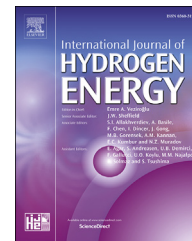




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Microstructural and oxidation behavior of Ni–Cr–Al super alloy containing hafnium at high temperature

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ABSTRACT

In this research, Ni–12Cr–4.5Al–xHf alloys containing 0.1, 0.2 and 0.4 wt% Hf were produced and their isothermal oxidation behavior has been studied in air for 100 h at the temperature of 1000 °C. Microstructures of the oxidized samples were examined using scanning electron microscopy (SEM). The samples were observed to exhibit different microstructures, and oxidation behavior depending on their chemical composition. The results indicated that the sample containing 0.4 wt% Hf had the lowest weight gain, while the weight gain of the sample without Hf was the highest. Layer investigations show that an external scale of spinel overlying a region of internal oxides precipitates was formed on Ni–12Cr–4.5Al. On the other hand, an external Al₂O₃ formed on the samples containing Hf additions. The improvement in the oxidation resistance is believed to be due to the transition between the internal and external oxidation of aluminum and also due to the formation of the relatively continuous, adherent and protective Al₂O₃ oxide layer on the surface of the Ni–12Cr–4.5Al–0.4Hf alloy. Microstructural investigations using SEM show that the layer adhesion can be affected by mechanical keying at the alloy/scale interface resulting from the pegs' formation during oxidation.

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Introduction

Requirement for high-temperature resistant materials have been increasing due to improvements in aircraft engine and gas turbine system designs. Ni-based alloys have been widely used in highly demanded industrial parts such as turbine blade, aerospace components, chemical plants, jet engines, and solid oxide fuel cells (SOFC) due to their high oxidation and wear resistance, and also good mechanical properties at high temperatures [1–3]. Thus, huge efforts have been made toward improvement in oxidation properties of alloys that are

exposed to high temperatures and also extreme corrosive environments [4–7].

A fuel cell is a device that produces electricity through a chemical reaction, promising power generation with high efficiency and low pollution emission [8]. The cell generally operates at temperature range of 600–1000 °C where ionic conduction by oxygen ions takes place. To produce high voltages, individual cells are joined by interconnects to form a fuel cell stack. During recent years, oxidation-resistant alloys such as Fe–Cr and Ni–Cr based alloys have become promising candidates as the interconnect materials [9,10]. During the fuel cell operation, the interconnect material is

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simultaneously exposed to a fuel on the anode site and air on the cathode site. Hence, the alloys used as interconnect materials are required to exhibit sufficient oxidation resistance at the high operating temperatures and also very challenging dual environments.

The formation of a protective α -Al₂O₃ oxide layer on the surface of Ni-based alloys is the most desirable aim because of two reasons: first, the low diffusion rate of oxygen and metal ions through the alumina scale; second, its good chemical and thermal stability at high temperatures [11,12]. However, the oxide films formed on the Ni-based alloys at high temperatures generally consist of a rapid-growing non-protective oxide such as Cr-rich volatile oxides and spinels [13–15]. Furthermore, in some high temperature applications, like SOFCs, chromium oxides volatilization can act as a potential contaminant [16,17]. By using high contents of Al in Ni, Fe and Cr binary alloys systems, it is possible to develop an alumina scale on the surface [18–20]. Unfortunately, these high levels of Aluminum in binary alloys often cause undesirable mechanical properties. So, adding a third or even further alloying elements to the binary systems, in order to reduce the concentration of Al required establishing an alumina layer, is a good solution for this issue.

After the first report of reactive element effect (REF) on the oxidation resistance of Ni–Cr alloys in 1937 [21], an extensive literature have focused on the role of reactive elements (REs) in alumina-forming alloys [22–25]. The oxidation behavior of the Ni-based super alloys can be improved by the addition of reactive elements to their composition, which forms an adherent Al-rich oxide layer during oxidation at high temperatures. Most of the past investigations have focused on the alloys or coatings' compositions containing relatively small concentrations of reactive elements [26–29]. These studies demonstrated that addition of reactive elements can dramatically improve the oxidation resistance.

