An Experimental Study on High Temperature Corrosion of TP347H Stainless Steel in Molten Chloride and Sulfate

Weixin Yu1, Xi Chu1, Huihui Zhang2*, Yue Shen2, Kangning Tao1 and Jueyan Li2

1 China Power Huachuang Electricity Technology Research Company LTD, Suzhou 215123, China
2 Key Laboratory of Power Station Energy Transfer Conversion and System, Ministry of Education, North China Electric Power University, 102206, Beijing, China
Email: 1119359959@qq.com

Abstract. The corrosion resistance of TP347H stainless steel was evaluated by measuring mass loss in molten salt at 500-650°C. The corrosion mechanism was characterized by scanning electron microscope (SEM), energy dispersive spectrometer (EDS) and X-ray diffraction (XRD). The results show that the corrosion resistance of TP347H stainless steel increases with the increase of corrosion temperature. When the temperature is below 600°C, TP347H mainly generate low-stable Fe3Oy, and the oxides such as Fe2O3, Fe3O4, Ni1.43Fe1.7O4 and NiFe2O4 are dissolved with the increase of temperature. NbO with higher stability is formed on the surface at 650 °C, which help Cr2O3 and NiO retain for a longer time. Mn-containing compounds on the surface further improve corrosion resistance of TP347H. The corrosion of TP347H stainless steel is mainly intergranular corrosion, and the temperature range of corrosion is consistent with the melting range of alkali metal chloride. Therefore, the molten alkali metal chloride plays a decisive role in the corrosion of TP347H stainless steel.

Keywords. TP347H stainless steel, high temperature molten salt corrosion, oxidation reaction.

1. Introduction
Waste incineration power generation technology incinerates combustible materials in waste to generate electricity. During the incineration process, the flue gas with high content of alkali metal (sodium, potassium) chloride salt and sulfate is generated, and the corrosive deposited ash is formed on the heat transfer surface [1,2], which causes severe corrosion to waste incineration equipment such as superheater and so on [3-5]. At present, the service temperature of superheater tubes in waste incinerators is about 450-600°C [3]. Considering localized overheating, the temperature of heat exchange surface of superheater tubes may reach more than 650°C. The higher temperature increases the amount of molten phase in deposits, causing the tubes suffer more serious corrosion. Currently, austenitic stainless steel TP347H (1Cr19Ni11Nb) is widely used in superheater tubes because of its high-cost performance. TP347H contains a high content of Ni and Cr, and also contains a certain amount of Nb. These alloying elements form dense protective oxide scales to improve corrosion resistance under extreme conditions [6]. Ni can effectively inhibit Cl and Cl- induced stress corrosion, while promoting the formation of austenite which significantly improves the mechanical properties (such as elasticity and toughness) of TP347H stainless steel. Cr can significantly reduce intergranular corrosion [7, 8]. Both of Ni and Cr can react with oxygen to form dense protective oxide scales during service. When the total content of these two elements conforms to the n/8 law, the passive performance and corrosion resistance of the steel is significantly improved.
2. Material and Experimental Methods

TP347H stainless steel samples were wire cut according to the size of 20mm × 10mm × 2.7mm, and the alloy composition is shown in Table 1. All samples were ground with SiC paper from 400# to 1500# and prepared for the corrosion experiments.

Table 1. Chemical compositions of TP347H.

| Elements (wt.%) | Fe  | Ni  | Cr  | C   | Mn | Si  | Nb  | S   | P   |
|----------------|-----|-----|-----|-----|----|-----|-----|-----|-----|
| TP347H         | Bal.| 12.75 | 19.2 | 0.1 | 2  | 0.09 | 1.4 | 0.03 | 0.02 |

A corrosion reagent consisting of 25 wt.% NaCl, 25 wt.% KCl, 25 wt.% Na₂SO₄ and 25 wt.% K₂SO₄ was used to simulate the corrosion of TP347H in waste incineration environment. Before the corrosion experiment, all samples were embedded in the corundum crucibles containing corrosion reagent. Place the small corundum crucible in two large corundum crucible boats and put it in the isothermal position of resistance furnace. In order to ensure experimental reproducibility and consistency of the data, the same crucible must be placed in the same position.

