



Corrosion behavior of Ni-based alloys in molten NaCl-CaCl₂-MgCl₂ eutectic salt for concentrating solar power



Bo Liu^a, Xiaolan Wei^a, Weilong Wang^{b,*}, Jianfeng Lu^b, Jing Ding^b

^a School of Chemistry and Chemical Engineering, South China University of Technology, Guangzhou 510640, PR China

^b School of Engineering, Sun Yat-sen University, Guangzhou High Education Mega Center, Guangzhou 510006, PR China

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ABSTRACT

In this work, the corrosion behavior of Ni-based alloys including Inconel 625(In625), Hastelloy X(H X) and Hastelloy B-3(H B-3) alloys were investigated in a molten salt NaCl-CaCl₂-MgCl₂ by gravimetric methods. The corrosion test was conducted at 600 °C for a maximum immersion time of 3 weeks. The corrosion behavior was determined by measuring the mass loss of samples at different time intervals. The corrosion products and morphology were investigated in detail via X-ray diffraction (XRD) and scanning electron microscope (SEM). The microscopic area element analysis in corrosion surface and the mapping diagram of element distribution from corrosion profile were carried out by energy dispersive spectroscopy (EDS). The results show that In625 alloy resists corrosion better than H X and H B-3 alloys in molten salt NaCl-CaCl₂-MgCl₂ in the presence of air. The corrosion profile in In625 and H X alloy show a poor Cr zone in the inner corrosion layer, probably caused through the outward diffusion of CrCl₄(g) which is forecasted the formation by thermodynamic calculation. MgCr₂O₄ attached to the In625 and H X alloys specimens surface formed a compact protective layer and thus resist corrosion well. Thin and uniform corrosion layer was observed from profile in H B-3 alloy after immersed in NaCl-CaCl₂-MgCl₂ molten salt when air was present, however there is not a poor Cr zone in corrosion profile of H B-3 alloy. From above, the corrosion rate of Ni and Mo in alloy dominates the corrosion rate. Certain amount of Cr in the nickel-based alloys enhanced its corrosion resistance in molten salt NaCl-CaCl₂-MgCl₂, even if Ni and Mo in nickel-based alloys shows more stable performance in the corrosion test in chloride molten salt.

1. Introduction

Concentrating solar power (CSP) is an emerging sustainable technology for power generation. The sunlight is concentrated to heat the heat transfer fluid (HTF), which is then pumped to a heat exchanger to generate steam to drive steam turbines for power production. HTF is one of the most important components for overall performance and efficiency of the CSP system. Current CSP plants use nitrate molten salts as HTFs and can operate with multiple hours of energy storage capacity. Next-generation of solar power conversion system in CSP application are targeting high-temperature, advanced fluids to be operated at the range of 600–800 °C, which exceeds the stability temperature of nitrate [1]. At the higher operating temperatures achievable in solar power tower-type CSP plants, chloride salts are promising candidates for application as thermal energy storage materials, owing to their thermal stability and generally low cost [2]. Ionic metal chloride salts, such as NaCl, CaCl₂, MgCl₂, KCl are abundant in nature and boil at temperature higher than 1400 °C. As a class of potential high temperature heat transfer fluid, NaCl-CaCl₂-MgCl₂ eutectic system shows the advantages

of low cost, great heat storage temperature range, super heat of fusion, low viscosity and good thermal stability [3]. However, molten chlorite has a sharp corrosive action on container and piping alloys in the CSP system [4]. Corrosion is a major problem in the CSP system, especially under high temperature operating condition [2].

