Effects of yttrium on the oxidation behavior of 304 stainless steel with coating by laser remelting in high temperature water

Mengnan Liu¹, Dongxu Chen², Yanan Wang¹, Yanwen Zhou¹, Junwei Zhang¹ and Xiang Wang¹

¹ School of Material and Metallurgy, University of Science and Technology Liaoning, Anshan, 114051, People’s Republic of China
² CAS Key Laboratory of Nuclear Materials and Safety Assessment, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, 110016, People’s Republic of China

E-mail: dxchen11b@alum.imr.ac.cn

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Abstract

Effects of Y2O3 on the oxidation behavior of 304 SS with coating by laser remelting after exposed to 290 °C water containing 3 ppm O2 for 72 h were investigated. It was found that the corrosion behavior of remelted layer on 304 SS with the addition of Y2O3 was completely different from that of 304 SS and remelted layer on 304 SS without the addition of Y2O3. More Cr2O3 hematite oxides were formed around strengthening phase near the Y2O3 particles. Finally, the types of oxide formed on the surface of the remelted layer with the addition of Y2O3 were mainly Cr2O3 hematite oxides and a few of fine FeCr2O4 spinel oxides. The addition of Y2O3 in laser remelting layer can obviously change the corrosion behavior of 304 SS exposed to high temperature water and the related mechanisms are also discussed.

1. Introduction

304 stainless steel (SS) is an important structural material widely used in the nuclear power industry because of its excellent corrosion resistance. However, it is prone to localized attack such as stress corrosion cracking, pitting corrosion and crevice corrosion after long-term service [1–4]. It is generally believed that the oxidation process of SS in high temperature water has an important impact on corrosion resistance. Many researchers have investigated the structures of the oxide film formed on the 304 SS in high temperature water. It was found that the oxide film was mainly consisted of hematite particles and spinel particles. The different microstructures and morphologies in SS may lead to different oxidation process and then the corrosion behavior was affected.

The rare earth (Re) elements addition was widely used to improve the performance of the metal by surface modification [5–9]. It is believed that the grains in the modified layers were refined and the impurities were cleaned because of the addition of Re elements in the process of surface modification [10, 11]. Laser remelting is one of the effective surface modification methods because it can improve the wear resistance and the corrosion resistance of the matrix [12–16]. Moreover, there were very little effects on the matrix structure in the process of laser remelting because of the small heat affected zone [17, 18]. Therefore, more and more researchers focused their studies on the improvement of the performance of the metal by laser remelting [19, 20]. However, the effects of Re oxides additive on the corrosion behavior of 304 SS by laser remelting in high temperature water was not clear. It was necessary to develop the work to improve the corrosion resistance of 304 SS in high temperature water. The results are useful for future manufacture of the corrosion resistance coating on the surface of SS in the field of the nuclear materials.

Therefore, the purpose of the present work was to investigate the microstructures, morphologies and chemical composition of the oxide films formed on the surfaces of 304 SS and the remelted layers with or without Y2O3 by laser remelting after exposed to 290 °C water containing 3 ppm O2 for 72 h, using x-ray
diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), Raman spectrum and x-ray photoelectron spectrometer (XPS). The influences of laser remelting and the addition of Y₂O₃ on the microstructure and the oxidation behavior of 304 SS after the exposure test were carefully studied and the related mechanisms are also discussed.

2. Experimental

2.1. Materials and specimens
The chemical composition of the 304 SS used in the present work is listed in Table 1. The samples were cut from a mill-annealed plate and the size of the samples was 100 mm × 20 mm × 20 mm. The plate has been solution annealed at a temperature ranging from 1050 to 1150 °C. Before laser cladding, the samples were mechanically abraded with emery paper successively up to 1500 grit and washed ultrasonically in ethanol.

