



High-temperature oxidation behavior of minor Hf doped NiAl alloy in dry and humid atmospheres

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ABSTRACT

Oxidation behavior of NiAl and 0.05 at.% Hf doped NiAl alloys were investigated at 1100 °C in dry and humid atmospheres. Hf doping significantly improved the cyclic oxidation resistance. Water vapour promoted the formation of voids at the scale/alloy interface and accelerated scale spallation. Also, water vapour had effect on the phase transformation from θ - to α -Al₂O₃ at the initial oxidation stage. In humid atmosphere, more Hf segregated at the scale/alloy interface to form oxide pegs. Pre-oxidation process in O₂ + Ar could compromise the effect of humid atmosphere on the oxidation kinetics of the NiAl alloys.

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1. Introduction

NiAl intermetallic compounds exhibit many excellent properties including low density, high melting point and are expected to be promising materials for high temperature applications, especially as metallic protective coatings for superalloys or as bond coats in thermal barrier coatings (TBCs) [1–6]. Although β -NiAl has great potential, it also has many inherent problems, including low ductility and fracture toughness at ambient temperature and inadequate strength and creep resistance at elevated temperature. Another important issue concerning β -NiAl is that the oxide scale grown on the alloy tends to spall at elevated temperatures due to the formation of voids and surface rumpling of the scale [7–9]. In order to overcome these problems, the addition of reactive elements (REs) such as Hf, Zr and Y to NiAl has been widely investigated [10–12]. Experimental results showed that reactive elements had significant effect on the oxidation behavior of alumina-forming alloys at high temperature. The research of Guo et al. showed that the addition of Hf not only reduced the growth rate of the oxide scale, but also enhanced the oxide scale adherence [13–16].

Water vapour exists in nearly all atmospheres where metallic materials are used in power generation and aerospace sectors at high temperatures [17]. The combustion atmosphere in turbine

engine is very rich in water vapour because of the intake of hydro-sphere and the combustion of fuel oil. It has been reported that water vapour has a remarkable influence on the oxidation behavior of superalloys and high-temperature protective coatings [18–27]. However, the influence mechanism of water vapour on the oxidation behavior of NiAl based alloys modified by minor reactive elements is not yet fully understood.

In this work, a comparative research was carried out on NiAl alloys with and without Hf in dry and humid atmosphere at 1100 °C.

2. Experimental

Undoped NiAl alloys (Ni–50Al, in at.%) and 0.05 at.% Hf doped NiAl alloys were produced by arc melting and then annealed in vacuum for 24 h at 1300 °C in order to ensure homogenization of the alloys. The chemical compositions of tested alloys are listed in Table 1. Disk-shaped specimens (Φ 10 mm \times 3 mm) with two holes (Φ 1 mm) for hanging the specimens were fabricated by spark cutting. The specimen surfaces were reduced to an 800 grit SiC finish and ultrasonically cleaned in acetone prior to oxidation testing.

Oxidation tests in atmospheres containing water vapour were carried out in a set-up test rig, as shown in Fig. 1. The setup included two systems, one of which was the semi-closed gas circuit system consisting of gas cylinder (Fig. 1-1), flowmeter (Fig. 1-2), glass tubes, saturators (Fig. 1-4), quartz tubes (Fig. 1-8), paraffine (Fig. 1-9) and outlet (Fig. 1-10), the other was the furnace (Fig. 1-7) heating system. The content of water vapour in atmospheres was determined by the saturation vapour pressure of water at different temperatures. And the saturation vapour pressure of water

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Table 1

Chemical compositions of tested alloys determined by inductively coupled-plasma analysis (in at.%).

Alloys	Ni	Al	Hf	C (ppm)	O (ppm)	S (ppm)
NiAl	Balance	49.16	–	42	68	<20
NiAl–Hf	Balance	48.97	0.05	47	64	<20

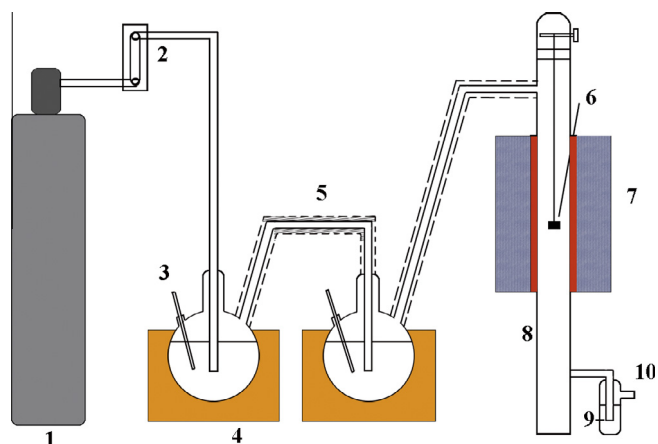


Fig. 1. Schematic illustration of the oxidation in humid atmosphere. 1. synthetic gas; 2. flowmeter; 3. thermocouple; 4. saturators; 5. heating tapes; 6. specimen; 7. furnace; 8. quartz tube; 9. paraffine; 10. gas outlet.

at 54 °C was 0.15 atm. Water vapour was generated in two distilled water saturators in series (nearly the desired dew point 54 °C measured by thermometers (Fig. 1–3)), which was carried by mixed air (20 vol.% O₂ + Ar) with a flow rate of approximately 100 ml/min. To avoid the condensation of water vapour, the glass tubes were heated by heating tapes (Fig. 1–5) to keep the atmosphere temperature in the glass tubes above the dew point. The two distilled water saturators and the glass tubes were heated for 2 h before oxidation to guarantee that the 15% water saturation of oxidation atmosphere was achieved. The atmospheres were 20% O₂ + Ar, 15% H₂O + 17% O₂ + Ar and 15% H₂O + Ar. During the oxidation in dry atmosphere, the saturators were removed, and then the mixed air flowed into the quartz tube directly. The specimens (Fig. 1–6) hung by platinum wires were oxidized for short time, 20 h and 100 h at 1100 °C and the mass change was determined by an electronic balance (Sartorius CPA225D, Germany) with a precision of 0.01 mg. The platinum wires for hanging the specimens were also exposed for 2 h in both dry and humid atmospheres prior to oxidation tests to ensure that the effect of the formation of PtO₂ (g) on the weight change could be eliminated.

