Research Article

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Corrosion behaviors of 316 stainless steel and Inconel 625 alloy in chloride molten salts for solar energy storage

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Abstract: Corrosion behaviors of 316 stainless steel (316 ss) and Inconel 625 alloy in molten NaCl–KCl–ZnCl₂ at 700°C and 900°C were investigated by immersion tests and electrochemical methods, including potentiodynamic polarization and electrochemical impedance spectroscopy. X-ray diffraction and scanning electron microscopy/energy dispersive spectroscopy were used to analyze the phases and microstructures of the corrosion products. Inconel 625 alloy and 316 ss exhibited high corrosion rates in molten chlorides, and the corrosion rates of these two alloys accelerated when the temperature increased from 700°C to 900°C. The results of the electrochemical tests showed that both alloys exhibited active corrosion in chloride molten salt, and the current density of 316 ss in chloride molten salt at 700°C was 2.756 mA/cm², which is about three times the value for Inconel 625 alloy; and the values of the charge transfer resistance (R_t) for Inconel 625 were larger than those for 316 ss. The corrosion of these two alloys is owing to the preferred oxidation of Cr in chloride molten salt, and the corrosion layer was mainly ZnCr₂O₄ which was loose and porous and showed poor adherence to metal.

Keywords: chloride molten salts, corrosion, electrochemical, Inconel 625 alloy, 316 stainless steel

1 Introduction

Chloride molten salts have been considered as the potential candidates for the heat transfer fluid and thermal energy storage (TES) for the next-generation concentrating solar power (CSP) plants, owing to their thermal stability, low cost, low melting point, high boiling point and good heat transfer property [1–4], e.g., NaCl and KCl (ionic chloride salts) are earth abundant and boil at temperatures higher than 1,400°C. When these are mixed with ZnCl₂ (low melting, covalent metal halides), the melting point of the mixture of chloride salts will reduce owing to the eutectic reaction between them. However, severe corrosion of the structural materials in the chloride molten salts is a big challenge for CSP technologies [3–5].

The corrosion resistance of different alloys in chloride molten salts has been widely studied [6–10], and these works focused on the effects of alloy elements on the corrosion rate of alloy in chloride molten salts. Ding et al. [11] presented an overview on the corrosion behaviors of alloys in chloride molten salts, and the corrosion mechanism of alloys in chloride molten salt was also studied in this review. It is well-known that the corrosion rate of alloys in chloride molten salts depends on the alloy composition [12], impurities in salts [3], salts' temperature and the gas environment [13].

The corrosion processes of alloys in chloride molten salts include chemical and electrochemical corrosion, interface reaction and oxidant solution [14]; however, the corrosion mechanism, especially the role of chlorine is still not clear. Activation-oxidation mechanism [15,16] or dissolution as anode-oxidizing–reduction–chlorination mechanism [17] has been used to explain the corrosion process of alloys in molten chloride salts. As mentioned above, the corrosion of alloys in chloride molten salts is an electrochemical process; thus, the electrochemical method is suitable to study the corrosion behaviors of alloys caused by chloride molten, which is

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helpful to understand the ion transition during the corrosion process.

The 316 ss and Ni-based Inconel 625 are the potential candidates for the next-generation CSP systems using chloride molten salts as the heat fluids and TES because of their good mechanical properties at high temperature [18]. It is known that 316 ss and Inconel 625 alloy demonstrated good corrosion resistance in nitrite molten salts [19]. However, as mentioned above, chloride molten salts are much more corrosive than nitrite molten salts. To the best of our knowledge, the corrosion mechanism of alloys in chloride molten salts is still not clear, and the corrosion data of these two alloys in molten chloride salts, especially in NaCl–KCl–ZnCl₂ system molten salts, are scattered. In the present work, to understand the corrosion mechanism and to evaluate the corrosion resistance of 316 ss and Inconel 625 alloy in chloride molten salts, the corrosion behaviors of these two alloys in molten NaCl–KCl–ZnCl₂ at 700°C and 900°C were investigated by immersion test and electrochemical methods. The scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) were applied to study the microstructural morphology and phase composition of the corrosion products.

2 Experimental

2.1 Materials and chloride salts

The present study used both 316 stainless steel (316 ss) and Inconel 625 alloy for testing, and their chemical compositions are listed in Tables 1 and 2. The specimen was cut into 30 mm × 5 mm × 5 mm by using an electric spark cutting machine, followed by grinding down to 200–1,000 grit SiC paper, ultrasonically degreased with acetone and then dried.