There are several reports available on corrosion issue of chloride salt mixtures. The corrosion behavior and mechanisms of pure nickel (Ni), GH4033 and GH4169 in 793 K molten eutectic NaCl-MgCl₂ were studied by Jun-wei Wang et al. [4]. They reported that the corrosion resistance of pure Ni is better than GH4033 and GH4169. The Cr and Fe elements in the alloys would preferential oxidization and chlorination. After comparing the corrosion of the alloys with different Cr content in the chloride salts, Manohar S. Sohail points out that increasing Cr content increased the rate of corrosion, while changes in nickel content seemed to have no effect [5]. Also, it has been reported that Cr is not an effective element for improving corrosion resistance of Fe-based and Ni-based alloys. Because Cr₂O₃ has a higher solubility than iron and nickel oxides, it will react more readily with chloride salt [6,7]. However, K. Vignarooban et al. [8] have studied the corrosion resistance of

* Corresponding author.

E-mail addresses: wlong@mail.sysu.edu.cn (W. Wang), dingjing@mail.sysu.edu.cn (J. Ding).

Hastelloys C-276, C-22 and N types in eutectic molten salts containing NaCl, KCl and ZnCl₂ and found that Hastelloys N showed higher corrosion rates than C-276 and C-22, even though Hastelloy N has higher nickel content. Hastelloy N is more susceptible for increased corrosion, because Hastelloy N has lower Cr content (7%) compared to C-276 and C-22. The effect of the elements on the corrosion resistance of the alloy remains to be studied. In addition, by using electrochemical method, Inconel 625 was reported as the most corrosion-resistant alloy in molten 34.42NaCl-65.58LiCl (wt%) at 650 °C, with a corrosion rate of 2.80 ± 0.38 mm/year in a nitrogen atmosphere than SS347, SS310, and Incoloy 800 H [1]. These alloys exhibited localized corrosion. In the presence of oxygen, the alloy forms oxides that eventually dissolve into the molten chloride salt liberating the oxygen component which then later is able to keep the oxidization process going. Intergranular corrosion is the major corrosion type found in alloys exposed to molten chlorides in oxidizing atmosphere [9]. Shunv Liu et al. [10] have studied the isothermal corrosion of TP347H, C22 alloy and laser-cladding C22 coating in molten alkali chloride salts at 450–750 °C. At lower test temperatures, chemical corrosion plays a dominated role. The corrosion mechanism involves accelerated oxidation of alloys by gaseous Cl/Cl-species, which is often referred to as active oxidation. At higher test temperatures, electrochemical corrosion plays a dominated role. The corrosion may be further enhanced if the protective surface oxide scale is dissolved by molten salts. Under this circumstance, accelerated corrosion happens for oxygen as well as other aggressive media can still access to the fresh alloy and diffuse in the alloy substrate.

It have been reported that the corrosion rate of the nickel-based alloys was found to increase with increasing partial pressure of oxygen [11]. Water vapor also could play a role in the corrosion process [2]. Atmospheric air plays a big role in inducing the corrosion in molten-salt/alloy systems [12]. Thus, in a non-inert environment, the effect of water vapor and oxygen in the air and molten salts on the corrosion of molten salt / alloy systems should be taken into account.

However, there are few reports available on corrosion issues of alloys in contact with molten eutectic NaCl-CaCl₂-MgCl₂. The corrosion mechanism is not clear yet. Corrosion characteristics of the elements in the alloys in the molten salt under low oxygen partial pressure also need to be studied.

Nickel-based alloys are widely used in industry under aggressive conditions owing to their perfect corrosion resistance [13–15]. Therefore, nickel-based alloys are frequently employed in corrosive applications. In this study, Inconel 625, Hastelloy X and Hastelloy B-3 alloys were chosen as research subjects.

The corrosion behaviors of Inconel 625, Hastelloy X and Hastelloy B-3 alloys in thermal storage medium of molten eutectic NaCl-CaCl₂-MgCl₂ has scarcely been reported in the literature. Therefore, this paper aims to investigate the corrosion of Inconel 625, Hastelloy X and Hastelloy B-3 alloys in eutectic NaCl-CaCl₂-MgCl₂ at 600 °C with low oxygen partial pressure. Corrosion behavior was investigated based on the changes in mass and analyses of chemical composition and microstructure of specimens following induced corrosion.