2.2. Experimental procedures
Laser remelting experimental was conduct on a high power cross-flow CO₂ laser system (HGL-6000). The main technical parameters of the laser system are listed in Table 2. The additive of coating was selected by the Y₂O₃ powder with the particle size of 45 μm ~ 180 μm and the Y₂O₃ powder was mixed uniformly with light-absorbing material. After that, the mixed powder was spread on the entire surface of the samples with a thickness of 2 mm. Then the sample was placed in a drying oven and dried at 50 °C for 8 h. The most optimum experimental parameters in the present work were chosen as follows. The laser power was 3 kW, the diameter of the laser spot was 3 mm, the scanning speed was 5 mm s⁻¹ and the protective gas was argon. Figure 1 is the schematic of the sampling position for exposure test after laser remelting. As shown in Figure 1, the samples with a size of 5 mm × 10 mm × 10 mm were cut from the sheet samples after laser remelting with or without Y₂O₃ and the observe directions were marked in the schematic. Then the samples were mechanically abraded with emery paper successively up to 3000 grit and were polished to bright specular surface. The samples were washed ultrasonically in ethanol and dried for the detection. After the laser remelting experimental, the surface morphologies of the remelted layers with or without addition of Y₂O₃ and 304 SS were examined by a scanning electron microscope (ΣIGMA HD, manufactured by ZEISS) with EDS (M6480LV, manufactured by JEOL).

After laser remelting experimental, the samples were cleaned and dried carefully. Then the exposure test of the samples in high temperature water was performed in an autoclave made of the 316 stainless steel with a volume of 2 L. The autoclave was also pre-oxidized before the exposure tests. Detailed information on the testing loop and control system has been described in the previous work [21]. The temperature and pressure of the exposure pure water were 290 °C and 8 MPa respectively. In order to understand the effects of dissolved oxygen (DO) to the samples after laser remelting with or without Y₂O₃, a high DO (3 ppm) was used for the exposure test. The experimental parameters of the exposure test are summarized in Table 3.

After the exposure test, the samples were cleaned and dried carefully. The surface morphologies of the oxide films formed on the remelted layers with or without Y₂O₃ and 304 SS were examined by a scanning electron microscope (ΣIGMA HD, manufactured by ZEISS) with EDS (M6480LV, manufactured by JEOL). The phase analyses of oxide films formed on the remelted layers with or without addition of Y₂O₃ and 304 SS were performed using an x-ray diffract meter (D/Max 2500 XRD) with Cu K alpha radiation and a custom Raman

### Table 1. Composition of 304 SS used in the present work (wt%).

| Element | C     | Si    | Mn    | S     | P     | Cu    | Co    | B     | Ni    | Cr    | Fe    |
|---------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|         | 0.035 | 0.66  | 1.88  | 0.005 | 0.023 | 1.00  | 0.06  | 0.0018| 9.27  | 18.65 | Bal.  |

### Table 2. Technical parameters of HGL-6000 laser system used in the present work.

| Technical name                  | Technical parameter |
|---------------------------------|---------------------|
| Laser model                     | Multimode           |
| Output power capacity           | ≤6000 W             |
| Unstable degree of power        | ±2%                 |
| Efficiency of photoelectric conversion | ≥16%               |
| Divergence angle of laser beam | ≤3 mrad             |
| Working gas                     | CO₂, N₂, He or Ar   |
| Scan rate                       | 0~10000 mm min⁻¹    |
system (XploRA PLUS). The Raman system contains a laser at 532 nm. The Raman shift range is $100 \sim 3000 \text{ cm}^{-1}$. The integration time used was 20 s. XPS measurements were performed with ESCALAB250 x-ray photoelectron spectrometer. The photoelectron emission was excited by monochromatic Al K$_{\alpha}$ source. Depth profiling was performed over an area of $2 \times 2$ mm under 2 keV Ar-ion sputtering. The sputtering rate was about 0.2 nm s$^{-1}$ (with reference to the Ta$_2$O$_5$ layer) [22].

