Abstract

The kinetics of the Cr₂O₃-based scale oxidation and volatilization were studied in the presence of water vapour (H₂O). A commercial Cr₂O₃-based scale forming Type 310S stainless steel was examined at the ambient pressure (0.1 MPa) and 550 ºC in relatively low and high H₂O containing environments of air-10% H₂O and air-70% H₂O, respectively. The increase in the partial pressure of H₂O (pH₂O) from 10% to 70% resulted in transition of the oxidation and volatilization kinetics from the parabolic rate law in air-10% H₂O to the paralinear rate law in air-70% H₂O. The kinetics transition was attributed to the increase in the Cr loss rate from the base scale after coupons exposure in air-70% H₂O. The significant role of Mn alloying element in the base scale protectiveness was discussed in the context of the Cr₂O₃-based scale stability.

Keywords: Kinetics, Cr₂O₃-based scale, Oxidation, Volatilization, Water vapour, Type 310S stainless steel, Alloing element
1. Introduction

High-temperature oxidation and oxide scale volatilization in oxygen (O_2) and H_2O containing environments are unavoidable phenomena that limit the performance of the corrosion-resistant alloys used as structural components for steam-generating applications such as: steam boiler power plants, solid oxide fuel cells, nuclear reactors and geothermal power plants.\textsuperscript{1-8} One method to increase the steam oxidation resistance of the alloy structural components is to modify the composition to form a protective external Cr_2O_3-based scale.\textsuperscript{9-12} It is well-known that increasing an alloy Cr-content can enhance the formation of a protective external Cr_2O_3-based scale.\textsuperscript{9-12} Even though the increased Cr content in an austenitic Fe-Cr-Ni alloy is desirable from the Cr_2O_3-based scale formation perspective, one major concern exists. The Cr_2O_3 scale is known to form volatile Cr species such as CrO_2(OH), Cr(OH)_3, CrO_3, CrO(OH)_2 and CrO_2(OH)_2.\textsuperscript{13-16} Thermodynamic calculations of volatile Cr species partial pressures have shown that the most dominant phase that is responsible for oxide scale breakaway at temperatures below \( \sim 900 \) °C is Cr oxy-hydroxide: CrO_2(OH)_2 (equation 1), whereas at temperatures above \( \sim 900 \) °C, CrO_3 and CrO_2(OH) species form significantly as well.\textsuperscript{14}

\[
\frac{1}{2}Cr_2O_3(g) + H_2O(g) + \frac{3}{4}O_2(g) = CrO_2(OH)_2(g)
\]  

(1)

It is known that the high Cr-containing alloys oxidation and formation of an external protective Cr_2O_3-based scale follows a parabolic rate law as such scale reduces further ion diffusion, therefore decreases the oxidation reaction rate.\textsuperscript{1} Oxidation kinetics of the Cr_2O_3-based scale forming alloys in the presence of H_2O and any possible transition in the reaction kinetics during scale volatilization, however, is not well understood yet.
The volatile species during the Cr₂O₃-based scale volatilization can be carried away with the oxidizing environment flow and deplete the scale from Cr.¹⁷ Further scale protectiveness then relies on the Cr supply from the alloy substrate to the scale.¹⁷ As a result, Fe present at the oxide/alloy interface could diffuse through the vacant lattice sites of Cr in the oxide scale and enhance the formation of a less protective, more Fe-rich Cr₂O₃-based scale.¹⁸,¹⁹

The oxidation and volatilization rates of the Cr₂O₃-based scale is highly dependent on the oxidizing parameters, with higher $p_{H₂O}$, higher temperature, higher dissolved oxygen content, and increased environment flow rate, increasing the oxidation and volatilization rates.²⁰ In the case where the environment is not purely steam (e.g. air- $H₂O$ mixture), the oxidation reaction highly depends on the vol.% $O₂$ and $H₂O$ that exists in the mixture.¹ The calculated vapour pressures of CrO₂(OH)₂ for different total steam pressures and air-10% $H₂O$ mixture as a function of temperature shows that a significantly high vapour pressure of CrO₂(OH)₂ is produced in air-10% $H₂O$ mixture.¹ It is also shown that the increase in the vol.% $H₂O$ increases the $p_{H₂O}$ that could encourage the Cr₂O₃-based scale breakaway.¹,²⁰