A ternary mixture of NaCl (13.4%) + KCl (33.7%) + ZnCl₂ (52.9%) (mol%) were used for studying corrosion. After drying at 200°C for 48 h, 500 g mixture of NaCl–KCl–ZnCl₂ was put into an alumina crucible. The salt mixture was further dried at 200°C in the reaction chamber under vacuum before corrosion tests.

Table 1: Composition of 316 stainless steel (wt%)

C	Si	Mn	P	S	Ni	Cr	Mo
0.03	1.0	2.0	0.045	0.03	12	17	2.5

Table 2: Main chemical composition of Inconel 625 alloy (wt%)

Elements	Ni	Cr	Mo	Nb + Ta	Fe
Content	61.2	22.25	9.65	3.75	3.15

2.2 Preparation of working electrode

A Fe–Cr wire was spot welded to one end of the specimen for electrical connection. The sample was then sealed in an alumina tube with high-temperature cement, with a length of 20 mm exposed. The cement was dried at room temperature for 24 h and then further solidified at 80°C and 150°C for 2 h, respectively. The exposed surfaces of the specimen were polished again with 1,000 grit SiC paper, rinsed and dried prior to electrochemical testing.

2.3 Corrosion tests

The immersion tests were performed in the muffle furnace at 700°C and 900°C, respectively. At the interval of every 10 h, three samples were taken out from the graphite crucible and ultrasonic cleaned in distilled water and ethanol after cooling to room temperature. The mass change in the samples was measured by an electronic analytical balance with an accuracy of 0.1 mg (TE124S).

The electrochemical tests were conducted in a stainless steel chamber under the protection of high-purity Ar, as described in ref. [20]. Electrochemical measurements including potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were conducted by electrochemical station (AutoLab). Potentiodynamic polarization was undertaken at a scan rate of 20 mV/min, using a conventional three-electrode system with a Pt wire electrode as the reference electrode, a Pt plate as the counter electrode and the prepared specimens as the working electrode. The EIS measurements were carried out at the open-circuit potential between 0.01 Hz and 100 kHz using a two-electrode system (two working electrodes). The amplitude of input sin wave voltage was 10 mV. The impedance spectra were calculated and fitted using ZSimpWin software.

2.4 Identification of the corrosion products

After corrosion test, the samples were examined by SEM coupled with an energy dispersive X-ray spectrometer

(EDX; Quanta 200) and XRD with a Cu radiation source (Shimadzu XRD-6100).

3 Results

3.1 Corrosion rates of 316 ss and Inconel 625 alloy in chloride molten salt

Figure 1 shows the corrosion rates of 316 ss and Inconel 625 alloy in ternary chloride molten salt at 700°C (Figure 1(a)) and 900°C (Figure 1(b)), respectively. All experimental values of mass change were averaged using the data of three parallelly tested alloys. After immersion testing, the corrosion rates were calculated based on the following equation [11]:

$$r_{\text{corr}} = \frac{8.76 \times 10^4 \times W}{S_1 \cdot t \cdot \rho} \quad (1)$$

where r_{corr} is the corrosion rates (mm/a), W is the mass loss of the specimen due to corrosion (g), S_1 is the specimen area (cm²), t is the immersion time of the specimen in molten salts (hour) and ρ is the specimen density (g/cm³).

The corrosion rates of 316 ss and Inconel 625 alloy increased when the testing temperature increased from 700°C to 900°C, which indicated that higher temperature promoted the corrosion of these two alloys in chloride molten salt. The weight loss of the samples is linear with corrosion time, which indicated that the oxide scales formed on the surface are non-protective. Additionally, 316 ss demonstrated severe corrosion compared to Inconel 625 alloy.

3.2 Morphologies of the corrosion products

3.2.1 Morphologies of the corrosion products formed on 316 ss

Figure 2 shows the surface SEM images of the 316 ss after exposure to molten NaCl–KCl–ZnCl₂ at 700°C (a,b,c) and 900°C (d,e,f) for 60 h. The oxide scales were non-homogeneous, as seen in Figure 2(a) and (d), and cracking and spalling occurred during the formation of the oxide scales. Figure 2(c) and (f) is the magnified image of the spalled area located in Figure 2(b) and (d), respectively; the oxide scales beneath the spalled part were loose. Polyhedral-shaped crystals can also be seen in Figure 2(b) and (e), where the EDS analysis showed that the main elements in these polyhedral-shaped crystals were Cr, Zn and O.

