



Corrosion of Inconel 601 in molten salts for thermal energy storage



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ABSTRACT

Inconel 601 superalloy was examined for compatibility with the eutectic mixtures of NaCl + Na₂SO₄ and NaCl + Na₂CO₃ at 700 °C and Li₂CO₃ + K₂CO₃ + Na₂CO₃ at 450 °C in air for thermal energy storage. Electrochemical measurements combined with advanced microscopy and microanalysis techniques were employed. Oxidation was the primary attack leading to the development of oxide deposits on the alloy surface. The deposit was not stable in NaCl + Na₂CO₃ because of the fluxing action and high solubility of chromium in the salt leading to very high corrosion current density values. De-alloying threatened the material at 700 °C while the availability of oxygen controlled its rate. The availability of oxygen was seen to limit the corrosion rate in NaCl + Na₂SO₄ and NaCl + Na₂CO₃. De-alloying also appeared as pits on the metal surface at 700 °C. For the metal in contact with NaCl + Na₂SO₄ an additional layer attacked by sulfur was found under the pitting corroded layer. The attack morphology on the metal surface in contact with Li₂CO₃ + K₂CO₃ + Na₂CO₃ at 450 °C was found to be uniform.

1. Introduction

Nickel-base superalloys are well-known for their excellent performance at elevated temperatures like outstanding surface stability, high mechanical strength and resistance to corrosion, oxidation and thermal creep [1,2]. Inconel 601 (IN601/alloy 601) is an ideal superalloy for heat and corrosive applications owing to its enhanced oxidation resistance. This is because of high chromium and minor aluminum content in the alloy composition that leads to the formation of a protective and adherent oxide scale on the material surface [3,4]. Such capabilities make the alloy a prime candidate as containment material for Thermal Energy Storage (TES) [5–11].

Consisting of a storage medium as Phase Change Material (PCM) retained in a containment material, TES suffers from hot corrosion and high temperature oxidation in molten salt environments [11,12]. Since most metals tend to oxidize over a wide range of conditions at elevated temperatures, oxidation rate and its morphology are significant factors to estimate the material lifetime. Superalloys have been found susceptible to oxidation because of sensitization resulting from heating and/or stress [13–16]. The presence of intermetallic compounds (IMCs) also threatens these alloys at high temperatures [17]. Brittle intergranular failure, due to the penetration of oxygen through grain boundaries besides the existence of residual stresses in the lattice, has been previously reported for Inconel alloys at high temperatures [11,18–21].

Investigations on several Ni-based alloys and 316 stainless steel in molten KCl-MgCl₂ eutectic salt at 850 °C in a quartz container confirmed grain boundary corrosion attack accompanied by chromium

dissolution into the molten salt for most of the studied alloys [22]. Results showed an improvement in corrosion resistance by increasing the nickel content of the alloy and decreasing chromium. Reports point the finger of suspicion at grain boundary carbides that accelerate intergranular attack. Accordingly, lowering carbon content and reduction of grain size could be helpful. Sridharan et al. [23] argue that surface Cr-carbides are directly related to pronounced grain boundary attack so that alloys containing higher Cr content suffer from more corrosion attack when carbon content is also high. Another examination on iron, nickel and cobalt based alloys in a eutectic sodium-potassium-magnesium chloride salt at 900 °C showed it to be aggressive towards grain boundaries in the range of temperature for solar thermal energy systems [22].

Other studies on Inconel 600 in contact with molten fluoride salts showed the formation of uniformly distributed voids in the microstructure of corroded material [23]. Authors believe that chromium diffusion out into the salt causing the chromium from the interior of the alloy to diffuse down the concentration gradient to the surface. Such a substitutional diffusion accompanied by a vacancy migration process can coalesce to form voids. Cr depletion followed by the oxidation of chromium and formation of soluble chromate ions in the liquid has been reported elsewhere for stainless steels and nickel-base alloys in molten nitrates [10,24].

However, research shows that alloying elements like Cr, Al and Si contribute to the development of self-healing protective oxide films on the alloy surface which act as diffusion barriers against further oxidation [23,25]. An early study by Pang et al. [20] showed the deposition

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Table 1
Nominal elemental composition and crystal structure of the IN601 superalloy (wt%).

Alloy	Structure	Ni	Fe	Cr	C	Mn	Si	Cu	S	Al
IN601	fcc	Bal	15	22	0.1	1	0.5	1	0.015	1.7

of an oxide layer enriched in Cr, Al and Nb on the surface of an Inconel alloy at elevated temperatures. The authors' recent study [16] also confirms the development of oxide films on the surface of IN601 in contact with a molten carbonate salt at 450 °C.

The compatibility of containment material with molten salt and its stability is a concerning issue in TES systems [8]. The selection of appropriate and optimum structural materials is essential in developing economic and functionally efficient systems. This study will examine the corrosion behavior of IN601 superalloy in three eutectic mixtures of molten salts (as PCM) for the next generation of TES applications.

2. Experimental procedure

IN601 superalloy was examined in eutectic mixtures of molten chloride sulfate, chloride carbonate and ternary carbonate salts. Table 1 summarizes the structure and nominal composition of the alloy used in the current study.