Hafnium, as a typical reactive element, can enhance the high-temperature oxidation resistance and scale adherence of Ni-based alloy systems [30–32]. Despite the high level of interest in using Hf and other reactive elements, there is relatively little information available about the effect of different dosage of Hf additions on the oxidation performance in a Ni–Cr–Al alloy system.

The Ni-based intermetallic compounds have good high-temperature properties including high melting point, relatively high density and good corrosion performance; however, their application as an interconnect in SOFCs has poorly been studied. The aim of this research is to study the oxidation behavior of the alloy from Ni–Cr–Al system and to determine the effect of 0.1–0.4 wt percent addition of hafnium at the temperature of 1000 °C. Moreover, Microstructure, composition and oxidation resistance of the tested alloys were investigated in this study and the role of Hf was discussed.

Experimental procedure

Materials and preparation

Three Ni–12Cr–4.5Al–xHf alloys containing 0.1, 0.2 and 0.4 wt % Hf and one Ni–Al–Cr alloy with similar aluminum and

chromium contents (Ni–12Cr–4.5Al) were prepared from high purity metals by vacuum induction melting. In order to remove the residual mechanical stresses and ensure homogenization of the alloys, the ingots were then annealed at 1200 °C for 24 h in 1 atm argon environment. The alloy without Hf is referred as “the base alloy” in the following for comparison with other alloys. Rectangular samples with dimensions of 10 mm*10 mm*3 mm were cut from the ingots. Prior to oxidation tests, all the specimens were ground with SiC abrasive papers up to 800 grit, and then degreased in ethanol.

Oxidation experiment

Isothermal oxidation experiments were carried out in air at 1000 °C for 100 h in a tube furnace. Samples were placed in individual alumina crucibles with covers in order to capture any spalled oxides.

Characterization of the oxide scales

Weight changes of the samples after oxidation process were measured using a balance with 0.001 mg accuracy at room temperature. Parabolic rate constant (k_p) for all the oxidized samples were also measured using Eq. (1).

$$\Delta m = k_p \sqrt{t} \quad (1)$$

where, Δm is the mass changes of the samples before and after oxidation, and t is the oxidation time.

Oxidized samples were mounted in an epoxy resin and then ground with silicon carbide papers to 1200 grit finish. Scanning electron microscopy (SEM; Philips XL-30) equipped with energy dispersive spectroscopy (EDS) was used for examination and identification of the oxide films formed on samples after 100 h exposure time at 1000 °C. EDS analysis was employed to identify the elemental composition of the phases present in the oxide scales. XRD experiments were carried out on a Siemens D-500 diffractometer with Cu K α a radiation in the range of 20–90° with a step width of 0.02° in order to determine the phases present in the oxides.

Results

The oxidation kinetics of the samples after 100 h oxidation at 1000 °C in air are presented in Fig. 1 in form of the specific mass gain as a function of oxidation time. Fig. 1a and b shows the mass gain and mass changes plots, respectively. The parabolic curves of the samples also presented in Fig. 2. Higher mass gain can be observed for the Ni–12Cr–4.5Al alloy and the lowest mass gain was obtained for the alloy containing 0.4 wt% Hf. As shown in Fig. 1a, the base alloy keeps a fast oxide layer growth throughout the entire oxidation process and yields a total mass gain of more than 2 mg/cm² at the end. In contrast, the total mass gains for the alloys containing 0.1, 0.2 and 0.4 wt% of Hf after 100 h are 1.6 mg/cm², 0.68 mg/cm² and 0.35 mg/cm², respectively. For the sample containing 0.1 wt% Hf, the slope of the plot (Fig. 1a) is still high even at higher oxidation times, which indicates the formation of a non-protective oxide film on the surface. Furthermore, measurements for Ni–12Cr–4.5Al–0.2Hf and

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