In this experiment, the tubular resistance furnace which used electrothermal alloy 0Cr27Al7Mo2 as heating source, was used to simulate the waste incineration environment of the equipment. The isothermal corrosion experiment was carried out at 500℃, 550℃, 600℃ and 650℃, taking 12h as a cycle for a total of 180h. Maintain the temperature at the isothermal area to fluctuate within the set temperature ±1℃ through the temperature control instrument.

The mixed corrosive reagents have different melting states at different experimental temperatures, and the melting states at every test temperature was listed in table 2. Therefore, in order to ensure the consistency and repeatability of the experiment, it is necessary to renew the new corrosive reagents before the beginning of each corrosion cycle to maintain the sample covered by the mixed reagent during the corrosion experiment.

Table 2. Melting state of corrosion reagent at different temperatures in air atmosphere (The composition of a solid remains unchanged after it is converted into a liquid).

| Corrosion reagent | Temperature (℃) |
|-------------------|------------------|
|                   | 500              | 550              | 600              | 650              |
| R                 | Not melt         | Partly melt      | Melt             | Melt             |

Due to corrosion products were easy to spall and corrosion reagents were easily adhered to the surface, it is difficult to obtain accurate experiment data of corrosion mass gain. Under these circumstances, the non-recycling method is adopted for the experiments. One sample was removed at the end of each cycle for pickling and weighing to obtain more accurate and reasonable corrosion mass loss data. The samples corroded for 12h and 180h were first kept in their original state for SEM, EDS and XRD experimental examination of the corrosion products, followed by the same pickling and weighing. The following equation was used to measure the corrosion resistance of the samples[9]:

\[
\gamma_{corr} = \frac{\Delta m}{A}
\]  

where \( \Delta m \) (g) is the cumulative mass loss determined simultaneously with increasing corrosion time, and \( A \) (m²) is the original surface area of the sample.

When a corrosion cycle was finished, the samples were removed and cleaned by deionized water. Then samples were pickled in hydrochloric acid bath (25 wt.%, 80 ℃) for 3-10 minutes to further remove the corrosion products [9]. According to the actual situation, the pickling time need appropriately extend to ensure the removal of corrosion products and residual corrosion reagents. Finally, the samples were ultrasonically cleaned in deionized water, completely dried and then
weighed by an electronic balance with an accuracy of ± 0.01 mg. The corrosion time-$\gamma_{corr}$ mass loss graph was used to compare the corrosion resistance of the samples at different temperatures.

The cross-section of the samples corroded for 12h and 180h was ground through SiC paper from 400# to 1500# and prepared for further test. The surface morphology was observed by scanning electron microscope (SEM). The Chemical compositions and distribution of elements were detected by energy dispersive X-ray spectroscopy (EDS). The corrosion products of the last corrosion cycle were pulverized into powders for phase identification. A high-power X-ray diffraction (XRD) meter(D/MAX-RS12KW) was used for phase identification with Cu Ka incident radiation operating at a scanning speed of 8°/min and 20 from 10° to 110° at 40 kV and 100 mA.

3. Result and Discussion

3.1. Mass Loss

Figure 1 shows that the corrosion mass loss curves of TP347H stainless steel at different experimental temperatures are almost straight line. With the increase of time and temperature, the corrosion loss shows an increasing trend. The mass loss was the minimum at 500°C (94.29g/m²) and the maximum at 650°C (462.32g/m²). The slope of the curve increases with the increase of temperature, indicating that the corrosion rate also shows the increasing trend. After 180h of corrosion at 650°C, the surface area of the sample did not change significantly, but the corrosion products generated at the grain boundary and the slagging on the surface made the actual situation deviate from the curve to some extent, but had little influence on the final results.

![Figure 1](image)

**Figure 1.** Mass loss ($\Delta$m/A) of TP347H stainless steel corroded in corrosion reagent in air at 500-650°C after pickling in the hydrochloric acid solution bath (25wt.%, 80°C) within 3-10min.

