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Stress assisted oxidative failure of Inconel 601 for thermal energy storage

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ABSTRACT

Degradation of an Inconel 601 superalloy subjected to a eutectic mixture of carbonate salt at 450 °C was studied using advanced microscopy and microanalysis techniques. On the outside of the vessel, long term exposure to elevated temperature led to intergranular stress corrosion cracking via a stress assisted oxidation process. Elemental analysis verified diffusion controlled de-alloying of Ni and Cr from grain boundaries. Transmission electron microscopy showed that a $(CrFeNiAl)_3O_4$ oxide phase was formed at grain boundaries as a result of oxygen penetration. Intergranular cracking appeared due to the presence of residual stresses and micro-structural heterogeneities facilitated the crack branching.

1. Introduction

Thermal Energy Storage (TES) points to a technology comprised of a storage medium, heat transfer mechanism and a containment material used in Concentrating Solar Thermal Power (CSTP) technologies [1–6]. CSTP uses a combination of mirrors to concentrate the sun's light energy onto a receiver which captures the energy and converts it into heat to generate electrical power [1,7]. Some TES systems employ Phase Change Materials (PCM) which typically undergo a physical phase change from solid to liquid and vice versa [2,6]. The most commonly used candidates for containment materials in TES systems are usually carbon or stainless steels and nickel alloys while chlorides, nitrites, carbonates and fluorides are favourite candidates for storage medium [6,8–13]. The compatibility of containment material with storage medium and its stability during repeated thermal cycling of a CSTP plant is a concerning issue [4].

Superalloys are a large number of alloys that exhibit certain characteristics like excellent surface stability, high resistance to corrosion and oxidation, resistance to thermal creep, high mechanical strength and good performance at elevated temperatures [14,15]. Nickel-base superalloys are ideal construction materials for high temperature applications as they combine high strength and good corrosion resistance at elevated temperatures [14,16]. Inconel 601 (IN601/alloy 601) is a general-purpose structural engineering alloy mostly used in heat and corrosive applications owing to the existence of nickel, chromium and minor aluminum content which enhances its oxidation resistance [17]. During exposure to elevated temperatures, these elements form a protective and adherent oxide film on the material surface [18].

However, a common issue in Ni-base alloys which threatens the corrosion resistance at high temperatures is the precipitation of continuous grain boundary (GB) carbides. It is known that heating of these alloys induces sensitization and makes the material susceptible to intergranular corrosion [16,19]. A correlation between microstructural evolution and the corrosion intensity of Inconel 601 in sulphuric acid has been reported previously with the greatest intergranular corrosion arising where an active grain growth followed by secondary recrystallization occurs [16]. Scarberry et al. [20] reported a strongly carbon dependent precipitation process of M_7C_3 type carbides along GBs at 700 °C in Inconel 600. M_7C_3 carbides were mostly reported in lower Cr content (about 15%) alloys while in larger Cr content the more thermodynamically stable carbides of $M_{23}C_6$ type are more common [21,22]. Smooth surfaces separated from GBs weakened by fine and dense GB carbides were reported elsewhere in which separation was attributed to the de-cohesion of carbide/matrix interface [22]. Transmission electron microscopy (TEM) investigations by Kaoumi et al. [23] recently showed that the evolution of precipitates (i.e. the dissolution of large M_6C carbides and re-precipitation/accumulation of small chromium-rich $M_{23}C_6$ precipitates) accompanied by the interaction of dislocations in combination with the formation of dislocation structures and serrated sub-grain boundaries are critical factors controlling tensile behavior of Inconel 617 at temperatures above 600 °C. Results predict that, at higher temperatures, recrystallization may dominate the deformation behavior of the alloy.

Another factor threatening Ni-base superalloys at elevated temperatures is the presence of an $L1_2$-type intermetallic compound (IMC) known as Ni_3Al. This IMC is a major strengthening phase with excellent characteristics at elevated temperatures. While the single-

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crystal Ni_3Al is ductile, the polycrystalline Ni_3Al is extremely brittle even at high temperatures [24]. Brittle intergranular failure, due to the penetration of oxygen through grain boundaries, has been well studied previously for Inconel 718 at elevated temperatures [25–29]. Pang et al. [28] reported the deposition of an oxide layer enriched in Cr, Al and Nb on the surface and segregation of Nb to grain boundaries as driving force for creep cracking of the alloy at elevated temperatures. Other studies [30] suggest a stress assisted grain boundary oxidation (SAGBO) mechanism in which the diffusion of oxygen through grain boundaries enhances the magnitude and intensity of the environmental failure directly proportional to stress intensity factor (SIF), oxygen partial pressure and temperature. The formation of γ' and γ" at 600 °C was reported by Rezende et al. [31] resulting in the precipitation hardening of alloy 718. However, the authors have not reported any evidence regarding detrimental influence of the precipitates on the material ductility. Lu et al. [32] attributed fatigue crack growth rate of HASTELLOY X at 816 °C and 927 °C to the stress intensity factor whereas a transition from transgranular to intergranular crack growth indicated the creep and oxidation effects on the crack behavior.

