Corrosion behavior of Inconel 625 deposited metal in molten KCl-MgCl₂

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Abstract

In order to investigate the corrosion behavior of Inconel 625 deposited metal in molten KCl and MgCl₂, the corrosion behavior of deposited metal immersed in molten salt for 60 h at 700 °C and 900 °C was studied by static corrosion immersion method. X-ray diffraction (XRD) and Geminisem300 were used to systematically study the phase composition, corrosion morphology and element distribution of the deposited metal. The results show that: the corrosion weight loss of the deposited metal showed an increasing trend at both temperatures, but the increasing range was different in different time intervals. The corrosion weight loss of the deposited metal increased slowly in the first 10 h, and increased sharply in the 10 h–60 h. It can be found that 10 h is the cut-off point of corrosion behavior. The corrosion rate is 0.03 mm/year at 700 °C and 0.50 mm/year at 900 °C for 10 h. The corrosion resistance of the deposited metal at 700 °C is better than that at 900 °C, which is due to the formation of dense MgO on the surface of the deposited metal at 700 °C, which hinders the corrosion reaction; at 900 °C, the content of CrCl₃ on the surface of the deposited metal increases, resulting in a ‘shell breaking effect’, which destroys the MgO shell and forms NiCr₂O₄ with spinel structure. Its corrosion resistance is thus weakened.

1. Introduction

Due to environmental pollution and the limitation of fossil fuel resources, solar energy has become an important part of human life [1]. Modern solar energy technology gathers sunlight and uses its energy to generate electricity or other energy. However, there are still some problems in solar energy technology that make it unable to work continuously. For example, solar energy is affected by night, dust and clouds, which greatly reduces the efficiency of the solar system [2–4]. In order to solve this problem, it is necessary to configure a set of energy storage device to ensure continuous energy output. At present, nitrate, fluoride molten salt and chloride molten salt are the main energy storage media. The highest temperature of nitrate medium is 540 °C, which is lower than the working temperature of energy storage device i.e. 700 °C–900 °C; The volume of fluoride molten salt increases during solidification, which is prone to ‘hot loosening’; however, chloride molten salt (KCl-MgCl₂) is more suitable for high-temperature energy storage due to its low melting point (426 °C), small thermal conductivity (0.4 W m⁻¹°C⁻¹), low price and good high-temperature stability [5–7]. Because the energy storage medium is working at high temperature, its loading equipment is mainly made of high temperature resistant materials, Inconel 625 alloy is widely used in solar energy storage devices because of its excellent corrosion resistance and weldability [8]. In order to explore the interaction mechanism between energy storage media and different materials, a large number of scholars have carried out relevant researches. J W Wang [9] studied the corrosion behavior of carbon steel and Fe–Cr–Ni alloy in molten eutectic NaCl and MgCl₂. It was found that Cr was the key factor affecting the corrosion, and MgO formed on the surface of the sample could hinder the corrosion. H Sun [10] analyzed the corrosive effect of molten NaCl–KCl–MgCl₂ by Raman spectroscopy and infrared
spectroscopy. The results show that magnesium treatment can remove corrosive impurities in molten salt, and improve the corrosion resistance of Hayness 242, Hastelloy & Hayness 230 alloys, which is basically consistent with J W Wang’s conclusion. However, the above documents only studied the corrosion behavior of nickel base alloy in chloride molten salt at 520 °C and 800 °C, but did not explore the corrosion behavior of Inconel 625 at higher temperature. J Y Li [11] studied the corrosion behavior of 316 stainless steel and Inconel 625 in ZnCl2, KCl and NaCl at 900 °C, and found that the two alloys were seriously corroded in ternary chlorides, forming loose oxide layer on the surface, and the oxide film did not play a protective role on the alloy. H F Ma [12] found that the corrosion rate of Inconel 625 alloy in alkaline earth metal is higher than that in alkali metal. Although the above-mentioned documents have also covered research on molten salt corrosion of Inconel 625 and obtained some useful conclusions, they are mainly aimed at as cast Inconel 625. In the casting process of Inconel 625, there are many casting defects, such as uneven coarse grains, porosity and segregation, which affect its properties. At the same time, Inconel 625 needs to face the corrosion of molten salt and form corresponding corrosion pits in the service process. If the defects and corrosion pits exceed the standard, they need to be repaired to ensure the continuous service of the product [13, 14]. However, there are few reports on molten salt corrosion performance analysis of Inconel 625 repair castings. Therefore, it is necessary to study the corrosion behavior and corrosion mechanism of deposited metal in chloride molten salt, which is of great significance to master the corrosion rate and predict the service life of repaired structural parts. In this paper, the molten salt corrosion behavior of Inconel 625 deposited metal was studied. The corrosion depth and corrosion rate of Inconel 625 deposited metal in 40 wt% KCl + 60 wt% MgCl2 at 700 °C and 900 °C were measured by static corrosion immersion method. The corrosion mechanism of the deposited metal in molten salt was explored, providing theoretical guidance and technical reserve for the repair of solar energy storage devices.