Figure 3 shows the cross-section and element distribution along the yellow line in the images of 316 ss after corrosion at 700°C and 900°C in chloride molten salts for 60 h. The cross-sections were composed of three separated zones: out oxide layer, inner oxidation zone and substrate. The thickness of the inner oxidation zone increased with increasing corrosion temperature. The corrosion rate was also calculated from the depth of the oxide scales, and it consist of the results obtained from the weight loss well. As seen in the cross-sectional morphologies after corrosion in chloride molten salts at 700°C and 900°C, the presence of cracks between the oxide layer and the inner oxide layer indicated that the adhesion of the oxide layers was poor. The oxide scales formed on the surface of 316 ss were enriched with Zn, Cr and O and the contents of Fe and Ni were very low, indicating the preferred oxidation of Cr in 316 ss.

Figure 1: Corrosion weight loss curve of 316 stainless steel and Inconel 625 alloy in ternary chloride molten salt at (a) 700°C and (b) 900°C.

Figure 2: 316 ss surface morphology after corrosion in ternary chloride molten salt at 700°C (a–c), 900°C and (d–f) for 60 h.

3.2.2 Morphologies of the corrosion products formed on Inconel 625 alloy

The surface SEM images of the Inconel 625 alloy after corrosion in NaCl–KCl–ZnCl₂ at 700°C (a,b,c) and 900°C

(d,e,f) for 60 h are presented in Figure 4. Holes and spallation can be found in the oxide scales formed at both 700°C and 900°C; however, Inconel 625 alloy suffered severe corrosion at 900°C. Compared with those formed on the surface of 316 ss at the same temperature

Figure 3: Cross-sectional morphologies of 316 ss after corrosion in ternary chloride molten salt at 700°C (a) 900°C (b) for 60 h together with element distributions along the white lines.

(seen in Figure 2), the oxide scales formed on Inconel 625 were more compact and the grain sizes of the oxide were finer.

Figure 5 shows the cross-sectional morphologies of Inconel 625 alloy after corrosion in chloride molten salts at 700°C and 900°C for 60 h together with the element distribution along the yellow lines. The oxide scales were loose and unprotective; and because of the cracking and spallation, no outer oxide scales were observed after corrosion at both temperatures. Unlike that for 316 ss, no obvious inner oxidation zone was found after corrosion at 700°C (Figure 5(a)), but intergranular corrosion was observed beneath the loose oxide scale formed at 900°C (Figure 5(b)). The content of Cr in oxide scale formed at 700°C was higher than that in substrate, while the contents of Ni and Fe in oxide scale were lower than those in the substrate.

3.3 XRD patterns of the corrosion products

Figure 6 is the XRD patterns of the corrosion products formed on 316 ss and Inconel 625 alloy after corrosion in chloride molten salt at 700°C and 900°C. Combined with the EDS analysis results shown in Figures 3 and 5, it can be determined that the main phase of the oxide scales was ZnCr_2O_4 .

3.4 Potentiodynamic polarization measurements

Figure 7 shows the potentiodynamic polarization curves of 316 ss and Inconel 625 alloy in molten NaCl-KCl-ZnCl_2 at 700°C. Both potentiodynamic polarization curves of these two alloys showed active dissolution characteristics. The corrosion current density (I_{corr}), the corrosion potential (E_{corr}) and the anodic and the cathodic Tafel slopes (b_a and b_c) had been fitted using Tafel extrapolation method, as listed in Table 3. The corrosion potentials of 316 ss and Inconel 625 alloy were about -416.04 mV and -336.19 mV, respectively. From the point of thermodynamic tendency, the more negative E_{corr} is, the more active the alloy begins to corrode. Therefore, the corrosion resistance of 625 alloy in molten NaCl-KCl-ZnCl_2 is higher than that of 316 ss. The I_{corr} for 316 ss was 2.756 mA/cm^2 , which is about three times the value of 0.953 mA/cm^2 for Inconel 625 alloy, implying that the corrosion rate of 316 ss is higher than Inconel 625 alloy, which was based on the results obtained from immersion testing.