3. Results

3.1. Morphologies and composition of the remelted layers

Figure 2 shows the surface morphology and the EDS results of the remelted layer with Y$_2$O$_3$. It can be found that the particle of Y$_2$O$_3$ was distributed at the grain boundary (figure 2(a)). It was also found that the contents of Mn element and Si element were increased around the particle of Y$_2$O$_3$ (figures 2(b) and (c)). Figure 3 shows the XRD patterns of 304 SS and remelted layers by laser remelting with or without Y$_2$O$_3$. Characteristic peaks of Y$_2$O$_3$ (at the position of $2\theta = 36^\circ$), Fe$_{19}$Mn (at the position of $2\theta = 44^\circ$ and $66^\circ$) and Fe$_{4}$Mn$_{77}$Si$_{19}$ (at the position of $2\theta = 42^\circ$ and $47^\circ$) were detected in the remelted layer with Y$_2$O$_3$. The XRD results showed that the particle of Y$_2$O$_3$ was added to the remelted layer. Moreover, the characteristic peaks of Fe (at the position of $2\theta = 43^\circ$, $51^\circ$ and $75^\circ$) was also be found, the XRD patterns of the remelted layer without Y$_2$O$_3$ were similar to that of 304 SS.

3.2. Morphologies and structures of the remelted layers after the exposure test

Figure 4 shows the SEM morphologies of the oxide films formed on 304 SS and remelted layers with or without Y$_2$O$_3$ after exposed to 290 $^\circ$C water containing 3 ppm O$_2$ for 72 h. The typical massive spinel oxide particles were observed on the surfaces of 304 SS and remelted layers with or without Y$_2$O$_3$. Moreover, fine spinel oxide particles and irregular hematite oxide particles were observed on the surfaces of 304 SS and remelted layers (figure 4). However, there were some differences between the morphologies of 304 SS and that of remelted layers. It was found that the massive spinel oxide particles were the main oxides and the fine spinel and irregular hematite particles were distributed on the surface of 304 SS sparsely (figure 4(c)). Meanwhile, the morphologies of the oxide film formed on the remelted layer without Y$_2$O$_3$ was similar to that of 304 SS (figure 4(b)). However, it can be found that the mainly oxides formed on the surface of remelted layer with Y$_2$O$_3$ were compact fine

| DO (ppm) | Pressure (MPa) | Time (h) | Temperature ($^\circ$C) | Conductivity of the inlet water (\(\mu S/cm\)) | Flow rate of the inlet water (L/h) |
|---------|---------------|--------|------------------------|---------------------------------|---------------------------------|
| 3       | 8             | 72     | 290                    | $65 \sim 85$                    | $8 \sim 9$                      |
spinel and irregular hematite particles (figure 4(a)). Moreover, it was also found that the amount of massive spinel particles formed on the remelted layer with Y2O3 was less than that of 304 SS.

Figure 5 shows the XRD patterns of the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y2O3 after exposed to 290 °C water containing 3 ppm O2 for 72 h. The positions of characteristic peaks suggest a hematite structure dominates at various sites. Characteristic peaks of hematite (at the position of $2\theta = 34^\circ$, $36^\circ$, $40^\circ$ and $44^\circ$) and spinel (at the position of $2\theta = 30^\circ$ and $36^\circ$) were detected in 304 SS and the
remelted layers with or without Y$_2$O$_3$. Figure 6 shows the Raman spectra of the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y$_2$O$_3$ after exposed to 290 °C water containing 3 ppm O$_2$ for 72 h. It was believed that the hematite structure was responsible for the Raman peaks at 221, 309 and 411 cm$^{-1}$ and the spinel structure was responsible for the Raman peaks at 698 cm$^{-1}$ [23–26]. It can be found that there were both the Raman peaks of hematite and spinel located at 698 cm$^{-1}$. So, the Raman peak at 698 cm$^{-1}$ was fitted (Figure 6(b)). It can be found that the intensity of Raman peaks of spinel oxide formed on the surfaces of the remelted layer with Y$_2$O$_3$ was weaker than that of 304 SS. Moreover, it was also found that the intensity of Raman peaks of hematite oxide formed on the surfaces of remelted layer with Y$_2$O$_3$ was stronger than that of 304 SS (Figure 6(a)). And the intensity of Raman peaks of hematite oxide and spinel oxide formed on the surfaces of remelted layer without Y$_2$O$_3$ was similar to that of 304 SS. This is consistent with the above results of the surface morphologies.

