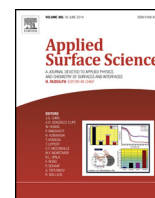




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Cyclic oxidation kinetics and oxide scale morphologies developed on alloy 617

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ABSTRACT

In this paper, an attempt was done to investigate the cyclic oxidation behaviour of alloy 617, cyclic oxidation tests were carried out in laboratory air at 750, 850, and 950 °C up to 12 cycles (14 h/cycle). The oxidation behaviour of alloy 617 approximately followed the parabolic rate law and the average activation energy is about 206 kJ/mol. At lower temperatures, a two-stage oxidation kinetics were observed and the transition time decreased as the oxidation temperature increased. SEM observations indicated that continuous and relatively irregular oxide layers were formed that had a surface nodular-type structure thickening with temperatures. XRD-patterns and SEM-EDS analysis revealed that the oxide scales were mainly composed of Cr₂O₃ scale mixed with minor amounts of MnCr₂O₄. Other oxides were detected such as NiO, TiO₂ and MnTiO₃. Also, the geometrical-irregularities and Ni-metallic inclusions were detected at the oxide/alloy interface. Moreover, Aluminium was internally oxidized to form Al₂O₃ as elongated particles, which were grown along grain boundaries via branch-like growth. The internal oxidation depth was increased as the temperature increased.

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1. Introduction

With regard to the broad high-temperature applications, a set of solid solution hardening Ni-based superalloys are utilized for steam generator (SG) and intermediate heat exchanger (IHXs) components. As well known, SG would operate with reactor outlet temperatures from 750 to 800 °C, while IHXs would operate up to an outlet temperature of 950 °C [1]. Among solid solution hardening Ni-based superalloys, alloy 617 has been selected as a potential candidate alloy for SG and IHXs components because of its excellent combination of high-temperature strength and good oxidation resistance [2–4]. Currently available chromia-forming alloys exhibit excellent oxidation resistance at temperatures lower than 1000 °C. This is due to their rely on the formation of continuous and coherent protective Cr₂O₃ scale, which requires a sufficient of Cr-content and higher Cr-diffusion supply for long service life. Moreover, the service life of the chromia-forming alloys is considerably reduced by the increase in Cr-depletion depth, presence of Cr-oxide volatilization and under thermal-cycling conditions [5–7].

The good oxidation resistance of alloy 617 is derived from its high Cr-content as well as Al is added primarily to improve the

high-temperature oxidation resistance by partitioning into and stabilizing the surface oxides [8,9]. 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. Consequently, a brief review of the previous findings on a similar model Ni–Cr–Al ternary system is necessary to understand the oxidation behaviour of alloy 617. Ni–Cr–Al ternary system had been extensively studied [10–20], which reported that the oxide scale structures and oxidation mechanisms of Ni–Cr–Al alloys can be classified into:

- Group-I has a low Cr < 5 wt.% and low Al < 2 wt.% contents that leads to the formation of a less-protecting NiO scale and internal oxidation of Al and Cr.
- Group-II with high Cr > 15 wt.% and low Al < 2 wt.% contents, forms an external layer of Cr₂O₃ and the internal oxidation of Al takes place to form Al₂O₃.
- Group-III has a low Cr < 15 wt.% and high Al > 7 wt.% contents, which develops a continuous external layer of Al₂O₃.

Christ et al. [21,22] showed that there is no significant effect of Mo and Co on the oxidation behaviour of alloy 617 and hence, it can be treated as the Ni–Cr–Al alloys group-II. The role of alloying

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Table 1
 Nominal chemical composition of alloy 617.

Ni	Cr	Si	Al	Fe	Co	Mo	C	Ti	Mn
Bal.	22.1	0.1	0.96	0.95	11–14	8.5–10	0.08	0–0.2	0.03–0.3

elements such as Ti and Mn on oxidation behaviour of alloy 617 must be considered. Several considerable studies have been carried out to evaluate the oxidation behaviour of alloy 617 in air, water vapor, and helium environments [23–42]. In general, Jang et al. [35] showed that compared to air environment, the oxidation rate of alloy 617 was not significantly affected in helium and steam environments. On the other hand, the oxide morphology and structure of alloy 617 were strongly affected by the environments. They have shown that the oxidation kinetics followed a parabolic oxidation rate law that is related to the growth of external oxide scales as well as to internal oxidation for all of the above environments [21–42]. The oxide scales developed on alloy 617 specimens oxidizing in air are mostly composed of Cr-oxide scales with NiO and/or NiCr₂O₄ additions [35,36,41,42]. While, the oxidizing of alloy 617 in helium environment resulted in an external oxide scale that was mostly composed of Cr₂O₃ mixed with less amounts of MnCr₂O₄ and TiO₂ isolated grains [26–28,31,35]. In addition, isolated nodular MnTiO₃ oxides were formed on the MnCr₂O₄/Cr₂O₃ and sub-layer Cr₂O₃ oxide was formed in steam and steam + 20 vol.% H₂ [36,40].

The evolution of cyclic oxidation kinetics and oxide scales developed on the alloy 617 at high temperatures has not been clearly identified. Also, the effect of temperatures and the role of alloying elements also has not been properly investigated. So, the present work is a part of the attempt to understand the oxidation behaviour of alloy 617 in air at 750, 850, and 950 °C for up to 12 cycles (168 h).