2. Experimental

2.1. Materials

The NaCl, MgCl₂ and CaCl₂ were purchased from Alfa Aesar for making the ternary eutectic mixture with a melting point of 425 °C for a composition of 53.43, 14.95 and 31.61 mol% [3]. All the salts used in this study were stored in inert atmosphere or under vacuum before loading them into the furnace where the salt mixture was heated above 200 °C within 30 min since there was little bulk water. Then the mixtures were heated to 600 °C, held for 3 h to ensure a homogeneous mixture and then cooled to ambient temperature. The solidified salt mixture was ground into power using mechanical rolling, sealed in

Table 1
Main composition of the samples (wt%).

Alloy	Cr	Fe	Ni	Mo	Mn	W	Si	Other
Inconel625	22.85	3.41	59.7	8.6	0.089	0.08	0.176	Nb + Ta 3.65
Hastelloy X	22	18	47	9	-	-	-	Co 1, W 0.5
Hastelloy B-3	1.5	1.5	65	28.5	0.595	3.00	0.01	Co ≤ 3.00

sample bags and kept in desiccators. These steps were carried out in an isolated air environment.

All the alloys used in this experiment were purchased from GoodFellow. The alloys composition is shown in Table 1. The specimens of Inconel 625(In625), Hastelloy X(H X) and Hastelloy B-3(H B-3) alloys in the gravimetric corrosion test were cut to the dimension of 30 mm × 15 mm × 2 mm. The specimens were polished by 400, 800 and 1200 grit emery paper in turn and then with diamond paste to obtain a mirror-like finish for corrosion testing, followed by washing and cleaning with water and acetone. After being dried, the sample dimensions were measured with an electronic caliber and weighed with an analytical balance with an accuracy of 0.00001 g.

2.2. Corrosion experimental procedure

The isothermal corrosion was carried out in air atmosphere using a muffle furnace at 600 °C. The specimens were completely immersed in 12 covered alumina crucibles, which contained the mixture NaCl-CaCl₂-MgCl₂. The 12 covered alumina crucibles was numbered 1–12. Among them, No.1 to No.4, No.6 to No.9 and No.11 crucibles was placed only one piece of alloy, three pieces of alloys were placed in No.5, No.10 and No.12 crucibles, respectively. Remove the crucible from the furnace every 1 day until No.10, and then remove crucibles No.11 and No.12 on days 14 and 21. After the immersion tests, the specimens were taken out of the muffle furnace and cooled together with the molten salt to room temperature to avoid oxidation. The post-corrosion treatment was carried out to one piece of alloy in alumina crucibles No.1 to No.12 according to the standard method described in ASTM G1-03 [16]. The mass loss and corrosion rate curves of the alloy was calculated.

The Eq.(1) for calculating the mass loss over time is [17]:

$$\frac{\Delta m}{S_0} = \frac{m_i - m_f}{S_0} \quad (1)$$

where m_i is the initial mass of the specimen in gram, m_f is the mass of this specimen at time t in gram, and S_0 is the initial area of the specimen in cm².

The corrosion rate (CR) values were calculated using the following equation [18], assuming uniform dissolution:

$$CR(\mu\text{m/y}) = (365) \cdot 10000[\Delta m/(\rho \cdot S \cdot T)] \quad (2)$$

where Δm is the weight loss in gram, ρ is density(g cm⁻³) of the alloy, S is the total immersed area in cm², T is the immersion duration in days.

After ultrasonic washing and drying, the other two pieces of alloys in the crucibles No.5, No.10 and No.21 would be used for XRD analysis and SEM observation.

2.3. Analysis method

The surface and cross section morphology of specimens after corrosion were observed by scanning electron microscope (SEM). Specimens were prepared for cross-sectional metallographic examination using standard grinding and polishing procedures. The specimens were coated with organic polymer materials to avoid damaging the corrosion products on the surface of the specimens during the sample preparation. Energy dispersive spectroscopy (EDS) was used to analyze the chemical composition of the specimens. The crystallographic structures of the corrosion products on the surfaces of the specimens

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