Sodium chloride AR (CAS no. 7647-14-5), sodium sulfate AR anhydrous powder (CAS no. 7757-82-6), sodium carbonate anhydrous LR (CAS no. 497-19-8), potassium carbonate anhydrous LR (CAS no. 584-08-7) and lithium carbonate 99% (CAS no. 554-13-2) were placed for 24 h in a 180 °C furnace to dry and then were measured and mixed according to Table 2. The eutectic mixtures melting points and test temperatures are also provided in the table. Test temperature for each salt was selected close to its melting point based on the assumption that the salt will be used as a PCM in a TES system. Hereinafter, eutectic mixtures will be referred to as chloride sulfate, chloride carbonate and ternary carbonate, according to Table 2.

2.1. Electrochemical corrosion investigation

Electrochemical experiments were conducted using a three-electrode cell containing the molten salts in alumina crucibles open to air at 700 °C and 450 °C in a preheated cylindrical furnace. Test coupons of 25 mm long, 5 mm wide and 2 mm thick were mechanically wet ground and polished down to 0.04 μm by colloidal silica, washed with ethanol and dried in air. Measurements were implemented by means of a VMP3-based BioLogic instrument controlled by EC-Lab® software. The three-electrode cell was implemented with the polished sample as the working electrode and two similarly sized platinum sheets (25 × 5 × 1 mm) as pseudo reference and counter electrodes [26–29]. Samples were subjected to open circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) measurements. Equilibrations of potentials (OCP) were carried out for 1 h immediately after exposure. EIS measurements were then obtained using a frequency range of 100 kHz to 100 MHz with the amplitude

Table 2
Chemical composition, melting point and test temperatures of salt mixtures.

Eutectic mixture	Composition (wt%)	Melting point (°C)	Test temperature (°C)
Chloride sulfate	26.5NaCl + 73.5Na ₂ SO ₄	626	700
Chloride carbonate	40NaCl + 60Na ₂ CO ₃	632	700
Ternary carbonate	33.4Na ₂ CO ₃ + 32.1Li ₂ CO ₃ + 34.5K ₂ CO ₃	397	450

of ± 10 mV. Finally, PDP was conducted at the potential scan rate of 10 mV/min and potential range of ± 300 mV with respect to the open circuit potential. ZFit analysis of EC-Lab software was used to fit successive impedance cycles.

2.2. Static corrosion

Fresh metal coupons were cut to around 25 × 7 × 2 mm for static corrosion tests while the front sides were mechanically wet polished down to 1 μm in colloidal silica using standard grinding and polishing procedures. A schematic representation of the test condition and alignment of samples in the furnace have been already shown [30]. Cylindrical alumina crucibles were used as salt vessels and the furnace temperature was set to 700 ± 10 °C for binary salts and 450 ± 10 °C for the ternary carbonate.

The salt containing vessels were placed into the furnace at room temperature and then gradually heated up to the test temperature. Once the salt melted and the chamber conditions stabilized, the metal coupons were immersed so that the top half was exposed to the air and bottom half submerged into the molten salt. Such a configuration enabled us to make a comparison not only between the impact of the salt and that of the oxygen from the environment, but also the decomposition gases of the salt which are expected to increase the corrosive impact in the atmosphere. One group of metal coupons were removed after 1 h and the other after 120 h of exposure. All coupons were then mounted exposing the side indicated previously [30] into a conductive resin, ground and polished down to 0.04 μm from the side of the sample in colloidal silica using standard procedures, washed with ethanol and finally dried in air. All microscopy analyses were conducted on top ends and bottom ends of the samples. Another group of metal coupons sized 7 × 7 × 2 mm were submerged into the molten salt and removed after 750 h exposure. Salt analysis was conducted for the latter group to study the dissolution tendency of the metal alloying elements in the salts.

2.3. Macro and micro-structural investigations

2.3.1. Optical microscopy analysis

Optical microscopes model Leica DMi8A (magnification 1.25 ×–50 ×) and Leica M125 (magnification 0.8 ×–10 ×) both equipped with Leica Application Suite software were used to take macro and micro-images for microstructural and corrosion attack observations.

2.3.2. SEM and EDS analysis

Complimentary techniques like scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) were employed for further microstructural investigations using a field emission SEM machine (model: JEOL 7001F, with automated feature detection equipped with secondary electron and EDS analysis system).

3. Results and discussion

3.1. Electrochemistry

OCP and PDP graphs of the alloy specimens in the studied molten salts are shown in Fig. 1. IN601 seems to gain nobler values over time which could be attributed to the development of protective films on the surface. The alloy stabilizes at potential values around – 400 mV after 1 h in all molten salts. It should be noted that, as the salts compositions and temperatures are different, corrosion mechanisms might not be similar. Therefore, any comparison here is just an indication of the alloy behavior among the studied salts regardless of the mechanisms involved.

E_{corr} values in PDP plots in Fig. 1b reflect OCP values in Fig. 1a. Data extracted from PDP curves are summarized in Table 3. IN601 shows the lowest corrosion current density value in the ternary carbonate at

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