2. Experimental procedure

The alloy 617 was used in the as-received conditions. Its nominal chemical composition is shown in Table 1.

Fig. 1a illustrates the as-received microstructure that was observed with an optical microscope after chemical etching. The optical image of the as-received alloy 617 showed a polycrystalline microstructure and is typical austenite with many annealing twins and precipitation of titanium nitrides and carbides preferentially located at the grain boundaries and inside the matrix-γ grains [43], and an average grain size of 55 μm was calculated using image-J as shown in Fig. 1b.

The sheet of alloy 617 was cut into square coupons with dimensions of 20 × 20 mm² and 1 mm in thickness. The square coupons were polished with SiC paper up to the 1200 polishing grade, then ultrasonically cleaned and dried.

Cyclic oxidation tests were carried out in laboratory air up to 12 cycles. Each cycle was composed of heating (5 °C/min) to the

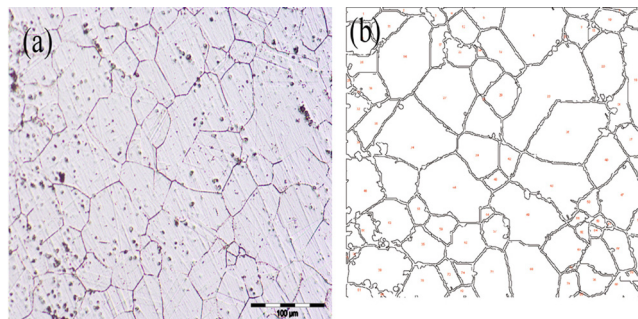


Fig. 1. (a) Surface microstructure optical image of as-received alloy 617 and (b) Image-J statistical result.

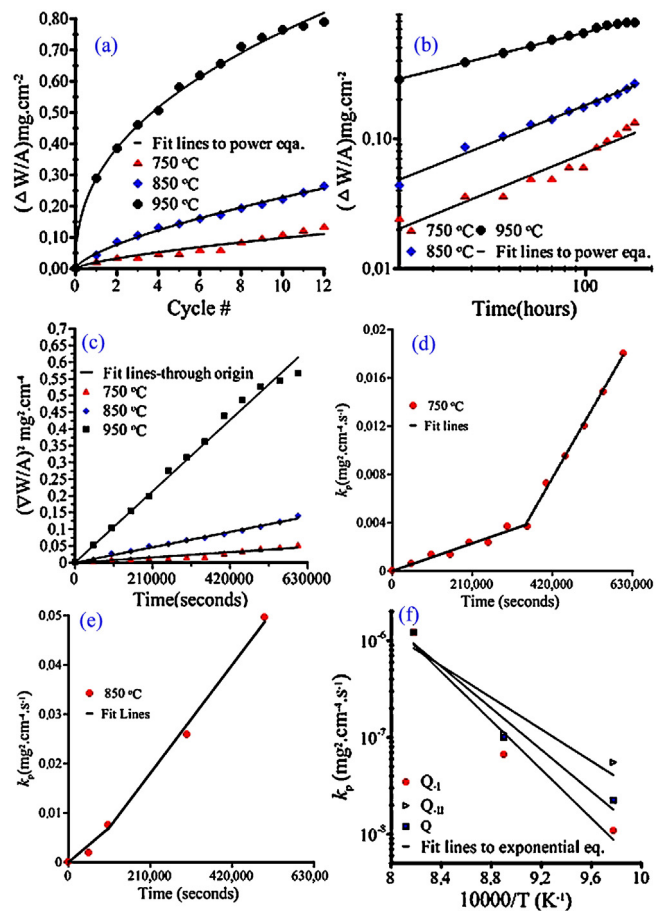


Fig. 2. Kinetic curves of alloy 617 samples oxidized in air up to 12 cycles: (a) Linear plots, (b) Log-log plots, (c) square of weight gain vs time in seconds, (d) and (e) two-stage oxidation at 750 and 850 °C, and (f) k_p vs T^{-1} .

desired temperature, then 14 h holding at the desired temperature, followed by furnace cooling (5 °C/min) with a subsequent weight measurements using electronic balance with a measurable sensitivity of 0.1 mg. The characteristics of the oxide scales were investigated by X-ray diffraction (XRD), scanning electron microscopy in combination with energy-dispersive X-ray spectroscopy (SEM-EDS) techniques. XRD was performed using Cu K_α (λ = 1.5418 Å) at 2θ° scale ranging from 16 to 90° and scanning rate of 5 s/step (step 0.03°).

3. Oxidation results

The details on the cyclic oxidation kinetics and oxide scale microstructural observations are discussed in the following sections.

3.1. Cyclic oxidation kinetic results

The cyclic oxidation kinetics curves of alloy 617 have been illustrated in Fig. 2. Fig. 2a represents the kinetics curves of weight gains (Δw/A) in mg cm⁻² vs the number of cycles. It shows that a longer time was needed to detect a significant change in the weight gain at 750 °C, but a quite identical behaviour with significant increase in weight gain occurred at 850 °C. At 950 °C, oxidation was accelerated compared to lower temperatures. As well known, external and internal oxidation of Cr, Ni, Al, Mn and Ti could be the main reason for the weight gains increased as time and temperature increased [9,44]. As shown in Fig. 2b, the slopes in log–log scale

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