The Fouling Behavior of Steam Generator Tube at Different Positions in the High-Temperature Water

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Abstract: The fouling behavior of a steam generator (SG) tube was investigated at different positions after 500 h of immersion in high-temperature water. A triple-layer structure of fouling appeared at both the crevice position and the free span position, namely, the large, dispersedly distributed deposition layer on the top; the small and faceted outer layer; and the relatively continuous inner layer. There was no obvious positional effect on the thickness of the inner layer. However, in the crevice position, the density of the deposited particle and the thickness of the outer layer was much higher than those of the free span position. The tube support plate (TSP) made of 410 stainless steel contributed significantly to the fouling behavior of the SG tube in the crevice between the SG tube and the TSP.

Keywords: fouling behavior; crevice position; SG tube; high-temperature water; tube support plate

1. Introduction

As a clean energy source, nuclear power has attracted the attention of many researchers. However, nuclear leakage can cause catastrophic consequences such as the accidents that happened in Chernobyl in the USSR and Fukushima in Japan. The performance of the structural materials used in nuclear power plants (NPPs) plays a crucial role in the safety of NPPs. For a long time, researchers have been focusing on different kinds of degradation of nuclear structural materials, such as corrosion, fretting, aging, etc. [1–7]. After decades of study, the degradation mechanisms of materials have become well understood, and many countermeasures have been conducted to improve the safety of NPPs. However, in recent years, the fouling problem of steam generator (SG) tubes has raised the concerns of NPP operators. In terms of the structure of fouling, it can be roughly divided into two kinds: one is smooth and dense fouling, the other is rough and multi-hole fouling [8–10]. The fouling can enhance the thermal resistance of the tubes and, consequently, degrade the heat transfer efficiency. Fouling is usually caused by iron oxide deposition and/or soluble iron precipitation upon the surface of the SG tubes [11–17]. During the operation of the nuclear power plant, the water quality is monitored and is refreshed all the time. Although the water quality was well controlled, a certain amount of fouling also appeared at the sewage outlet of the NPP.

Lu and Xin [18,19] analyzed the fouling samples collected in the SG, and the results indicated that, among the chemicals, the main substance was magnetite (Fe₃O₄). Moreover, in the sludge found after the chemical cleaning of the SG tube and TSP, magnetite was also the main substance. It should be noted that the SG tube was made of nickel-based alloys (Alloy 690 or Alloy 600), which had good corrosion resistance, while the TSP was made of 410 stainless steel (SS) with inferior corrosion resistance. In the past, researchers mostly
focused on the growth of oxide films on the SG tube itself, but ignored the synergistic effect between the SG and the TSP on the formation of fouling, namely, iron ions dissolved from the TSP and deposited on the surface of the SG tube or accumulated in the crevice between the SG tube and the TSP.

In this study, the fouling layers formed on the SG tube at the crevice position and free span position were investigated by immersing the mockup of an SG tube and a TSP in high-temperature water. The fouling layer features were characterized by using various surface analysis technologies such as scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), Raman spectroscopy, and transmission electron microscopy (TEM). Focused ion beam (FIB)-SEM was used to observe the cross-section morphology and measure the thickness of the fouling layers. The related mechanism of fouling formation was also discussed.

2. Materials and Methods

The SG tube made of Alloy 690TT and the TSP made of 410 SS were provided by China Nuclear Power Engineering Co., Ltd., Shenzhen, China, and their compositions are displayed in Tables 1 and 2, respectively. Alloy 690TT was thermally treated at 715 °C for 11.5 h and then air cooled. The outer diameter of the SG tube was about 17 mm, and the tube wall thickness was about 1.2 mm. The SG tube was transversely cut into 50 mm lengths by wire cutting, and the 410 SS was fabricated into a clover shape, which was similar to that used in NPPs. After the fabrication, the specimens were ultrasonically cleaned by using ethanol and pure water for 30 min to remove the contamination on the specimens. Then, specimens were quickly dried by hot air. The SG tube and the TSP were assembled in the method as indicated in Figure 1. The whole assembled unit was composed of the SG tube and the TSP. The SG tube can be roughly divided into two parts, namely, the free span part and the crevice part. Two positions were selected for investigating the fouling behavior. One was in the free span part (position 1) and the other was in the crevice part (position 2). The distance between the SG tube and the TSP at position 2 was about 100 µm. The surface morphology of a position on the TSP (position 3) was also observed.