2. Experiments

2.1. Experimental materials and methods

Inconel 625 welding wire is used in the test, and the main components are shown in Table 1. The diameter of Inconel 625 welding wire is 1.2 mm. MAG welding method was adopted. The welding parameters were: welding current 150 A, welding voltage 24 V, welding speed 250 mm min⁻¹. According to the technical standard provided by welding material manufacturer, the shielding gas is set as 80% Ar + 20% CO2. The welding wire was deposited on Q235 substrate to prepare Inconel 625 deposited metal with a height of 12 mm. Because the interlayer temperature has a great influence on the surfacing welding, the interlayer temperature should be strictly controlled ≤120 °C. In order to reduce the dilution ratio of surfacing layer to the minimum, a total of 4 layers were built up in this experiment. According to ASTM G31-2012a: Standard Guide for Laboratory Immersion Corrosion Testing of Metals. The static immersion corrosion method was used to simulate the working environment of the solar thermal storage device. The samples were taken from 2nd and 3rd layers of the surfacing layer, and the sample size was 10 mm × 10 mm × 2 mm. The samples were first polished with metallographic sandpaper and cleaned with acetone ultrasonic, then the molten salt was dried at 110 °C for 48 h. In the static immersion corrosion experiment, the sample was placed in 40 wt% KCl + 60 wt% MgCl2 molten salt for high temperature heating, to ensure that the molten salt completely covered the sample. Temperature was set at 700 °C and 900 °C, kept for 2 h, 6 h, 10 h, 20 h, 30 h, 40 h, 50 h and 60 h, respectively. The static immersion corrosion diagram is shown in Figure 1. After the molten salt test, the samples were boiled in boiling water for half an hour and cleaned via ultrasonic to remove the molten salt attached to the surface. After drying, the samples were weighed in an electronic balance of 0.1 mg and compared with the original weight to calculate the weight loss. In this paper, three experiments are repeated to ensure the reproducibility of the experimental data. At the same time, in order to ensure the representativeness of the test data, three samples are used for each group of tests, and the test results are their average values.

2.2. Properties of deposited metal

XRD-7000 x-ray diffractometer was used to determine the phase composition of the deposited metal after corrosion. The specific parameters are as follows: pure copper target, tube voltage 40 kV, current 30 mA, scanning speed 2° min⁻¹, scanning range 20 °C–90 °C. The microstructure of deposited metal was observed and

| Table 1. Chemical composition (mass fraction) of Inconel 625 welding wire. |
|-------------------|----------------|---|---|---|---|---|---|---|---|---|---|---|
|                  | Cr  | Mo  | Mn  | Fe  | Si  | C   | Cu  | P  | S   | Ti  | Nb + Ta | Ni  |
|-------------------|-----|-----|-----|-----|-----|-----|-----|----|-----|-----|---------|-----|
|                  | 21.2| 8.6 | 0.23| 0.9 | 0.43| 0.02| 0.02| 0.01| 0.006| 0.16| 3.50     | Bal  |