Field emission scanning electron microscopy (FE-SEM Hitachi S4800, Japan) equipped with energy dispersive X-ray spectrum (EDXS) was used to investigate the morphologies and compositions of the oxide scales grown on the specimens. The phases of oxides were identified by luminescence spectra excited with a probing argon-ion laser (RM 2000 Renishaw).

3. Results and discussion

Fig. 2 shows the mass changes of NiAl and NiAl–0.05Hf samples during 100 h oxidation in 20% O₂ + Ar, 17% O₂ + 15% H₂O + Ar and 15% H₂O + Ar at 1100 °C, respectively. For the undoped NiAl samples, apparent mass loss was observed after 20 h oxidation in O₂ + Ar, indicating severe oxide scale spallation. In O₂ + H₂O + Ar mixed atmosphere, scale spallation occurred even earlier, since abrupt mass loss was observed within 10 h. While in H₂O + Ar,

spallation of oxide scale became more severe. It can be inferred that the undoped NiAl alloy revealed very poor cyclic oxidation resistance in both dry and humid atmosphere and water vapour accelerated scale spallation of the undoped NiAl.

3.1. The initial oxidation

Saunders et al. [17] reported that water vapour might have a greater effect on the initial stages of oxidation and affect the stability of the transition alumina. The research of Pint et al. [28] showed that larger ions such as Y, Zr, La and Hf appeared to slow the θ - to α -Al₂O₃ phase transformation. To investigate the effect of water vapour and reactive element Hf on the initial oxidation of β -NiAl alloys, the surface morphologies of the oxide scales formed on the NiAl and Hf-doped NiAl alloys after short-time oxidation at 1100 °C in different atmospheres were examined.

Fig. 3 shows the surface microscopic morphology of the undoped NiAl alloy after the initial oxidation at 1100 °C in different atmospheres. Typical scale ridges (Fig. 3a) were observed on the undoped NiAl after 5 min oxidation in O₂ + Ar. The scale ridges grew up after further oxidation, as shown in Fig. 3b. However, granular oxides (Fig. 3c) rather than scale ridges were firstly observed on the surface after 5 min oxidation in O₂ + H₂O + Ar mixed atmosphere. Interestingly, obvious scale ridges (Fig. 3d) were formed around granular oxides on the surface after 15 min oxidation in mixed gases. Compared with O₂ + H₂O + Ar mixed atmosphere, the number of the granular oxides formed on the NiAl alloys were much more after 5 min oxidation in H₂O + Ar, as shown in Fig. 3e. The granular oxides grew gradually after another 10 min oxidation, which were presented in Fig. 3f. Oxide ridges (Fig. 3g) became apparent on the oxide scale after 30 min oxidation in H₂O + Ar at 1100 °C.

Fig. 4 shows the FE-SEM images of surfaces of NiAl–0.05Hf after the initial oxidation at 1100 °C in different atmospheres. Needle-like oxides (Fig. 4a) rather than typical oxide ridges were formed on the NiAl–0.05Hf after 5 min oxidation in O₂ + Ar, suggesting that Hf addition greatly inhibited the outward short circuit transport of Al. With the increase of oxidation time in O₂ + Ar, the surface morphology of the oxide scale changed and needle-like oxides gradually disappeared (Fig. 4b and c). In O₂ + H₂O + Ar mixed atmospheres, needle-like and blade-like oxides were presented during the initial oxidation (Fig. 4d–f). Similar oxides (Fig. 4g) were also visible on the Hf-doped NiAl alloys after 5 min oxidation in H₂O + Ar. In the subsequent oxidation process, the morphology of the oxides changed (Fig. 4h and i), probably due to phase transformation. A noted phenomenon was that crack-like ditches formed

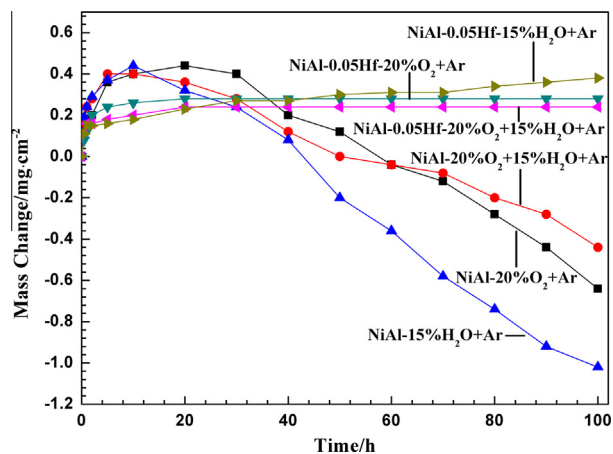


Fig. 2. Oxidation kinetics of NiAl and NiAl–0.05Hf in dry/humid atmosphere.

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