The selection of appropriate and optimum structural material as a thermal energy storage vessel, subject to corrosive atmospheres and high temperatures, is essential in developing the economic and functional efficiency of a TES system. Recently, grain boundary engineering efforts have established noteworthy developments in studying the stress corrosion cracking (SCC) behavior of Ni-base superalloys; as an illustration, the impact of special boundaries on the sensitivity to oxygen-induced intergranular brittle fracture (dynamic embrittlement) of IN718 at 650 °C in air has been studied [33].

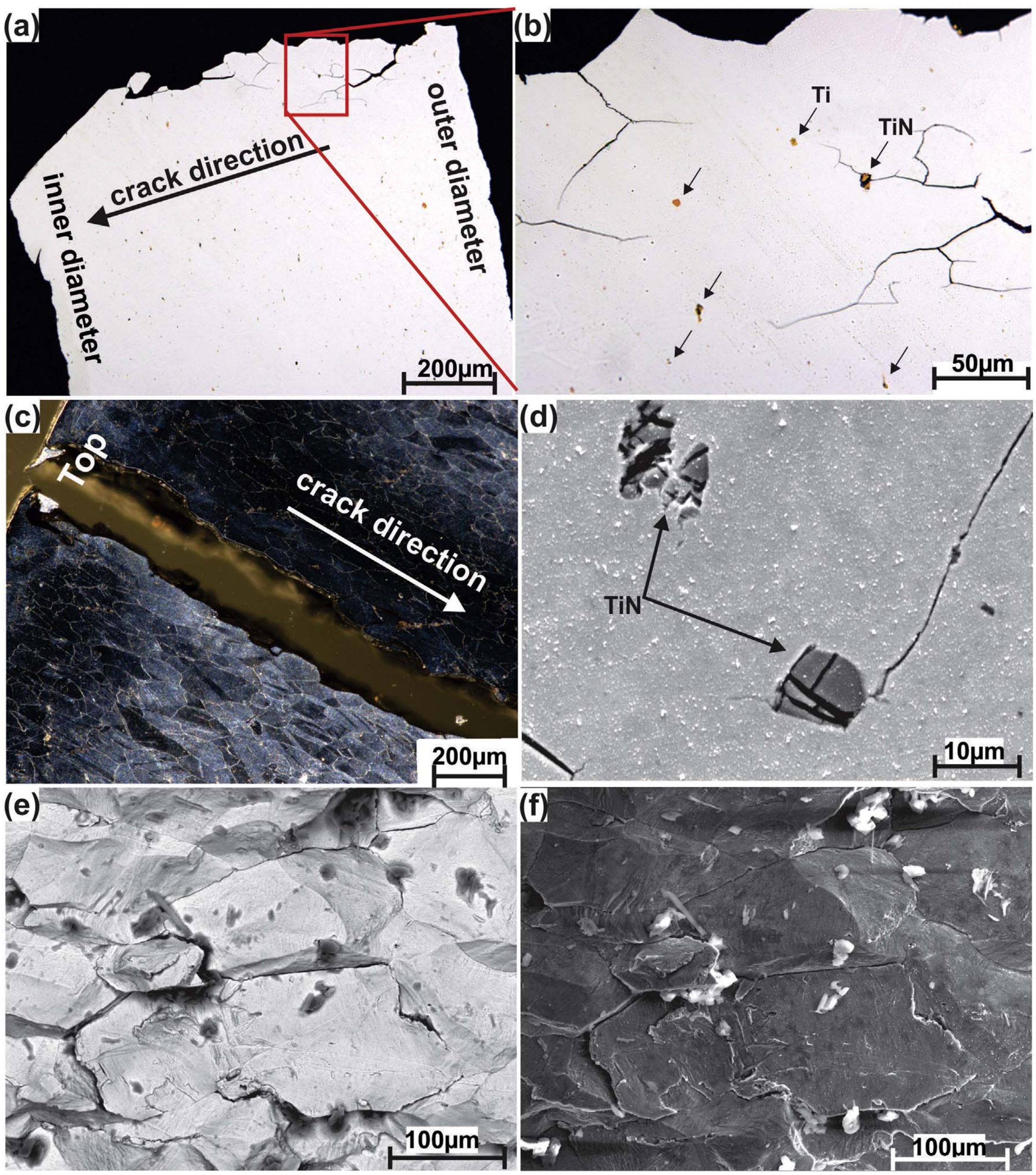

Fig. 1. (a) Top view of the crucible sidewall showing outer and inner diameters and crack growth direction across the crucible thickness (*un*-etched sample), (b) greater magnification of the red rectangular area in (a), (c) optical microscopy image showing crack initiation area from top of the crucible, (d) SEM image showing dispersion of Ti-rich particles in the matrix, (e) SEM secondary electron, and (f) SEM backscattered electron images of the same fracture surface showing the intergranular morphology of crack and salt residue (dark and light particles in (e) and (f), respectively).