3.2. Corrosion Surface Morphology and Composition

Figure 2 shows the SEM surface morphology and EDS scanning results of corrosion products of TP347H stainless steel corroded for 12h at different temperatures (500-650°C). It can be seen that the surface of the sample corroded at 500°C showed slight nodular corrosion products and some micro holes, while the surface of the sample corroded at 550°C showed flake corrosion products (figure 2(b)) and corrosion products showed slight spalling phenomenon. When the temperature rose to 600°C, a relatively flat layer of corrosion products (figure 2(c)) and particle-shaped corrosion products (figure 2(d)) appeared on the sample surface, accompanied by layered spalling of corrosion products. EDS results show that Cr content in the surface layer (point A) is significantly higher than that in the inner layer (point B), indicating that more Cr$_2$O$_3$ was generated on the surface of the sample. At 650°C, the corrosion reagent completely melted, and the particle-shaped phase in corrosion products gradually increased and micro cracks appeared (figure 2(f)). At high magnification (figure 2(e)), needle-shaped and flap-shaped corrosion products were formed on the surface and the corrosion products were layered. Intergranular corrosion was found at the grain boundary, the concave phenomenon and crack
appeared on the surface accelerated the intergranular corrosion. EDS analysis shows that the corrosion products mainly consist of Fe-rich oxides and a small amount of Ni- and Cr-oxides. Both the white flap-shaped corrosion product (point A) and the needle-shaped corrosion product (point B) are composed of Cr-rich compounds. The corrosion products (point C) mainly contain Ni-rich compounds. When the temperature increased from 600°C to 650°C, the relative content of Fe in the external corrosion products decreased significantly, indicating that the oxidation of Fe and the loss of corrosion products occurred at high temperature.

![Figure 2. SEM surface morphology and EDS scanning results of the corrosion products of TP347H stainless steel corroded in corrosion reagent air for 12 h at different temperatures (500-650°C).](image)

Figure 3 shows the surface morphology and EDS scanning results of samples corroded for 180h at 500-600°C. At 500°C, multilayer corrosion products appeared on the surface of the sample accompanied by serious spalling phenomenon (figure 3(a)). Obvious intergranular corrosion occurs at 550°C (figure 3(b)). EDS scanning results at point A show that the corrosion products at the grain boundary are mainly Fe-rich oxides and some oxides containing Ni and Cr. The content of O at grain boundaries is higher than that at grain boundaries, indicating that oxidation of Fe, Ni and Cr occurs at grain boundaries. The severe oxidation of Fe and the formation of NiO and Cr₂O₃ consume Fe, Ni and Cr, resulting in the lack of Ni, Cr and Fe at grain boundaries. The particle-shaped corrosion products at point C are mainly composed of Nb and O. When the temperature rises to 600°C, the corrosion reagent was in the coexistence state of melting and semi-melting, forming slagging on the surface and the thick oxide layer. The melting point of the corrosion reagent is about 600°C, which is the same as the temperature of slagging phenomenon, so the occurrence temperature of slagging phenomenon is related to liquid transition of the corrosion reagent.

Figure 4 shows the cross-section morphology photos and surface scanning results of the samples corroded for 180h at 650°C. From the figure 5, the intergranular corrosion of the alloy can be clearly observed which diffused from the surface to the interior of the alloy. Therefore, it can be judged that the failure of the sample in the corrosive environment is caused by the intergranular corrosion. The intergranular corrosion diffusion is the deepest in the samples at 650°C, so it can be found that the intergranular corrosion increases with the increase of temperature. The results show that there are high contents of S, Cl and O in the corrosion products, and a small amount of Cl and S in the uncorroded area, indicating that intergranular corrosion of the alloy causes Cl and S continue to corrode the matrix through the oxide layer. The corrosion product layer contains a large number of metal oxide layers and the matrix has less O content. The distribution of Fe, Ni and Cr is relatively uniform, indicating that the oxide of these three metal elements can effectively protect the matrix from further oxidation. The corrosion products on the surface of the sample are rich in Ni, Cr and Nb, while the content of Fe is relatively low, which suggests that Fe was serious oxidized, and the oxide film of Ni, Cr and Nb is
more stable than that of Fe, resulting in the enrichment of Ni and Cr on the surface. Although NbO has high stability, the relative content is low.