Table 1. Chemical composition of the 690TT used in this work (wt.%).

| Cr  | Mn  | Fe  | Si  | C   | P   | S   | Ni  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| 30  | 0.23| 8.88| 0.07| 0.02| 0.0011| 0.0055| Balance |

Table 2. Chemical composition of the 410 SS used in this work (wt.%).

| Cr  | Mn  | Ni  | Si  | S   | P   | C   | Fe  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| 13  | 1.1 | 3   | 0.56| 0.03| 0.04| 0.08| Balance |

Figure 2 shows the schematic diagram of the experimental apparatus. The volume of the autoclave used in this experiment was 10 L. The apparatus were roughly divided into two parts, including the high-temperature part and the room temperature part. The room temperature part (including a circulating low-pressure pump, a dissolved oxygen (DO) probe, a conductivity (Con) probe, and a pH probe) was used to adjust and monitor the water chemistry during the experiment. A sampling unit was also installed in the outlet of the room temperature part.

In order to simulate the water chemistry of the second side of an NPP, the experiment temperature was set to 290 °C, and the pressure was set to 10 MPa. The solution in this experiment was prepared with pure water and analytical grade reagent. First, the pure water removed from the pure water machine was pumped into the storage tank. Then, a certain amount of ammonia was added to the loop to adjust the pH value in the loop to 9.6–9.8, measured by the pH sensor. The DO was controlled to less than 5 ppb during the experiment by bubbling N₂ into the feed tank. The total immersion time was 500 h.
During the experiment, the water was extracted intermittently from the sampling unit to analyze the concentration of Fe, Cr, and Ni ions by using inductively coupled plasma atomic emission spectroscopy (Optima 8300, PerkinElmer, DC, USA). After the experiment, the assembled unit was taken out and quickly dried. The morphology of the fouling layer at different positions of the SG tube was observed by SEM, and the composition of the fouling layer was analyzed by EDS (M6480LV, Guo yi liang zi, SH, China). Raman spectroscopy was used to identify the phase structure of the oxide film at different positions. FIB-SEM was used to observe the cross-section morphology of the fouling film and measure the thickness of the fouling layer (SMI3050MS2, SEIKO, Tokyo, Japan). The cross-section morphology of the fouling film was analyzed by high-resolution TEM (JEM 3200fs, Japanese electronics, Tokyo, Japan).

Figure 1. Schematic diagram of the assembled unit used in the present work. Positions 1 and 2 are on the surface of the SG (Alloy 690TT) and position 3 is on the surface of the TSP (410 SS).

Figure 2. Schematic diagram of the experimental apparatus used in the present work.
3. Results

3.1. Water Analysis

Concentrations of Fe, Cr, and Ni ions of water extracted at different times from the water loop are shown in Table 3. It should be noted that the dashed line in the table indicates that the ion concentration is below the detection threshold. The thresholds for Fe and Ni ions are 0.01 mg/L and 0.02 mg/L, respectively. During the experiment, the ion concentration did not have an obvious alternation.

Table 3. The content of cations in the aqueous solution with different immersion times in this work (mg/L).

| Time, h | Fe, mg/L | Cr, mg/L | Ni, mg/L |
|---------|----------|----------|----------|
| 200     | 0.012    | 0.136    | —        |
| 300     | —        | 0.107    | —        |
| 400     | —        | 0.098    | —        |
| 500     | 0.012    | 0.123    | —        |

Note: —indicates that the concentration is below the detection threshold. The Fe threshold was 0.01 mg/L and the Ni threshold was 0.02 mg/L.

3.2. Surface Morphology

Figure 3 shows the surface morphologies of the fouling layer at different positions of the SG tube observed by SEM with a low magnification. Bright particles with a size of several hundred nanometers were dispersed on the surface at both positions. On the other hand, the number of particles at position 2 was much higher than that at position 1.

Figure 4 shows the high magnification images of the surface morphologies of the fouling layer at different positions of the SG tube marked in the rectangles in Figure 3. The bright particles observed in Figure 3 had faceted structures. Besides the bright particles, fine particles which were smaller than 100 nm could be observed on the surface. Due to the magnification in Figure 4 being very high for SEM (50k×), the image quality is not very good. In order to analyze the oxide layer in more detail, the cross-section of the fouling layer was observed by using TEM with a much higher magnification. Table 4 shows the EDS point analysis at different points in Figure 3b. Point 1 had a higher Fe content and a lower Cr content compared with point 2.

Figure 3. Surface morphologies of the fouling layer at different positions of the SG tube observed by SEM with a low magnification after 500 h of immersion at 290 °C. The magnified image of the rectangular area is shown in Figure 4; (a) position 1, (b) position 2.
Figure 4. The magnified surface morphologies indicted in the rectangle area of the fouling layer at different positions of the SG tube observed by SEM with a high magnification after 500 h of immersion at 290 °C; (a) position 1, (b) position 2.