4. Discussion

The analyses of surface morphologies, EDS results (Figure 2) and XRD patterns (Figure 3) in the present work suggest that the remelted layer formed on the surface of 304 SS with or without Y$_2$O$_3$ have different morphologies and structures. During laser remelting, Y$_2$O$_3$ particles were accumulated on the grain boundaries. It is generally believed that Y$_2$O$_3$ has the effect of solution strengthening, and the Y$_2$O$_3$ particle can promote the formation of strengthening phase. Therefore, Mn atoms and Si atoms in the 304 SS matrix were aggregated around the Y$_2$O$_3$ particles and the Fe$_x$Mn$_y$Si$_z$ strengthening phases were formed during laser remelting. Moreover, according to the results of XRD patterns (Figure 3), it can be indicated that the distributions of Fe, Mn and Si elements in the remelted layer near the Y$_2$O$_3$ particles were changed because of the addition of Y$_2$O$_3$. These changings may lead to a completely different oxidation behavior of 304 SS remelted by laser with Y$_2$O$_3$ in high temperature water.

It is believed that the structure of the oxide film formed on the surface of 304 SS exposed to high temperature water have two layers [4, 22, 27, 28]. The inner layer was composed by fine spinel oxide particles and irregular hematite oxide particles and the out layer was composed by the massive spinel oxide particles. In the previous works [21, 22], it was indicated that the massive spinel oxides formed on the surface of 304 SS in high temperature water were mainly Fe$_3$O$_4$, the irregular hematite oxides were mainly (Fe, Cr)$_2$O$_3$ and the fine spinel oxides were mainly FeCr$_2$O$_4$. It is believed that the dissolution rates of Fe and Ni elements in 304 SS were both higher than that of Cr elements. The hematite particle was formed on the inner layer of oxide film on the surface of remelted layer and 304 SS. With the dissolution of Fe, many Fe ions were composed with OH$^-$ and H$^+$ to be Fe$_3$O$_4$ spinel oxide at the out layer. Other Fe ions were changed to irregular hematite oxides or fine spinel oxides at inner layer during exposure to high temperature water [21].

The XRD patterns (Figure 5) and Raman spectra (Figure 6) in the present work suggest that the oxide films formed on surfaces of 304 SS and remelted layers with or without Y$_2$O$_3$ are mainly have different structures. In the oxidation process of 304 SS, the oxide film was composed by hematite and spinel. It was indicated that there was the similar structure of oxide film formed on the surface of the remelted layer without Y$_2$O$_3$ and 304 SS.
However, in the oxide film formed on the surface of remelted layer with Y$_2$O$_3$, the content of spinel oxide was decreased. It suggests that the formation of spinel oxide formed on the remelted layer was hindered after the addition of Y$_2$O$_3$. According to the SEM morphologies (figure 4(a)), it can be thought that the Fe$_3$O$_4$ oxide at the out layer of oxide film was decreased. Moreover, the content of hematite oxide was increased and it was believed that the mainly composition of hematite oxide in Fe–Cr–Ni alloy is Fe$_2$O$_3$ or Cr$_2$O$_3$ [29]. It was generally believed that the XRD patterns and Raman spectra cannot give the accurate composition of the hematite oxides. Therefore, XPS was used to characterize the accurate composition of the oxide films.