In the current study, the high-temperature oxidation behavior of the commercial Type 310S stainless steel (~ 24 wt% Cr) is examined in 0.1 MPa and 550 ºC. The 550 ºC temperature is used as it is in the operating temperature range for many $H₂O$ containing environments such as those in steam turbines, low-temperature solid oxide fuel cells, and fuel cladding materials in supercritical water cooled reactors.²¹-²³ In addition, the importance of the oxidation and volatilization kinetics at the relatively lower 550 ºC temperature is often underestimated and requires further consideration. To examine the effect of $p_{H₂O}$ on the Cr₂O₃-based scale stability and kinetics of the Cr₂O₃-based scale oxidation and volatilization, two environments were chosen with relatively low (air- 10% $H₂O$) and relatively high (air- 70% $H₂O$) vol.% $H₂O$. A more complete physical
description of the Cr₂O₃-based scale volatilization mechanism using the targeted commercial alloy and advanced electron microscopy characterization techniques was obtained. The kinetics of oxidation and volatilization in the two environments was also discussed.

2. Experimental procedures

2.1. Material

Commercial plate of Type 310S stainless steel (Cr: 24.3 wt%, Ni: 19.5 wt%, C: 0.06 wt%, Mn: 1.0 wt%, Si: 0.8 wt%, Mo: 0.8 wt%, and Fe: balance) was solution annealed at 1150 °C for 1 h followed by water quenching to room temperature. Rectangular test coupons with the dimensions of 15 mm × 9.2 mm × 1.5 mm were prepared from the plate using a diamond wheel saw. The provided coupons were then ground to a 400 grit surface finish using SiC abrasive papers with H₂O as a lubricant and cleaned in ethanol. Prior to the coupons exposure in the oxidizing environments, their dimensions and weight were measured using digital caliper and analytical balance, respectively.

2.2. Wet oxidation testing

A 120 cm quartz tube with the inner diameter of 11 mm was fitted inside a 80 horizontal furnace. The temperature inside the quartz tube and at the center of its length was kept at 550 ± 3 °C. Two exposure conditions were chosen to examine the effect of $p_{H₂O}$ on the scale stability: air-10% H₂O and air-70% H₂O. Deionized water was first heated inside a water flask to 46 °C for the air-10% H₂O test and 90 °C for the air-70% H₂O test. An air stream was flushed into the heated deionized water flask and the air-x vol.% H₂O mixture flew into the quartz tube with the flow rate of 200 mL.min⁻¹ (velocity of 4.2 cm.s⁻¹). Five coupons (per exposure time and exposure condition) were then inserted in the center of the quartz tube parallel to the flow direction. Exposure times
considered were 100 h, 250 h, and 500 h. All coupons weights were measured after exposure using the analytical balance with the precision of ± 0.0001 g in order to obtain the amount of weight gained due to oxidation.