Figure 3. SEM surface morphology and EDS scanning results of the corrosion products of TP347H stainless steel corroded in corrosion reagent for 180h at 500-600℃. (a)500℃, (b)550℃, (c)600℃

Figure 4. Cross-section morphology and EDS scanning results of the corrosion products of TP347H stainless steel corroded in corrosion reagent in air for 180h at 650℃.

Figure 5 shows the XRD results of the samples corroded for 180h at 500-650℃. When the temperature is below 600℃, Fe-rich oxide layer was formed on the surface of the sample, which is mainly composed of Fe$_3$O$_4$, Fe$_2$O$_3$, Ni$_{1.43}$Fe$_{1.7}$O$_4$ and NiFe$_2$O$_4$. As the temperature rises to 600-650℃, Fe$_3$O$_4$ became the only Fe-containing compound. NiO and NbO were detected in the corrosion products, while Cr$_2$O$_3$ had been dissolved, which further demonstrated the stability of NiO. The sample only generated highly stable NbO at 650℃, which increases the existence time of Cr$_2$O$_3$ and NiO in the corrosion environment. Mn-containing compounds were also detected, which further improved the corrosion resistance. It should be noted that KCl, NaCl, K$_2$SO$_4$ and Na$_2$SO$_4$ contained in the corrosion reagents were detected in all test samples, but they are not marked. And metal chloride has generated in the corrosion process, which was easily volatile in high temperature environment. Before XRD detection, all samples were cleaned with deionized water, so no metal chloride was detected in the corrosion products.

| T(℃)   | Phase identification |
|--------|----------------------|
| 650℃   | Fe$_3$O$_4$+NiMn$_{0.5}$Cr$_{1.5}$O$_4$+NiO+ NbO |
| 600℃   | Fe$_3$O$_4$+Fe$_2$O$_3$+Cr$_2$O$_3$+Ni$_{1.43}$Fe$_{1.7}$O$_4$ |
| 550℃   | Fe$_3$O$_4$+Fe$_2$O$_3$+Cr$_2$O$_3$+NiFe$_2$O$_4$ |
| 500℃   | Fe$_3$O$_4$+Fe$_2$O$_3$+Cr$_2$O$_3$+NiFe$_2$O$_4$ |

Figure 5. XRD patterns of corrosion products formed on the surfaces of TP347H stainless steel corroded in corrosion reagent in air for 180h at 500-650℃.
3.3. Related Reactions during Corrosion

3.3.1. Corrosion at Lower Test Temperatures. At 500-550°C, the corrosion reagent hardly melts, as shown in Table 2. The main corrosion mechanism at these temperatures is chemical reaction. \( \text{O}_2 \text{ can directly react with the alloy through the solid corrosive medium to form a metal oxide layer, and SO}_4^{2-} \) in the sulfate can directly react with the alloy as follow reactions:

\[
\begin{align*}
x\text{M}(s) + (y/2)\text{O}_2(g) & \rightarrow \text{M}_y\text{O}_x(s) \quad (2) \\
x\text{M}(s) + y\text{SO}_4^2(g,l) & \rightarrow \text{M}_y\text{O}_x(s) + y\text{SO}_2 + y\text{O}_2 \quad (3)
\end{align*}
\]

where, \( \text{M} \) refers to metal elements (Ni, Cr, Fe, Nb, etc.), and the oxide film (mainly composed of NiO, Cr\(_2\)O\(_3\), Fe\(_3\)O\(_4\), NbO, etc.) can prevent further corrosion of the matrix. The Gibbs free energy of Cr\(_2\)O\(_3\) is the smallest, followed by Fe\(_3\)O\(_4\) and NiO. Therefore, the formation of Cr\(_2\)O\(_3\) was preferred in the corrosion process to effectively improve the oxidation resistance. A large amount of white floccule was found at the opening of the tubular resistance furnace, which was presumed to be crystallized after condensation of volatile NaCl and KCl. The melting point of metal chloride is relatively low. Under the combined action of pressure and high temperature, alkali metal chloride is easy to reach the saturated vapor pressure and volatilize. The outward diffusion of chloride accelerates the formation of acid gas in the corrosion reagent. The reaction is as follows:

\[
\begin{align*}
\text{RCl}(s, g) + \text{H}_2\text{O}(g) & \rightarrow \text{ROH}(s, g) + \text{HCl}(g) \quad (4) \\
4\text{HCl}(g) + \text{O}_2(s, g) & \rightarrow 2\text{Cl}_2(g) + 2\text{H}_2\text{O}(g) \quad (5)
\end{align*}
\]

where, \( \text{R} \) is the alkali metal elements (Na, K, Ca, Mg, etc.) existing in the corrosion environment. Alkali metal chloride volatilizes at high temperature to form HCl and Cl\(_2\), which accelerating the chlorination of alloy. Cl\(_2\) at high temperature has strong permeability and can directly react with metal through the ash layer and oxide film on the surface to form metal chloride with low melting point. These products volatilize and decompose with the increase of temperature and react with O\(_2\) to form corresponding metal oxides and Cl\(_2\). Abels and Strehblow [10] found that Cl\(_2\) was the most important corrosive medium rather than HCl in a short period of time in the HCl-O\(_2\) atmosphere. The corrosion mechanism of Cl\(_2\) and Cl-containing gas alloys is as follows [11-13]:

\[
\begin{align*}
\text{M}(s) + \text{Cl}_2(g) & \rightarrow \text{MCl}_2(s) \quad (6) \\
\text{M}(s) + 2\text{HCl}(g) & \rightarrow \text{MCl}(s) + \text{H}_2(g) \quad (7) \\
\text{MCl}(s) & \rightarrow \text{MCl}(g) \quad (8)
\end{align*}
\]

where, \( \text{M} \) is alloy elements in the material. The Gibbs free energy of CrCl\(_2\) is smaller than that of FeCl\(_2\) and NiCl\(_2\) [14], and was preferentially formed during corrosion. The partial pressure of chlorine will affect the formation of chloride. The vapor pressure of metal chloride with low melting point can be higher at lower temperature, and its volatilization ability increase gradually with the increase of temperature. In the process of outward diffusion of chloride, O\(_2\) concentration increases gradually with the increase of the distance from the alloy. Some chloride ions are selectively oxidized and generates Cl\(_2\) which reacts with the alloy surface, as shown in the following chemical equation:

\[
x\text{MCl}_2(g) + (y/2)\text{O}_2(g) \rightarrow \text{M}_y\text{O}_x(s) + x\text{Cl}_2(g) \quad (9)
\]

It should be noted that the partial pressures of different metal oxides are different. Among the metal elements in the alloy, the conversion of Cr chloride compounds into chromium oxide requires less partial pressure of O\(_2\), which leads to selective oxidation in high temperature environment. Although active oxidation caused by Cl\(_2\) and Cl\(_2\) can accelerate metal oxidation, the formation of Cr\(_2\)O\(_3\), Fe\(_3\)O\(_4\) and NiO with high temperature stability on the alloy surface can make TP347H stainless steel exhibit good corrosion resistance at relatively low temperatures [10, 12, 15, 16].


The melting points of Na$_2$SO$_4$ and K$_2$SO$_4$ are 884°C and 1067°C respectively, which can produce pyrosulfate (Na$_3$S$_2$O$_7$ and K$_3$S$_2$O$_7$) with relatively low melting points (400-590°C) [17] and adhere to the sample. Pyrosulfate can react with Fe$_2$O$_3$ to dissolve it, reducing the corrosion resistance of TP347H stainless steel. The chemical reactions are as follows:

\[ \text{R}_2\text{SO}_4(s, l) + \text{SO}_4(g) \rightarrow \text{R}_2\text{S}_2\text{O}_7(s, l) \tag{10} \]

\[ \text{R}_2\text{S}_2\text{O}_7(s, l) + \text{Fe}_2\text{O}_3(s) \rightarrow 2\text{R}_3\text{Fe}\text{(SO}_4\text{)}_3(s) \tag{11} \]

Where, $\text{R}_2\text{SO}_4$ is the alkali metal sulfate, such as Na$_2$SO$_4$ and K$_2$SO$_4$ in the corrosion reagents. It can be seen from the above reactions that the sulfate will form SO$_2$ and SO$_3$ at high temperature, which will react with alkali metal sulfate to form pyrosulfate, and then destroy the oxide film on the alloy surface.