Figure 7 shows the XPS spectra of Fe$_2$p$^{3}$/2 in the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y$_2$O$_3$. It was believed that the binding energy around 710.6 eV and 708.5 eV were characteristics of Fe$^{3+}$ and Fe$^{2+}$ [30]. It can be found from the XPS spectra that the peak area of Fe$^{3+}$ in the oxide film formed on the surface of remelted layer with Y$_2$O$_3$ was less than that of 304 SS. This suggested that the amount of Fe$_2$O$_3$ hematite oxide was decreased in the remelted layer with Y$_2$O$_3$. However, according to the

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**Figure 4.** SEM morphologies of the oxide films formed on 304 SS and remelted layers with or without Y$_2$O$_3$ after exposed to 290 °C water containing 3 ppm O$_2$ for 72 h (a) with Y$_2$O$_3$ (b) without Y$_2$O$_3$ (c) 304 SS.
morphologies (figure 4), XRD results (figure 5) and Raman spectra (figure 6) above, it was found that the amount of hematite oxides formed on the surface of remelted layer with Y2O3 was increased. Therefore, it can be thought that the increase of hematite oxides in remelted layer with Y2O3 was mainly caused by the formation of Cr2O3 hematite oxides. It is mainly because that in the process of oxidation, the formation of Fe3+ was hindered because of the strengthening phases of Fe19Mn and Fe4Mn77Si19 nearby the particles of Y2O3. So the amount of Fe2O3 was decreased and more Cr2O3 hematite oxides were formed. Moreover, figure 8 is the Cr element composition in the oxide films formed on 304 SS and the remelted layer with or without Y2O3. It can be indicated that at the corrosion process of 304 SS, the oxide film formed on the surface of the remelted layer with Y2O3 was mainly composed by Cr2O3 hematite oxide and fine FeCr2O4 spinel oxide because of the more difficult diffusion nearby the particles of Y2O3. So, higher content of Cr element was detected in the remelted layer with Y2O3. This result has further explanation that the mainly hematite oxides formed on the surface of remelted layer with Y2O3 was Cr2O3. Moreover, it can be found that the element composition of the Cr in the oxide films formed on the remelted layer without Y2O3 was similar to that of 304 SS and there were both increasing trends in the inner layer of oxide film. It was indicated that the structure of oxide film formed on the remelted layer without Y2O3 was similar to that of 304 SS, both of them have a bilayer oxide film after exposed to high temperature water. The result indicated the outer layer was composed by Fe3O4 spinel oxide, the inner layer was composed by Cr2O3 and Fe2O3 hematite oxides and fine FeCr2O4 spinel oxide. This is consistent with the above conclusion of the XRD patterns and Raman spectra.

Figure 9 shows the cross-section morphologies of the oxide films formed on remelted layers with or without Y2O3 after exposed to 290 °C water containing 3 ppm O2 for 72 h. Two types of oxide can be found in the remelted layer without Y2O3 after exposure test. Oxides of the inner layer were mainly small size hematite, and the massive spinel oxide was found above the hematite oxides, which was the mainly composed of the out layer (figure 9(a)). However, it was also found that the microstructures of the oxides formed on the remelted layer with Y2O3 were different from that of the remelted layer without Y2O3. Some small size hematite oxides and FeCr2O4 spinel oxides were formed around the Y2O3 particle and a few of Fe3O4 spinel oxides were formed beside the Y2O3 particle (figure 9(b)).