2.3. Oxide scale characterization

Scanning electron microscopy (SEM) was initially used for the analysis of the nature and morphology of the oxide scales formed on the coupons in plan-view. This was done using a JEOL JAMP-9500F AUGER/FE-SEM microscope equipped with a Schottky Field Emission Gun (FEG) filament and an integrated Oxford Synergy system with INCA EDS X-ray micro-analysis using an accelerating voltage of 10 kV and a working distance of ~ 20-23 mm. Site-specific TEM thin film specimens were prepared from the coupons exposed for 500 h (the longest exposure time) in each of the environments considered. This was done using Zeiss NVision 40 focused ion beam (FIB) with a Ga ion beam voltage of 30 kV, a current beam of 80 pA and a working distance of 5.6-5.9 mm. To protect the oxide scales during the ion milling, a ~ 200 nm thick C layer first and a ~ 2 μm thick W strap layer next, were deposited on the coupons. TEM analysis was conducted using JEOL 2010F TEM/STEM operating at 200 kV. High resolution TEM (HRTEM) images were obtained from the base scale and the alloys substrate to characterize the structure of the phases present. The very thin oxide scales formed were characterized using Electron Energy Loss Spectroscopy (EELS) with a dispersion of 0.2 eV/channel while the microscope was operated in the high angle annular dark field (HAADF)-Scanning (S)TEM mode. EELS qualitative analysis was obtained by comparing the intensity of a specific energy loss (eV) window to each one of the characteristic edges that is observed in a typical EELS spectrum and the position of these edges as compared to reference materials. For characterizing different types of oxides formed on an austenitic Fe-Cr-Ni alloy, O-K, Cr-L\textsubscript{2,3}, Fe-L\textsubscript{2,3}, and Ni-L\textsubscript{2,3} edges can all be used for oxide
analysis, however, the O-K edges typically show more variation in each oxide type. Figure 1 shows the reference EELS spectra that were used in the current study as re-plotted using the Plot Digitizer software. The red arrow in Figure 1(a) shows the characteristic feature of a spinel oxide structure: a shoulder peak at ~ 550 eV.

3. Results and discussion

Figure 2 shows the weight gain comparison of coupons exposed in 0.1 MPa and 550 °C flowing air-10% H₂O and air-70% H₂O for up to 500 h. The error bars show the 95% confidence interval for 5 coupons per exposure condition. The weight gain data for the air-10% H₂O test, considering the error bars did not show a noticeable change over the exposure times considered. The oxidation kinetics followed the parabolic rate law with the alloy parabolic rate constant ($k_p$) of $\sim 1.7 \times 10^{-12} \text{ g}^2/\text{cm}^4.\text{s}$. The weight gain data for the air-70% H₂O showed a paralinear kinetics, that is the combination of an initial parabolic kinetic rate law ($k_p$ of $\sim 3.5 \times 10^{-13} \text{ g}^2/\text{cm}^4.\text{s}$) followed by a linear kinetic rate law with the alloy negative linear rate constant ($k_l$) of $\sim 2 \times 10^{-12} \text{ g/cm}^2.\text{s}$. The values measured in this study are comparable with those published in the literature for high Cr-containing Fe-Cr-Ni alloys exposed in air and/or in the presence of H₂O. The $k_p$ value in the air-70% H₂O test was lower than that of the air-10% H₂O due to the lower $p_{O_2}$ (required for the oxide formation) in this environment.

Figure 3 shows plan-view secondary electron images of the coupons surfaces after each of the exposure times and conditions considered. The coupons surfaces in all cases consisted of fine-grained oxide scale. The grinding lines from the surface preparation prior to exposure were noticeable on the surface, implying that the scale had very small (~ few hundred nanometers) thickness. It should be mentioned that small bright particles were randomly detected on the coupons surface after 250 h exposure in air-70% H₂O (Figure 3[b]). The SEM-EDS spot analysis
showed that these particles were enriched in Mn and O. The presence of the Mn-enriched oxide particles on the coupon surface could be as the result of the outermost oxide layer breakaway. The similarity in the morphology of the scale formed on coupons in all the exposure times considered implies that the oxidation and volatilization rates in the two environments at 0.1 MPa and 550 ºC is relatively low.

Figure 4(a) and Figure 4(b) show the HAADF-STEM cross section images of coupons exposed for 500 h (the longest time) in air-10% H₂O and air-70 % H₂O, respectively. The black and white areas on top of the base scale are the deposited C and W layers, respectively during FIB milling. In both cases, a uniform base scale was formed on the substrate. Recrystallized grains from the surface deformation during the surface preparation were detected below the scale/alloy interface. The base scale thickness in air-10% H₂O was ~ 68 nm compared with that of the ~ 50 nm in air-70% H₂O. The relatively similar scale thickness and small weight gain difference in the two environments as was seen in Figure 2 suggest the slow oxidation and volatilization rates at 0.1 MPa and 550 ºC.