### 3.3.2. Corrosion at Higher Test Temperatures.

At 600-650°C, the corrosive medium has completely melted, as shown in table 2. The liquid corrosive medium provides electrolyte for ion transfer, so that the corrosion mechanism changes to electrochemical corrosion. This can be proved by the severe intergranular corrosion of the alloy. Austenitic stainless steel (>0.03 wt.% C) begin sensitization and precipitate (Cr, Fe)$_2$C$_6$ at grain boundaries [18], resulting in the lack of Cr and Fe elements and promoting the diffusion of more C, Cr and Fe from ingrain to grain boundaries. As the diffusion rate of C is higher than that of Cr and Fe, it is always C that diffuses to the grain boundary first, promoting the diffusion of more C, Cr and Fe from ingrain to grain boundaries. As the diffusion rate of C is higher than that of Cr and Fe, it is always C that diffuses to the grain boundary first, so that Cr and Fe are consumed at the grain boundary. That can explain the phenomenon of Cr and Fe deficiency at the grain boundary. When Cr content is lower than 12wt.%, the alloy stops passive but forms a corrosion micro cell with grain boundary as anode and grain as cathode, which dissolves the grain boundary but the grain boundary is still in the passivation state [19]. In the chloride system, the degradation process of the alloy plays an important role [20, 21]. The dense oxide layer composed of Cr$_2$O$_3$, NiO and Fe$_2$O$_3$ formed on the alloy surface can effectively protect the matrix from further corrosion.

When the molten salt is completely molten, the surface corrosion products dissolve and O$_2$ diffuses inward to corrode the uncorroded matrix which accelerating the corrosion. Diffusion of chloride ions along grain boundaries and destruction of slatted grain boundary by oxides are the main causes of failure, and the reactions are as follows:

\[ \text{RCl}(s, l) + \text{Fe}_2\text{O}_3(g) + (1/2)\text{O}_2(g) \rightarrow \text{R}_3\text{Fe}_2\text{O}_4(s, l) + \text{Cl}_2(g) \tag{12} \]

\[ \text{RCl}(s, l) + (1/2)\text{Cr}_2\text{O}_3(s) + (5/4)\text{O}_2(g) \rightarrow \text{R}_3\text{Cr}_2\text{O}_4(s, l) + \text{Cl}_2(g) \tag{13} \]

where, RCl corresponds to NaCl and KCl in the mixed molten salt corrosion reagent. In addition to the deposited NaCl, the gaseous NaCl can also cause damage to Cr$_2$O$_3$.

The low melting point compounds produced by the reaction can destroy the oxide film. Alkali metal chlorides can react with other chlorides to form eutectic compounds with low melting points, while Fe and Ni sulfides also have low melting points and can form volatile eutectic compounds. Corrosion is accelerated by outward transfer of metal elements. It can be found that the temperature range of metal corrosion is consistent with the melting temperature range of alkali metal chloride, and it can be concluded that alkali metal chloride plays a decisive role in metal corrosion.

On the other hand, metal oxides can react directly with sulfate, thus destroying the compactness of oxide film. Take Fe$_2$O$_3$ and sulfate in TP347H stainless steel as an example, the following reactions occur:

\[ 3\text{R}_2\text{SO}_4(g, l) + \text{Fe}_2\text{O}_3(s) + 3\text{SO}_3(g) \rightarrow 2\text{R}_3\text{Fe}((\text{SO}_4)_3(s, l) \tag{14} \]

\[ 6\text{R}_2\text{SO}_4(g, l) + 2\text{Fe}_2\text{O}_3(s) + 6\text{SO}_3(g) + 3\text{O}_2(g) \rightarrow 4\text{R}_3\text{Fe}((\text{SO}_4)_3(s, l) \tag{15} \]

\[ 10\text{Fe}(s) + 2\text{R}_3\text{Fe}((\text{SO}_4)_3(s, l) \rightarrow 3\text{Fe}_2\text{O}_3(s) + 3\text{FeS}(s) + 3\text{R}_2\text{SO}_4(g, l) \tag{16} \]

where, $\text{R}_2\text{SO}_4$ is alkali metal sulfate (Na$_2$SO$_4$ and K$_2$SO$_4$). New sulfate produced by the reaction...
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