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High temperature cyclic oxidation of Ni based superalloys at different temperatures in air

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ABSTRACT

In present study high temperature cyclic oxidation behavior of Ni-based superalloy (Inconel 617) was investigated. The cyclic oxidation test was carried out at various temperatures 750 °C, 850 °C, 950 °C and 1000 °C under cyclic conditions in the air for 96 h. Weight gain is measured discontinuously at different oxidation temperatures and times. Weight gain was used to determine the kinetics of oxidation. The oxidation kinetics of the alloys followed a parabolic rate law at 850 °C, 950 °C and 1000 °C. Field emission scanning electron microscopy/energy dispersive spectroscopic analysis (FESEM/EDAX) technique was used to analyze the oxidation products. X-Ray Diffraction (XRD) was used to analyze the formed scale. The protective oxides of Cr_2O_3, NiO, and $NiCr_2O_4$ were formed, which contributed to the better oxidation resistance. These oxide scales increased with time and temperature and they are compact, dense, distributed uniformly and adherent to the surface of Inconel 617 alloy.

1. Introduction

Ni-based superalloys are commonly used in the manufacture of aerofoil components such as blades and nozzle guide vanes that work in the hot sections of advanced gas turbine engines [1,2], due to their good high temperature mechanical properties. These superalloys do not own adequate oxidation resistance in their service environment, especially in damaged areas where the bare metal is exposed to the oxygen-containing atmosphere. Continual oxidation of the moving crack tip can speed up the crack propagation through the component, causing in shorter service life, mainly under thermal cycling conditions. Therefore, high oxidation is a main reason for the failure of hot-section turbine blades [3]. The superalloys have been developed to achieve oxidation resistance by utilizing the concept of selective oxidation. The selective oxidation processes are affected by a number of factors such as alloy composition, surface conditions, gas environment and cracking behavior of the oxide scale [4]. When a clean component is exposed to an oxygen rich gas, small impinging nuclei of all the thermodynamically stable oxides develop on the surface and coalesce rapidly to give a complete layer. During the initial or transient stage, the rate of oxidation is rapid, all the elements in the alloy oxidize and the amounts of various oxides in the layer are approximately proportional to the concentration of the elements in the alloy [5,6]. High temperature oxidation involves the oxidation of reactive elements, formation of oxide scales, and internal oxidation [7]. The knowledge of reaction kinetics and the nature of the surface scales formed through high temperature oxidation is important for evaluating the alloys for their use in high-temperature applications. Alloy 617 is considered the greatest favorable structural material because of its high temperature strength and oxidation resistance [8]. Several studies have been conducted for Alloy 617 to investigate the high temperature oxidation behaviors in different environments [9–16]. In those studies, the external Cr_2O_3 layer and the internal Al_2O_3 oxides were developed in all testing conditions due primarily to the high Cr (22 wt.%) and low Al (1.5 wt.%) contents of Alloy 617 [10–14]. In addition the oxidation resistance and the stability of the surface oxide layer depend on the interplay between temperatures, alloy composition, thermal cycling and oxidizing environment. On the other hand, it is difficult to clarify the oxidation mechanisms of alloy 617 due to its complex chemical composition [12–16].

Degradation by oxidation is one of the main failure modes of hot section components in gas turbines, so an understanding of the oxidation resistance is very necessary for superalloys [17]. In the present investigation, the oxidation behavior of Ni-based superalloy (Inconel 617) has been studied various temperatures under

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cyclic conditions which have been chosen similar to actual service conditions. This will be achieved by cyclic oxidation process, calculating the kinetics of oxidation, Examination, studying the oxide layer by SEM, EDX and XRD and calculating the activation energy of oxidation.

2. Experimental work

2.1. Preparation of specimens

The alloy used during this study is Ni-based superalloy (Inconel 617). The nominal composition of the studied alloy is presented in Table 1. Specimens have been cut in the form of circular discs with dimensions 9.7 in diameter and 2 mm thickness. The specimens were mirror polished down to alumina using emery papers of 800, 1000 and 1200 grit sizes followed by cloth polishing using alumina powder (1 μm), then samples have been washed with distilled water and further cleaned with acetone.

2.2. Oxidation processes

The cyclic oxidation processes were done for the studied alloy Ni-based superalloy Inconel 617 at various temperatures 750 °C, 850, 950 °C and 1000 °C as shown in Table 2. The cyclic oxidation processes were done in a muffle furnace (VECSTAR Muffle Furnace) up 96 h. Every cycle of oxidation consisted of heating the furnace to the temperature of oxidation, then 6 h heating at the temperature of oxidation followed by 20 min furnace cooling. Weight of samples was taken after every cycle using digital weighing balance of 0.01 mg (Citizen CX 165) accuracy.