Figure 5 shows the cross-section HAADF-STEM image and Fe, Cr, Mn, Ni, and O EELS elemental maps of the base scale formed after 500 h exposure in air-10% H₂O. The black and white areas on top of the base scale in the HAADF-STEM image are the deposited C and W layers, respectively during FIB milling. The base scale was enriched in Cr and Mn. Figure 6 shows the corresponding EELS spectra from Areas 1, 2, and 3 in Figure 5. The base scale was characterized as Cr₂O₃ layer with small Fe dissolved in it (Areas 1 and 3). The Mn-enriched layer on top of the Cr₂O₃ layer was characterized as MnCr₂O₄ (Area 2). The formation of a spinel MnCr₂O₄ layer on the Cr₂O₃ layer is well established in Mn containing stainless steels because of the high Mn diffusivity in the Cr₂O₃ layer. It has been shown that a MnCr₂O₄ cap on the Cr₂O₃ layer could
positively affect the volatilization rate of the base scale in that the Cr loss rate from a MnCr$_2$O$_4$ layer is much less than that of the Cr$_2$O$_3$ layer.$^{34}$

**Figure 7** shows the cross-section HAADF-STEM image and the Fe, Cr, Mn, Ni, and O EELS elemental maps of the base scale formed after 500 h exposure in air-70% H$_2$O. The corresponding EELS spectra from areas in the HAADF-STEM image are shown in **Figure 8**. The black and white areas on the base scale in the HAADF-STEM image are the deposited C and W layers, respectively during FIB milling. The base scale was enriched in Cr and Mn. The base scale was characterized as Cr$_2$O$_3$ layer (Area 4). The Mn-enriched layer on top of the Cr$_2$O$_3$ layer was identified as MnCr$_2$O$_4$ (Area 5). Area 6 contained more Mn, and was identified as MnO.

It has been shown that Cr loss in the presence of H$_2$O, could also occur from a MnCr$_2$O$_4$ layer (equation 2)$^{34}$

$$\frac{1}{2} MnCr_2O_4(s) + H_2O(g) + \frac{3}{4} O_2(g) = CrO_2(OH)_2(g) + \frac{1}{2} MnO(s) \tag{2}$$

The physical evidence of MnO deposition on the Cr$_2$O$_3$ layer as was seen in this study (**Figure 3(b)** and **Figure 7**), after 500 h exposure of coupons in air-70% H$_2$O, supports the aforementioned thermodynamic theory.

**Figure 9** shows the cross-section HAADF-STEM image of the alloy after 500 h exposure in air-70% H$_2$O and the EELS spectra from the corresponding points shown in the image. Points 1-6 EELS spectra were obtained where the Cr loss from the MnCr$_2$O$_4$ resulted in the MnO deposition on the oxide surface and transformation of MnCr$_2$O$_4$ into a Mn$_x$Cr$_{2y}$O$_4$ layer. Points 7-12 EELS spectra were obtained adjacent to points 1-6 for comparison. In points 1-6, the Cr-L$_{2,3}$ peaks in the base scale decreased from the alloy-oxide interface to the oxide-air-70% H$_2$O interface, which confirmed the Cr depletion from the oxide surface due to base scale volatilization. In the
meanwhile, the Mn-L₂,₃ peak increased from the alloy-oxide interface to the oxide-air-70% H₂O interface (points 1-6). Complete disappearance of Cr-L₂,₃ peaks while Mn-L₂,₃ peaks were still present at the oxide-air-70% H₂O interface, confirmed the volatilization of Cr from a MnCr₂O₄ layer and deposition of MnO, which is in agreement with the previously published thermodynamic theory.34 The adjacent points 7-12 on the other hand, showed that the MnCr₂O₄ layer stayed intact on top of the Cr₂O₃ layer. Very small amount of Fe was dissolved in the MnCr₂O₄ layer. This suggests that the Cr loss from the MnCr₂O₄ layer and deposition of MnO, also Fe dissolution in the MnCr₂O₄ layer on the surface, are localized phenomena.