The above results and discussion in the present work suggest that Y2O3 by laser remelting has obviously influence on the process of the oxidation behavior of 304 SS exposed to 290 °C water containing 3 ppm O2 for 72 h. Figure 10 shows the schematics of the oxidation behavior of 304 SS and 304 SS by laser remelting with or without Y2O3 during exposed to 290 °C water containing 3 ppm O2 for 72 h in the present work. At the very early stage of oxidation on the surface of the remelted layer without Y2O3 and 304 SS, the dissolution rate of Fe was higher than that of Cr. Therefore, a Cr-rich layer was formed because of the massive dissolution of Fe ion (figure 10(a)). Then Cr ions on the surface were composed with OH\textsuperscript{-} and H\textsuperscript{+} to be the fine hematite nucleation in the high temperature water (figure 10(b)). With the dissolution of Fe, many Fe ions were composed with OH\textsuperscript{-} and H\textsuperscript{+} to be bulk Fe3O4 spinel oxide layer at the out layer. Meanwhile, the hematite oxides on the inner layer start to grow up. Irregular hematite oxides and the fine spinel oxides were formed on the inner layer of the oxide film (figure 10(c)). On the other hand, because of the addition of Y2O3, the Mn atoms and Si atoms were aggregated around the particles of Y2O3. It is believed that the Mn atoms in the 304 SS can compose with Fe
atoms and Si atoms easily [31]. Therefore, the strengthening phases of Fe$_x$Mn$_y$Si$_z$ were formed around the particles of Y$_2$O$_3$ (figures 2 and 3). At the very early stage of oxidation, because of existence of the strengthening phase, the diffusion of Fe atoms nearby the Y$_2$O$_3$ particles was decreased. Only a little of Fe atoms that far away from the strengthening phase were diffused into the solution (figure 10(d)). With the development of oxidation, the amount of dissolution of Cr was more than that of Fe, there were not enough Fe atoms provided. Therefore, the amount of Fe$_2$O$_3$ was decreased. More Cr ions was composed with OH$^-$ and H$^+$ and many Cr$_2$O$_3$ hematite oxides were formed around the particles of Y$_2$O$_3$ (figure 10(e)). With increasing exposure time, a little number of Fe$_3$O$_4$ spinel oxides were continuing formed on the surface outside the Mn/Si enrichment area. Moreover, the growth of Fe$_3$O$_4$ spinel oxides were hindered because of the massive Cr$_2$O$_3$ hematite oxides around the Y$_2$O$_3$ particles. Therefore, the size of Fe$_3$O$_4$ spinel oxides was small. Meanwhile, some Fe atoms were dissolved to Fe$^{2+}$ and they were combined with Cr$^{2+}$ and OH$^-$ to be FeCr$_2$O$_4$ spinel oxide at the inner layer of oxide film. Finally, the types of oxide film formed on the surface of the remelted layer with Y$_2$O$_3$ were many Cr$_2$O$_3$ hematite oxides and a few of fine FeCr$_2$O$_4$ spinel oxides (figure 10(f)).

Figure 6. Raman spectra of the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y$_2$O$_3$ after exposed to 290 °C water containing 3 ppm O$_2$ for 72 h.
Figure 7. XPS spectra of Fe2p3/2 in the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y2O3 after exposed to 290 °C water containing 3 ppm O2 for 72 h (a) with Y2O3, (b) without Y2O3, (c) 304 SS.

Figure 7. XPS spectra of Fe2p3/2 in the oxide films formed on the surfaces of 304 SS and remelted layers with or without Y2O3 after exposed to 290 °C water containing 3 ppm O2 for 72 h (a) with Y2O3, (b) without Y2O3, (c) 304 SS.
5. Conclusions

Effects of Y_2O_3 on the oxidation behavior of 304 SS with coating by laser remelting in high temperature water were investigated. The following conclusions could be drawn based on the present results.

The corrosion behavior of remelted layer on 304 SS with the addition of Y_2O_3 was completely different from that of 304 SS and remelted layer on 304 SS without the addition of Y_2O_3. At the very early stage of oxidation, because of existence of the strengthening phase, the diffusion of Fe atoms nearby the Y_2O_3 particles was
decreased. There were not enough Fe atoms provided and the amount of Fe$_2$O$_3$ was decreased. More Cr$_2$O$_3$ hematite oxides were formed around the particles of Y$_2$O$_3$. With increasing exposure time, a little number of Fe$_3$O$_4$ spinel oxides were continuing formed on the surface outside the Mn/Si enrichment area. Moreover, the growth of Fe$_3$O$_4$ spinel oxides were hindered because of the massive Cr$_2$O$_3$ hematite oxides around the Y$_2$O$_3$ particles. Some small Fe$_3$O$_4$ spinel oxides were formed. Finally, the types of oxide film formed on the surface of the remelted layer with the addition of Y$_2$O$_3$ were mainly Cr$_2$O$_3$ hematite oxides and a few of fine FeCr$_2$O$_4$ spinel oxides.

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ORCID iDs

Dongxu Chen  https://orcid.org/0000-0002-8854-9262

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