Table 4. EDS point analysis at different points in Figure 3b.

| Element | Point 1 | Point 2 |
|---------|---------|---------|
| O       | 22.9    | 18.99   |
| Cr      | 16.28   | 29.17   |
| Fe      | 35.13   | 11.81   |
| Ni      | 25.7    | 40.03   |

Figure 5 shows the surface morphologies of the fouling layer at the TSP observed by SEM. As can be seen from the figure, the surface of the TSP is covered with lots of oxides with different sizes. The shapes of the oxides are very similar to the bright particles in Figures 3 and 4.

Figure 5. Surface morphologies of the fouling layer at the TSP observed by SEM after 500 h of immersion at 290 °C.
3.3. Raman Spectrum

Figure 6 shows the Raman spectrum results of the fouling at different locations of the SG tube after 500 h of immersion. It can be seen that the fouling at position 1 was mainly a spinel NiFe$_2$O$_4$ structure, as indicated by the main peak at 700 cm$^{-1}$, while in position 2, apart from the NiFe$_2$O$_4$ peaks, a weak peak around 575–600 cm$^{-1}$ was also detected. The Raman peak around 575–600 cm$^{-1}$ can be attributed to either the subpeak of NiFe$_2$O$_4$, or the main peak of Fe$_3$O$_4$. By comparing with the Raman spectrum in Figure 6a,b, the height ratio of the two peaks (700 cm$^{-1}$ and 575–600 cm$^{-1}$) changes, which implies a new phase possibly appears on the surface. It should be noted that the 575–600 cm$^{-1}$ peak in Figure 6b is still very weak, indicating that the amount of Fe$_3$O$_4$ is very small.

![Figure 6. Raman spectrum of the fouling at different positions of the SG tube after 500 h of immersion at 290 °C; (a) position 1, (b) position 2.](image)

3.4. Cross-Section Morphology

Figure 7 shows the cross-section morphologies of the SG tube at different positions after 500 h of immersion observed by FIB-SEM, and the specific locations of the fouling are marked in the rectangle. The top part is the C coating, which was intentionally deposited to protect the surface during ion sputtering, and the dashed line indicates the interface of the outer layer and inner layer. The fouling was mainly composed of two layers based on the image contrast, and the inner layer was relatively continuous at both position 1 and position 2, while the morphology of the outer layer at different positions showed some distinctions. At position 1, the faceted particles were randomly distributed, while at position 2, the faceted particles were mainly overlapped. It should be noted that in order to have a high magnification image for observing the details of the fine particles in Figures 3 and 4, the large and bright particles with a size of several hundred nanometers observed in Figures 3 and 4 were intentionally avoided.

Figure 8 shows the TEM image of the cross-section of the oxide in position 2 of the SG tube and an EDS line scan profile. According to the TEM and EDS results, it is clear that the oxide layer is divided into three layers, namely, inner layer, outer layer, and outermost layer. The thickness of the inner layer is about 50 nm, and highly Cr enriched. According to Figures 4b and 7, the outer layer corresponds to fine particles with a thickness of about 40 nm. The large particles in the outermost layer are about 120 nm. The inset gives the details of the interface of the outermost layer and the outer layer. A clear boundary can be observed, and the orientation of the two phases is mismatched, which means the outermost layer is deposited onto the outer layer. Therefore, the outermost layer is designated as the deposition layer.
4. Discussion

Xie and Zhang [20] reviewed the corrosion and deposition on the secondary circuit of steam generators and pointed out the structure of fouling on the SG tube can be classified into three layers, namely, inner layer (corrosion layer, which is formed by the diffusion of elements and the combination with oxygen atoms), outer layer, and deposition layer. This agrees well with the current observation, as shown in Figures 3, 4, 7 and 8.

Figure 9 shows the average thickness of the inner layer of fouling at different positions of the SG tube according to Figure 7a,b, and the error bar indicates the standard deviation. The thickness of the inner oxide film was about 40 nm and did not show an obvious difference in position. The inner layer was mainly caused by the inward diffusion of oxygen into the material in high-temperature and high-pressure water, namely internal oxidation [21,22], and this layer was usually deemed as the protective layer of the alloy against corrosion [23]. It should be noted that no difference in the inner layer indicated that the position (crevice position or free span position) did not affect the protectiveness of the SG tube.
Figure 9. The average thickness of the inner layer of fouling at different positions of the SG tube measured according to Figure 7a,b, and the error bar indicates the standard deviation.