2.3. Surface characterization methods

X-ray Diffraction (XRD) investigations were carried out on the surface of the oxidized specimens by Philips X-ray Diffraction equipment to analyze the formed scale. The morphology was investigated by (Quanta 250 FEG) Scanning Electron Microscopy (SEM) and its Energy Dispersive X-Ray analysis (EDX) tool was used to study the chemical composition of the oxide layers, as each element has a specific atomic structure allowing X-ray wavelengths characteristic for each element to be identified separately. The second electron mode was used for imaging as well as the back-scattered mode for compositional contrast imaging. EDX is based on the analysis of X-ray emission spectra obtained after hitting the sample with an electron beam.

3. Results and discussion

3.1. Cyclic oxidation

Fig. 1 shows the mass change per unit area as a function of oxidation time at 750, 850, 950, and 1000 °C in air for the studied alloy. The total weight gain at the end of 16 cycles for the alloy at 750, 850, 950, and 1000 °C has been found to be 0.091, 0.3, 0.77, 1.37 mg/cm^2, respectively. It can be concluded from the plots that the total weight gain after oxidation at 1000 °C is the highest. It was noticed that the weight gain after oxidation at 1000 °C is more than double in general, and is also double of that at 950 °C, so the weight gain occurred after 200 °C oxidation in the range of 750 °C–950 °C is nearly equal to that occurred within 50 °C oxidation from 950 °C to 1000 °C. The weight gain after 850 °C is nearly quarter of occurred after 1000 °C. The increase of temperature increases the weight gain and the mass change is the sum of the mass gain by oxidation of alloying elements [12]. At high temperatures, oxidation was accelerated compared to lower temperatures. As well known, external and internal oxidation of Cr, Ni, Al, and Ti could be the main reason for the weight gains increased as time and temperature increased [13,16].

Table 2
Oxidation processes procedure.

Technique	Temperature	No. of specimens	Time periods	No. of cycles
1st technique	750 °C	1	Up 96 h	16
	950 °C	1	Up 96 h	16
	1000 °C	1	Up 96 h	16
2nd technique	850 °C	8	30 min.	–
			1 h	–
			6 h	1
			12 h	2
			24 h	4
			48 h	8
			72 h	12
			96 h	16

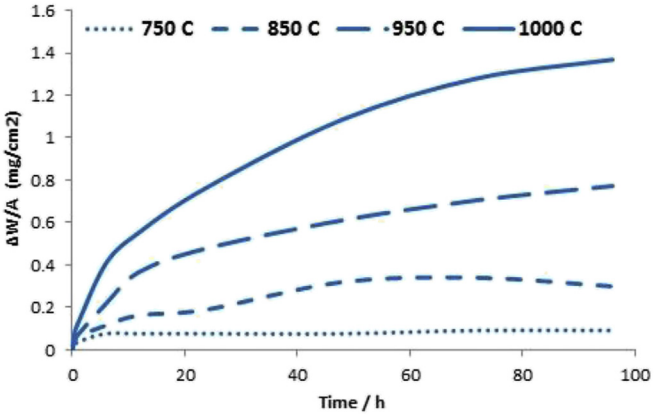

Fig. 1. Weight gain/Surface area (mg/cm^2) versus time of oxidation (h) for Inconel 617 alloy subjected to cyclic oxidation for 96 h in air at 750 °C, 850 °C, 950 °C and 1000 °C.

The oxidation process has nearly followed the parabolic rate law for the studied temperatures 850, 950, and 1000 °C.While it obeys subparabolic rate at 750 °C. Parabolic oxidation occurs when the metal or oxygen species diffuse through the growing oxidation layer. As the thickness of the oxidation layer increases, the diffusion path of the species also increases [13]. The values of parabolic rate constants (*Kp*) have been calculated for all the investigated cases, which are based on the parabolic rate equation for the high temperature oxidation process [18,19] given by

$$x^2 = Kpt$$

where x is the weight change per unit surface area, t the time and

Table 1
Nominal composition of Inconel 617 superalloys.

Alloy	Chemical Composition wt %										
	Ni	Cr	Fe	Co	Mo	Al	C	Mn	Si	Ti	Cu
Inconel 617	53.94	21.68	1.31	11.53	9.3	1.2	0.08	0.06	0.5	0.35	0.05