*Figure 10* shows the HRTEM image of the alloy/oxide interface after 500 h exposure of coupons in both environments. The EELS spectra corresponding to the amorphous layers are shown in *Figure 10(b)* and *Figure 10(d).* In both environments after 500 h exposure, a ~ 5 nm thick oxide layer was seen at the scale/alloy interface which was characterized as SiO₂ layer. Formation of a SiO₂ layer below the base scale in stainless steels during oxidation is very common. The thickness of this layer, however, depends on the Si concentration in the alloy, temperature, pressure and exposure time.35 It is known that the SiO₂ layer might positively enhance the oxidation rate of the alloys at high temperatures by reducing ions diffusion through the scale.35 The protective SiO₂ layer thickness is believed to be ~ 1 μm therefore the ~ 5 nm thick SiO₂ layer observed in this study does not seem to play a role in protectiveness of the alloy against high-temperature oxidation.35

*Figure 11* shows the summary of the scales characterization after 500 h exposure in 0.1 MPa and 550 °C air-10% H₂O and air-70% H₂O. The ~ 5 nm SiO₂ layer underneath the base scale is not shown and the scales thicknesses are not set to scale. In both cases, a MnCr₂O₄ oxide cap formed on top of the Cr₂O₃ layer. The Cr loss rate is known to be much less from the MnCr₂O₄ layer
compared with that of the Cr$_2$O$_3$ layer, which is responsible for the small weight gain values obtained in this study and effective protection of the Cr$_2$O$_3$-based scale against breakaway. With increase of the $p_{H_2O}$, the kinetics of the reactions changed from parabolic in air-10% H$_2$O to paralinear in air-70% H$_2$O. While the Cr$_2$O$_3$-based scale remained protected against volatilization in both environments, the Cr loss from the MnCr$_2$O$_4$ cap was higher in air-70% H$_2$O. The physical evidence of MnO deposition as a result of Cr loss from the MnCr$_2$O$_4$ layer (equation 2, Figure 3(b) and Figure 7) in air-70% H$_2$O environment, confirms that the higher $p_{H_2O}$ increased the Cr loss rate from the MnCr$_2$O$_4$ layer. Not to mention, further study on the Mn-O enriched particles observed in Figure 3(b) is suggested. It should be noted that the Mn and Cr concentration below MnO was different than the areas without MnO. This is shown in Figure 11 as Mn$_x$Cr$_{2y}$O$_{4z}$. Results from this work show that in the presence of the MnCr$_2$O$_4$ layer in the 0.1 MPa pressure and 550 ºC air-10% H$_2$O and air-70% H$_2$O, the volatilization rate of the base scale was reduced enormously and Cr$_2$O$_3$-based scale breakaway and Fe-rich oxide nodules formation was inhibited.

4. Conclusions

The effect of H$_2$O presence on the oxidation and volatilization mechanisms of commercial Type 310S stainless steel was examined in air-x vol.% H$_2$O at 0.1 MPa and 550 ºC. Comparison of the base scales formed in air-10% H$_2$O and air-70% H$_2$O environments, showed that the kinetics of oxidation was different in the two environments: parabolic in air-10% H$_2$O and paralinear in air-70% H$_2$O. The transition of oxidation kinetics was attributed to the higher volatilization rate of the scale formed in air-70% H$_2$O. It was shown that the presence of a MnCr$_2$O$_4$ on top of the Cr$_2$O$_3$-based scale reduced the Cr$_2$O$_3$ layer volatilization rate and inhibited the scale breakaway from formation of Fe-enriched nodules in both environments for up to 500 h. The relatively higher Cr loss rate from the MnCr$_2$O$_4$ in the higher $p_{H_2O}$ environment (air-70% H$_2$O) however, was
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responsible for the transition of kinetics from parabolic to paralinear rates, therefore, the vapour pressure of the volatile species for the minor alloying elements are important and must not be underrated.

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