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3. Experimental results and discussion

Figure 2 shows the weight loss curve of Inconel 625 deposited metal in molten salt at different temperatures. It can be seen from figure 2 that with the increase of test time, the corrosion weight loss at both temperatures shows an increasing trend, but the increase scale varies in different time intervals. The corrosion of deposited metal increased slowly in the first 10 h, and the corrosion weight loss increased sharply from 10 h to 60 h. This is because with the increase of time, the contact time between the deposited metal surface and molten salt is prolonged. With the development of deep corrosion reaction, the amount of corrosion products increases, while the corrosion products are mostly oxides or compounds, and the plasticity is very poor. In the subsequent cleaning process, cracking and spalling occurred with the substrate, resulting in a significant increase in corrosion weight loss of 10 h–60 h.

The corrosion weight loss curve can well reflect the variation of corrosion weight loss with time, but it cannot clearly reflect the corrosion rate of deposited metal in different stages. Therefore, the corrosion weight loss is converted according to the corrosion rate formula (1) and (2), and the corrosion rate curve with time is drawn, and the results are shown in figure 3 [15]. It can be seen from figure 3 that the corrosion rates at the two temperatures show the same change trend. When the corrosion time is less than 10 h, the corrosion rate

![Figure 1. Schematic diagram of static immersion corrosion test.](image1)

![Figure 2. Molten salt corrosion weight loss curve of Inconel 625 deposited metal at different temperatures.](image2)
increases gently. When the corrosion time exceeds 10 h, the corrosion rate increases by leaps and bounds. However, when the corrosion time is further increased to more than 20 h, the corrosion rate decreases, and with the extension of corrosion time, the corrosion rate basically keeps unchanged, and it has entered the stable corrosion range. This is because there is a certain protective film on the surface of the deposited metal at the initial stage of corrosion, which has a certain resistance to high-temperature oxidation. With the extension of high-temperature residence time, the original protective film will gradually break and lose its guarantee function. However, this process occurs randomly on the surface of the deposited metal, rather than uniformly. Therefore, the corrosion rate changes sharply at this time. When the protective film is completely broken and the cladding metal enters into uniform corrosion, the whole cladding metal surface participates in corrosion, and the corrosion rate relatively stabilizes.

\[ V_\text{weight loss} = \frac{m_0 - m_1}{St} \]  \hspace{1cm} (1)  

\[ V_L = \frac{V \times 24 \times 365}{1000\rho} \]  \hspace{1cm} (2)

\( V_\text{weight loss} \) is the corrosion rate at weight loss (g/m²h), \( m_0 \) is the original mass of the sample (g), \( m_1 \) is the mass of the sample after removing the corrosion products (g), \( S \) is the surface area of the metal sample (m²), and \( t \) is the corrosion time (h). \( V_L \) is the annual corrosion rate (mm/year) and \( \rho \) is the density of metal (g cm⁻³).

In order to analyze the corrosion mechanism of deposited metal, the double logarithmic curves of corrosion weight loss and corrosion time at 700 °C and 900 °C were made as shown in figures 4 and 5. It can be seen that the high temperature molten salt corrosion of the deposited metal can be divided into two stages. From 700 °C to 900 °C, the slope of the initial curve decreases from 1.32 to 1.01. In this stage, the sample is controlled by the surface oxidation and diffusion. The oxide layer is densified at 700 °C and the oxidation products fall off seriously at 900 °C; in the later stage, the slope of the curve in the later stage was 0.67 and 0.66 respectively, and the difference between them was very small. The results show that the deposited metal exhibits the same corrosion law in the later stage of corrosion. With the increase of corrosion time, the corrosion holes increase and the corrosion intensifies.

