adhesion, to better understand those factors affecting the strength of the RE effects.

2. Experimental procedures

2.1. Alloy preparation and evaluation

NiAl alloys containing various reactive elements were used in this work. To ensure that each reactive element in the alloy had an optimized concentration, the nominal doping level of each reactive element was ascertained according to literature values [11–15]. The designed compositions were Ni–50Al, Ni–49.95Al–0.05Dy, Ni–49.95Al–0.05Hf, Ni–49.9Al–0.1Zr, Ni–49.92Al–0.08Y and Ni–49.9Al–0.1La (in at.%), respectively. High purity nickel, aluminum and reactive elements (Dy, Hf, Zr, Y and La) were used as starting materials. The alloy buttons were produced by arc-melting and casting in argon atmosphere and annealed in vacuum at 1300 °C for 24 h in order to ensure homogenization of the alloys. The chemical compositions of the as-annealed NiAl alloys are listed in Table 1. The concentration of nonmetallic impurities including C, O and S was also determined. Note that the contents of elements in alloys were slightly different from those designed. However, it is believed that this difference should not have significant effect on the cyclic oxidation performance of the NiAl alloys. The slightly different impurity levels were caused by alloy preparation. Specimens were cut from the center of the button-shaped alloys into $10 \times 8 \times 3 \text{ mm}^3$, ground to an 800-grit SiC finish and ultrasonically cleaned in alcohol and acetone prior to oxidation.

Cyclic oxidation tests were carried out in an air furnace equipped with an automation system for moving the specimens in and out of the furnace. Each specimen was hung in a pre-annealed alumina crucible by Pt wire to capture any spalled oxide. One cycle includes 50 min heating in dry, flowing air at 1200 °C and subsequent 10 min cooling to ambient temperature by compressed air. Both mass changes and mass gains (specimen + spalled oxide) were measured using an electronic balance (Sartorius CPA 225D, Germany) with a precision of 10^{-4} g . The mass gain for each alloy was determined based on an average value of two specimens' weight gains and was corrected for the evaporation of the Pt wire.

The surface, cross-sectional and fracture morphologies of the specimens were characterized by a field emission-scanning electron microscopy (FE-SEM, Quanta 200F) equipped with energy dispersive X-ray spectrum (EDXS). The phases of the oxides were identified by X-ray diffraction (XRD, Rigaku D/max2200PC) using Cu K α radiation. The chemical compositions of the alloys and the oxides were determined by an electron probe micro-analyzer (EPMA, JXA-8100). For cross-sectional observation, the specimens

after desired oxidation time were embedded in epoxy, ground and finely polished.

2.2. Calculation of solid solubilities

The solid solubility of a RE dopant in β -NiAl corresponds to the maximum concentration that the dopant can attain. First-principles calculations [39–42] are introduced for predicting the concentration of RE dopants (including Dy, Hf, Zr, Y and La) in NiAl based on impurity formation energies (E_f). The impurity formation energy calculated from first-principles reflects energy variation induced by the ingress of ternary elements into NiAl lattice at 0 K. When the atom radius of the ternary element is larger than Ni and Al, it can only substitute Ni or Al atom in the lattice. Therefore, E_f can be obtained by the following equation [43]:

$$E_f = E_t^{M-X} - E_t^M + E_{\text{bulk-state}}^A - E_{\text{bulk-state}}^X \quad (1)$$

where M represents NiAl, X represents a RE atom, A denotes a substituted atom, E_t^M and E_t^{M-X} are the total energies of M and X -doped M at their equilibrium lattice constant, and $E_{\text{bulk-state}}^A$ and

Table 2
Impurity formation energies of different reactive elements in β -NiAl calculated from first-principles (in eV).

| Reactive element | E_f (Ni site) | E_f (Al site) |
|------------------|-----------------|-----------------|
| Dy | 2.71 | 0.79 |
| Hf | 2.74 | 1.08 |
| Zr | 3.61 | 1.92 |
| Y | 3.74 | 1.88 |
| La | 5.64 | 3.71 |

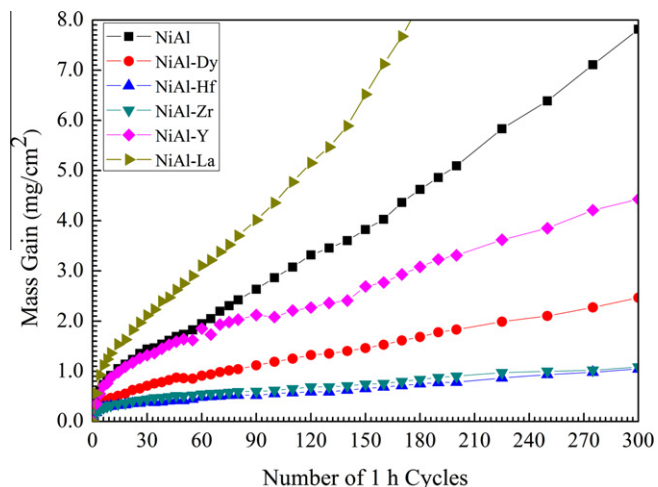


Fig. 1. Mass gains (specimen + spall) for NiAl alloys with various reactive element additions during 300, 1 h cycles at 1200 °C in air.

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