3.5 Electrochemical impedance spectra (EIS)

Figures 8 and 9 present the typical Nyquist and Bode plots of 316 ss and Inconel 625 alloy after corrosion in

Figure 4: Surface morphologies of Inconel625 alloy after corrosion in ternary chloride molten salt at 700°C (a–c) and 900°C (d–f) for 60 h, respectively.

molten NaCl-KCl-ZnCl_2 at 700°C under Ar for different times, respectively. During the initial stages of the experimental procedures, the Nyquist plots were composed of a

capacitive loop at high frequency and a line at low frequency, also called Warburg impedance, indicating that the corrosion was diffusion controlled. The Nyquist

Figure 5: Cross-sectional morphologies of Inconel 625 alloy after corrosion in ternary chloride molten salt at 700°C (a) and 900°C (b) for 60 h together with element distributions along the white lines.

Figure 6: XRD patterns of the corrosion products formed during the corrosion process in ternary chloride molten salt at 700°C and 900°C on the surface of (a) 316 ss and (b) Inconel 625 alloy.

plots composed of a huge capacitive loop after corrosion in chloride molten salts for 73 h (316 ss) and 62 h (Inconel 625 alloy) at 700°C, respectively.

Equivalent circuits shown in Figure 10 were chosen to fit the impedance spectra. It should be noted that the Nyquist plots with Warburg impedance were fitted as shown in Figure 10(a) and those composed of capacitive loops were fitted as shown in Figure 10(b), where R_s represents the molten salts resistance, C_{dl} the double-layer capacitance, R_t the charge transfer resistance and Z_w the Warburg impedance. Considering the dispersion

effect, a constant phase angle element CPE was used to replace the element C_{dl} . Thus, the total impedance of Figure 10 could be expressed as:

$$Z = R_s + \frac{1}{Y(j\omega)^n + \frac{1}{R_t + Z_w}} \quad (2)$$

$$Z = R_s + \frac{1}{Y(j\omega)^n + \frac{1}{R_t}} \quad (3)$$

where Y and n are constants representing the element Q_{dl} and Warburg impedance representing as $Z_w = A_w(j\omega)^{-1/2}$.

Figure 7: Potentiodynamic polarization curves of 316 ss and Inconel 625 alloy in ternary chloride molten salt at 700°C.

Table 3: Fitting results of polarization curves of 316 stainless steel and Inconel 625 alloy in ternary chloride molten salt at 700°C

	b_a (mV dec ⁻¹)	b_c (mV dec ⁻¹)	E_{corr} (mV)	I_{corr} (mA cm ⁻²)
316 ss	188.55	302.75	-416.04	2.756
Inconel 625 alloy	160.14	297.10	-336.19	0.9529

The fitting results are listed in Table 4 for 316 ss and Table 5 for Inconel 625 alloy. The charge transfers resistance (R_t) is the rate-limiting process in the melts. The values of R_t for Inconel 625 in the melt are larger than 316 ss, suggesting better corrosion resistance of Inconel

625 in chloride molten salt than 316 ss. The electrochemical impedance results are in good accordance with the potentiodynamic polarization and immersion test results.

4 Discussion

The results of immersion tests indicated that the corrosion resistance of 316 ss and Inconel 625 alloy demonstrated worse corrosion resistance in NaCl–KCl–ZnCl₂ at 700°C and 900°C, and protection is needed before using their structure materials for CSP system with chloride molten salts as the thermal storage mass. Both 316 ss and Inconel 625 alloys were attacked by pits and intergranular corrosion which were mainly attributed to the preferred dissolution of Cr in chloride molten salt.

The oxidizing gas atmosphere O₂ should be factors for the alloy corrosion, which could cause the significant corrosion. Although the corrosion processes were performed under Ar, the chamber was not vacuumed before introducing Ar, the residual O₂ would exist in the chamber. Otherwise, O₂ also produced by the decomposition of the hydrous water in the chloride molten salts during the heating. The element of alloys, such as Cr, Ni and Fe, could be oxidized by O₂, as followed:

Because of the more negative standard Gibbs free energy for reaction (4), Cr was preferred oxidized to form

Figure 8: Impedance Nyquist plots and bode diagrams of 316 ss in ternary chloride molten salt at 700°C.

Figure 9: Impedance Nyquist plots and bode diagrams of Inconel 625 alloy in ternary chloride molten salt at 700°C.

Figure 10: Equivalent circuit diagrams used for (a) Nyquist plots with Warburg impedance and (b) Nyquist plots composed of capacitive loops.