According to the above corrosion curve, it can be found that 10 h is the cut-off point of corrosion behavior. Therefore, in the subsequent analysis of corrosion products and corrosion mechanism, 10 h state is selected to carry out. Figure 6 shows the phase composition of deposited metal surface at different temperatures. There are four curves in figure 6, including the XRD patterns of corrosion products before and after cleaning at 700 °C and 900 °C. It can be seen from figures 6(a), (c) that NiCr₂O₄, Cr₂O₃ and MgO are found on the uncleaned deposited metal surface at two temperatures, while the corrosion reaction is accelerated at 900 °C, and MgCr₂O₄ and MoO₃ are also found on the uncleaned deposited metal surface; after boiling the deposited metal for half an hour and ultrasonic cleaning, it is found from figures 6(b), (d) that there are NiCr₂O₄ and MgO on the surface of the deposited metal cleaned at two temperatures, while the content of MgO is less and MoO₃ is precipitated at 900 °C. It can be seen that Cr₂O₃ is the main exfoliated corrosion product of deposited metal before and after cleaning at 700 °C, and Cr₂O₃, MgO and MgCr₂O₄ are the main exfoliated corrosion products before and after cleaning at 900 °C. Cr₂O₃ was not detected on the surface of deposited metal after cleaning, because Cr₂O₃ and molten salt fell off during boiling water bath.
Figure 7 shows surface morphology of Inconel 625 deposited metal after cleaning under 700 °C (a) and 900 °C (b) molten salt corrosion. When the temperature is 700 °C, the surface of Inconel 625 is mainly white shell material, as shown in point a in figure 7(a)A. Combined with XRD analysis, the surface product is mainly MgO with a small amount of spalling and corrosion pits, as shown in figure 7(a)B. When the temperature is 900 °C, the corrosion holes formed on the surface of deposited metal are larger and more than those formed at 700 °C, and the corrosion depth increases. Combined with XRD analysis, the surface products are NiCr2O4, MoO3 and MgO, and the grain size in the corrosion layer is different. With the increase of temperature, the adhesion between the corrosion products and the substrate becomes worse, and MgO falls off, resulting in the weight loss of the sample. Through SEM and XRD analysis, the main corrosion products are NiCr2O4 with spinel structure, as shown in figure 7(b)C. In addition, MoO3 precipitates, which is consistent with the results of reference [12].

In order to determine the phase composition in figure 7, the shell surface and spinel structure surface of the cleaned deposited metal after corrosion at 700 °C and 900 °C for 10 h were locally enlarged. The element content test area is shown in figures 8(a), (b), and the EDS scanning analysis results are shown in figures 8(c), (d). After high temperature molten salt corrosion, the content of Cr on the surface of the deposited metal decreases. At
700 °C, the content of Cr, Ni and Mo on the surface of the sample is 19.63%, 59.30% and 8.59%, respectively; At 900 °C, the contents of Cr, Ni and Mo on the surface of deposited metal are 4.86%, 61.04% and 19.92%, respectively. It shows that Cr element in the deposited metal is corroded and dissolved into molten salt. Considering that Mo is a more stable component, the atomic number is large and the diffusion rate is slow. With the corrosion reaction going on at 900 °C, Cr, Ni and other atoms in the deposited metal diffuse to the surface of the alloy and enter into the molten salt by reaction dissolution, resulting in vacancy in the original position of the deposited metal. With the appearance of vacancies, Mo diffuses and precipitates on the surface of the deposited metal, which leads to the enrichment of Mo on the surface [16].

In order to understand the distribution of elements on the surface of deposited metal at different temperatures, the surface scanning analysis was carried out at two temperatures. The results are shown in figures 9 and 10. Figure 9 shows the element distribution of Inconel 625 deposited metal after corrosion at 700 °C for 10 h. At 700 °C, the distribution of Ni, Cr, Mo, Mg and O is uniform to the deposited metal. In the process of corrosion reaction of deposited metal, Cr element oxidizes and dissolves into molten salt with diffusion precipitation, resulting in Cr poor layer with depth of 23.4 μm. The content of Mg in the outer oxide layer is rich, mainly Mg oxides. Combined with the previous XRD results, the outer oxide is mainly MgO, and a small amount of corrosion pits appear in the inner corrosion layer.