Figure 10 shows the average thickness of the outer layer of fouling at different positions of the SG tube according to Figure 7a,b, and the error bar indicates the standard deviation. The thickness of the outer layer at position 1 was about 30 nm, while the thickness of the outer layer at position 2 was about 61 nm. According to the previous studies [21,24,25], this layer was formed due to the outward diffusion of Ni and Fe ions and then precipitation. Therefore, the growth of this layer was affected by the Ni and Fe concentration near the material/water interface. The big difference in the fouling thickness implies a big difference in ion concentration at the two positions. Although the Fe and Ni ion concentrations in the sampling water from the outlet were very low, as shown in Table 3. The local ion concentration, especially in the occluded area such as at position 2 in the present study, can soar to high values. The source of the ions on the outer part of the oxide has been studied by Carrette et al. [26] by using different kinds of autoclaves. They found that the oxide was formed of a rather continuous and uniform internal Cr-rich layer and an external layer, the composition, morphology, and microstructure of which depend on the test conditions and test duration. For example, oxide scales grown in a static autoclave contained higher concentrations of Fe and Ni than oxide scales grown in a recirculating titanium alloy autoclave. This means that Fe and Ni in the outer part are mainly from precipitation or deposition from the solution. It is reasonable to infer that the 410 SS contributes significantly to the increased ion concentrations in position 2, namely the crevice position, due to its low corrosion resistance.

The duration of the experiment was only 500 h, and the difference in thickness of the outer layer at different positions reached 30 nm (Figure 10). Besides, the density of the deposition layer also differed greatly (Figure 3). Considering that the lifespan of a nuclear power plant is tens of years, the difference in fouling layer thickness can be very large, which can influence the heat transfer efficiency. The corrosion degradation of the material is out of the scope of the current work. Actually, we did not observe obvious differences in the inner layer at different positions, which is usually deemed as the corrosion barrier layer. Another issue is that during the long-term operation of a nuclear power plant, the service conditions (working time-maintenance and stop periods) of the SG are relevant to diagnosing the corrosion situation. In particular, an alternative oxidizer to DO is γ-FeOOH, present in a rust layer. This is relevant to a corroding system having dry–wet periods. However, these issues are beyond the scope of the current work.
Figure 10. The average thickness of the outer layer of fouling at different positions of the SG tube according to Figure 7a,b, and the error bar indicates the standard deviation.

Another issue is the crevice corrosion between the TSP and the SG tube [24]. The crevice corrosion is mainly caused by the gradient of DO from the bulk to the crevice, which leads to the significant difference in corrosion products. However, in our work, the difference in the inner layer (corrosion layer) at different positions was not obvious, which indicated that the crevice corrosion does not play an important role in the corrosion degradation.

Galvanic corrosion happens when two different metal alloys with different corrosion potentials are connected and immersed into a solution. However, according to the study performed by Kim and Andresen [27], the corrosion potential of SS and nickel-based alloys were similar in deaerated high-temperature water, which was similar to our current work. Therefore, the galvanic corrosion has not been considered so far.

From the viewpoint of corrosion resistance, the large and dispersedly distributed particles with a size of several hundred nanometers hardly supplied any protection to the material [25,28]. However, these particles may play a key role in the fouling phenomenon. The Raman spectrum shown in Figure 6 also indicated that the fouling was mainly NiFe₂O₄, and a small amount of Fe₃O₄ can also be detected at position 2. As shown in Figure 3, the number of large particles at position 2 was much higher than that at position 1, indicating the corrosion product of 410 SS was one of the main sources of these particles.

Based on the above discussion, the scenario of fouling behavior of the steam generator tube at the crevice position and the free span position can be depicted as follows: a triple-layer structure of fouling can be observed at both positions, namely the large, dispersedly distributed deposition layer on the top; the small and faceted outer layer; and the relatively continuous inner layer. The inner layer was formed by the inward movement of oxygen, and no big difference in thickness could be observed at different positions, which indicates no obvious positional effect on corrosion protectiveness. However, the outer layer was formed by the outward movement of Ni and Fe and the subsequent precipitation on the surface. The release of soluble ions on the interface of the material and the solution can affect the formation and growth of this layer. 410 SS can be a soluble ion supplier, and is more efficient in the crevice position, resulting in a much thicker outer layer. The deposition layer should be the corrosion product of the other Fe-based materials in the autoclave, including 410 SS. Therefore, these particles can easily be deposited on the SG tube from 410 SS in the crevice position, leading to a much higher particle density at position 2.
5. Conclusions
The fouling behavior of a steam generator (SG) tube has been investigated at different positions after 500 h of immersion in high-temperature water. According to the present results, the following conclusions are drawn:

1. A triple-layer structure of fouling appeared at both the crevice position and the free span position, namely, the top layer composed of a large, dispersely distributed deposition layer; the small and faceted outer layer; and the relatively continuous inner layer.
2. The inner layer is highly Cr-enriched, while the outer and deposition layer is rich in Fe and Ni.
3. There was no obvious positional effect on the thickness of the inner layer. However, in the crevice position, the outer layer was much thicker than the inner layer. The crevice position also had a much higher density of the top large particles than the free span position.
4. The tube support plate (TSP) made of 410 stainless steel contributed significantly to the fouling behavior of the SG tube in the crevice between the SG tube and TSP.

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