Table 4: EIS fitting results of 316 stainless steel in ternary chloride molten salt

Time/h	$R_s/\Omega\text{ cm}^2$	$C/F\text{ cm}^{-2}$	$R_t/\Omega\text{ cm}^2$	$A_w/\Omega\text{ cm}^2\text{ s}^{-0.5}$
15	0.97	0.062	0.23	0.28
38	0.78	0.085	1.53	0.17
45	0.80	0.0013	1.08	0.26
73	0.81	0.00057	1.20	0.10

Time/h	$R_s/\Omega\text{ cm}^2$	$Y_{0,dI}/s^n\text{ }\Omega^{-1}\text{ cm}^{-2}$	n	$R_t/\Omega\text{ cm}^2$
91	0.82	0.040	0.8	2.35

Table 5: EIS fitting results of Inconel 625 alloy in ternary chloride molten salt

Time/h	$R_s/\Omega\text{ cm}^2$	$C/F\text{ cm}^{-2}$	$R_t/\Omega\text{ cm}^2$	$A_w/\Omega\text{ cm}^2\text{ s}^{-0.5}$
16	1.41	0.00096	3.46	0.082
41	0.59	0.00067	4.27	0.081
62	0.64	0.0023	3.18	0.080

Time/h	$R_s/\Omega\text{ cm}^2$	$Y_{0,dI}/s^n\text{ }\Omega^{-1}\text{ cm}^{-2}$	n	$R_t/\Omega\text{ cm}^2$
89	0.72	0.027	0.8	4.55
100	0.59	0.022	0.8	9.14

a Cr_2O_3 layer on the surface of Inconel 625 alloy and 316 ss. The formation of Cr_2O_3 was controlled by diffusion, which was demonstrated by the Warburg resistance in EIS tests, as seen in Figures 8 and 9.

Besides reacting with metals, the existing oxygen would also react with chloride ion following the reaction:

Figure 11: Gibbs free energy of formation per molecule Cl_2 for various chlorides at 700°C.

Figure 12: Schematic diagram of activated oxidation.

The generated Cl_2 would diffuse through the oxide scale toward the base material because of high chlorine partial pressure and low oxygen partial pressure, which reacted with the alloys to form metal chlorides.

The standard Gibbs free energies of the metal chlorides per molar were calculated by HSC Chemistry 6.0 computer software and the results were listed in Figure 11. According to their standard Gibbs free energies of formation, it can be concluded that the stability of Cr, Fe and Ni elements in molten salt is in the following order: $\text{Cr} < \text{Fe} < \text{Ni}$. Therefore, Cr will be preferentially attacked by chloride molten salts due to the highest negative Gibbs free energy. This can explain the better corrosion resistance of Inconel 625 alloy than that of 316 ss.

The formed CrCl_2 , FeCl_2 and NiCl_2 will diffuse to the molten/oxide scale surface to react with O_2 , as followed:

Because of the highest volatile pressure of CrCl_2 and the most negative standard free Gibbs energy involved in the formation of Cr_2O_3 , reaction (12) would prefer to take place. The formed Cl_2 in reaction (12) would diffuse back to the oxide/metal interface again through the cracks and pores in the oxide scales to react with the metal. The

above processes were called “active oxidation,” and Figure 12 shows the schematic diagram of the activated oxidation.

However, the formed Cr_2O_3 were not stable and protective in chloride molten salts, with the prolonged time, and the followed reaction would take place.

The formation of ZnCr_2O_4 was confirmed by the XRD analysis of the corrosion products, as seen in Figure 6. The formed ZnCr_2O_4 layers were loose and nonprotective; as demonstrated in Figure 7, the potentiodynamic polarization curves of 316 ss and Inconel 625 alloy both showed active corrosion characteristic, and the Nyquist plot was composed of a large semi-circle [21], as shown in Figures 8 and 9.

5 Conclusions

Inconel 625 alloy performed better corrosion resistance than 316 ss in molten NaCl-KCl-ZnCl_2 at 700°C and 900°C , and the corrosion rate of these two alloys increased with increasing temperature. Both alloys show active corrosion in chloride molten salts. The current density of 316 ss was 2.756 mA/cm^2 about three times the value of 0.953 mA/cm^2 for Inconel 625, and the values of R_t for Inconel 625 were larger than that for 316 ss. A loose and porous corrosion layer, which mainly composed of ZnCr_2O_4 , was formed on the surface of these two alloys during the corrosion process in chloride molten salts owing to the selective corrosion of Cr in the alloys. The better corrosion resistance of Inconel 625

alloy can be explained by the higher Ni content than that in 316 ss.

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