Figure 10 shows the element distribution of Inconel 625 deposited metal after corrosion at 900 °C for 10 h. As shown in figure 10, the cross section photo of the sample after corrosion in molten chloride salt is divided into three layers from the outside to the inside: the outer oxidation layer, the inner oxidation layer and the substrate. With the increase of temperature, the corrosion depth of deposited metal increases. Compared with figure 9, the
corrosion depth increased from 25 μm to 97.2 μm. After corrosion at 900 °C for 10 h, the element diffusion path is longer, the Cr content in the outer oxide layer and the inner corrosion layer decreases, and the Ni content in the inner corrosion layer decreases. The activity of main metal elements in molten salt is in the order of Cr > Mo > Ni. For Inconel 625 deposited metal, due to the migration of Cr element, vacancy appears in the sample, and Cr dilution layer is formed under the action of concentration gradient [17–19]. Therefore, the
corrosion degree of the sample can also be reflected by measuring the depth of Cr dilution layer. With the increase of temperature, the depth of Cr poor layer increases to 95 μm. According to XRD analysis, the main corrosion products on the surface of deposited metal at 900 °C are NiCr₂O₄ with spinel structure and MoO₃, and a small amount of MgO precipitation.

4. Discussion

In order to explore the corrosion mechanism of Inconel 625 deposited metal in chloride molten salt, the corrosion process and corrosion products were elaborated. After 10 h of corrosion, the corrosion was mainly caused by oxygen ions in molten salt. In this experiment, there were no oxygen ions in MgCl₂ and KCl molten salts, and oxygen ions mainly came from air and water vapor. Cr reacted first and oxidized to form Cr₂O₃. However, the oxide film was unstable at the initial stage, which was easy to decompose into molten salt according to the reaction formula (3), (4).

\[ 2Cr + 1.5O_2 = Cr_2O_3 \]  \hspace{1cm} (3)
\[ 2Cr_2O_3 + 4O^{2-} + 3O_2 \rightarrow 4CrO_4^{2-} \]  \hspace{1cm} (4)

The ionic compounds were finally dissolved in molten salt, which is consistent with the weight loss curve of corrosion. With the increase of corrosion temperature, the dissolution rate of oxide film was accelerated. It is also proved that the Cr poor layer of the sample at 900 °C is deeper than that at 700 °C. The Cr poor layer is also related to the grain size. Through the XRD analysis of the exfoliated products, according to the modified Scheler formula (5), the average grain size of Cr₂O₃ increases from 11.6 nm to 13 nm with the increase of temperature [20]. The increase of grain size leads to the decrease of grain boundary, which reduces the bonding force between metals and increases the diffusion and dissolution of Cr, which also confirms the increase of dilution layer depth.

\[ L = \frac{\lambda[1.392 - \pi (\cos \varepsilon - 1)(P - 1)]}{2\pi \sin \varepsilon \cos \theta} \]  \hspace{1cm} (5)

L is the average thickness of grains perpendicular to the mirror direction; ε is the angle offset; θ is the diffraction angle; λ is the x-ray wavelength; P is the number of plane.

Another key factor affecting the corrosion rate of deposited metal in chloride molten salt is the corrosion products formed on the surface. In order to clearly describe the change of corrosion products with the increase of temperature, the corrosion mechanism of Inconel 625 deposited metal is shown in figure 11. The surface of deposited metal is mainly MgO after corrosion at 700 °C for 10 h. MgCl₂ molten salt has strong water absorption, easy to form MgCl₂·XH₂O and decompose into MgOHCl and HCl. According to formula (6)–(9), MgO is formed on the surface, which is consistent with XRD and SEM analysis. Although the drying treatment
was carried out before the experiment, H2O could not be completely removed. O2− itself has no redox performance, but it will react with corrosion products and affect the corrosion resistance of the sample [21–24].

\[
\begin{align*}
\text{MgO} \cdot \text{XH}_2\text{O} & = \text{MgOHCl} + \text{HCl} \\
\text{MgOHCl} & = \text{MgO} + \text{HCl} \\
\text{Cr} + 2\text{HCl} & = \text{CrCl}_2 + \text{H}_2 \\
\text{Cr} + \text{MgOHCl} & = 2\text{MgO} + \text{CrCl}_2 + \text{H}_2
\end{align*}
\]

MgO particles deposited on the surface of the sample with heterogeneous nucleation, and finally formed a continuous and dense oxide shell on the surface of the sample, which hindered the corrosion reaction. This result is consistent with [9]. MgO is joined along the grain boundary, which increases the strength. The adhesion between the oxide layer and the sample surface is enough to prevent corrosion.

According to the reaction formula (10), Cl− forms Cl atom, which diffuses to the matrix and reacts with Ni and Cr to form CrCl3 and NiCl2. During the diffusion process of reactant CrCl3, stress concentration will occur. With the increase of temperature, the surface oxide layer gets loose and porous, and corrosion is accelerated, which is consistent with the corrosion weight loss curve at 900 °C. According to formula (11), at 900 °C, CrCl3 reacts with MgO on the surface of deposited metal to form MgCr2O4, which is consistent with the XRD of exfoliated corrosion products at 900 °C [11]. With the increase of temperature, MgO on the surface of the sample falls off, and the surface is mainly attached with NiCr2O4 with spinel structure and MoO3. This is determined by the corrosion kinetics. Generally speaking, with the increase of corrosion temperature and the fast growth of grain, part of the grain boundaries can be eliminated. The diffusion of Ni along the grain boundary is 4–5 orders of magnitude faster than that within the lattice [25]. The rapid diffusion of Ni ions along the grain boundary will cause holes in the inner corrosion layer and the matrix. The previously formed MgO decomposes, and the decomposed O passes through the ‘diffusion channel’ shown in figure 11(b)A to form NiCr2O4 with spinel structure. At 900 °C, Mo in the outer oxygen layer of the deposited metal is oxidized to MoO3. MoO3 changes the way for passive film to choose ions, which can cause migration of cation and hinder the entry of Cl− and OH− anions. However, as can be seen from figure 10(d)A area, the MoO3 formed is discontinuous and can only protect the corresponding surface, while other surfaces can not be protected.

\[
\begin{align*}
\text{Cl}^- - e & \rightarrow \text{Cl} \\
2\text{CrCl}_3 + 4\text{MgO} & = \text{MgCr}_2\text{O}_4 + 3\text{MgCl}_2
\end{align*}
\]

5. Conclusion

In this paper, the molten salt corrosion behavior of Inconel 625 deposited metal at different temperatures was studied. By comparing the corrosion behavior at two temperatures, the main conclusions are as follows:

(1) Inconel 625 is more corrosion resistant at 700 °C than at 900 °C. The corrosion rate of deposited metal is 0.03 mm/year and the corrosion depth is 25 μm at 700 °C for 10 h; the corrosion rate of deposited metal is 0.50 mm/year and the corrosion depth is 97.2 μm at 900 °C for 10 h.

(2) On the surface of deposited metal at 700 °C, the content of Mg and O is rich, forming a dense MgO shell, which hinders the corrosion reaction; at 900 °C, the MgO shell on the surface of the deposited metal falls
off, and Ni and Cr elements dissolve and diffuse outward, forming NiCr₂O₄ with spinel structure, which reduces the corrosion resistance.

(3) With the increase of corrosion temperature, Cr element diffuses and dissolves. At 700 °C and 900 °C, the depth of Cr poor layer increases from 23.4 μm to 95 μm. With the increase of CrCl₃ content, stress concentration appears and ‘shell breaking effect’ will take place, destroying MgO shell and intensifying corrosion.

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