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Long term isothermal and cyclic oxidation behavior of yttrium dioxide (Y_2O_3) coated Fe-20Cr alloy was studied. Oxidation tests were carried out in air for periods of up to 200 hours at 1000°C and the oxidation behavior of the alloy was evaluated gravimetrically. The Y_2O_3 coating reduced markedly the mass gain of the alloy. Energy dispersive spectroscopic analysis of scale sections on oxidized Y_2O_3 coated specimens revealed a Cr rich layer close to the metal/oxide interface and the presence of Y and Cr near the oxide/air interface, indicating diffusion of Cr through the Y_2O_3 layer. Further evidence of this diffusion was seen as change in colour of the Y_2O_3 coating from white to green after prolonged oxidation. X-ray diffraction analysis of the scale revealed perovskite-type YCrO₃. This study attributes the increased oxidation resistance of the Y_2O_3 coated Fe-20Cr alloy to increased oxygen ion diffusion through the Cr_2O_3 scale due to YCrO₃ formation and grain boundary segregated Y ion blocking alloy cation diffusion.

Key words: Iron-chromium alloy, Yttrium dioxide, Coating, Oxidation behavior, High temperature

INTRODUCTION

Metallic materials for use at high temperatures are usually iron, nickel or cobalt based alloys. The oxides formed on these alloys in oxidizing environments are not sufficiently protective above 500 °C. Hence, chromium and/ or aluminium are added to these alloys to establish more protective oxides (scales) of chromium dioxide or alumina. Protection of the alloys by the chromium dioxide or alumina layer can be compromised by spalling or cracking of the oxides, often caused by growth or thermal stresses. The use of reactive elements, especially rare earths (RE) to improve high temperature oxidation resistance of chromium dioxide and alumina forming alloys is guite well documented. 1-14 The improvements are in the form of reduced oxidation rates and increased scale adhesion. The RE can be added to the alloy in elemental form or as oxide dispersions. It can also be applied as an oxide coating to the alloy surface. 3-7,15 Various mechanisms have been proposed to explain the effect of reactive elements in improving oxidation resistance and the most widely accepted mechanism attributes it to segregation of the reactive elements to the interface or to the oxide scale grain boundaries and blocking of Cr ion diffusion though the oxide scale 8-11 Studies carried out by Seo et al., about the effect of addition of Ce, La and Y to a Fe-22Cr-0.5Mn alloy on the oxidation behavior of the alloy at 800 °C, indicated that Y was the most effective element to reduce the growth rate of the oxide scale. 12 In recent years a number of studies have been carried out to exploit the benefits of rare earth additions on oxidation behavior of chromium dioxide and alumina forming alloys. 13,14,16-21 Most studies reporting the reactive element effect in chromium dioxide forming alloys were carried out at or below 800 °C. A limited number of

studies have reported this effect at temperatures above 950 °C, probably attributable to formation of volatile CrO_3 . To throw more light on reactive element effect at a higher temperature and after prolonged periods when CrO_3 formation is likely, this paper reports the effect of Y_2O_3 coating on oxidation behavior of Fe-20Cr alloy at 1000 °C for 200h.

METHODS AND MATERIALS

The Fe-20Cr alloy (composition in wt-% - 19.7Cr, 0.79Mn, 0.02Si and balance Fe) was prepared in a vacuum induction furnace and the cast ingot was forged at 980 °C. Rectangular specimens with total area of approximately 4 cm² were cut from the forged bar and its surfaces prepared by grinding with SiC paper to mesh size 220. The specimens were then degreased with acetone, dried and weighed in an analytical balance. A dispersion of Y₂O₂ powder (99.99% purity) in ethanol was prepared and sprayed with an airbrush on to the different sides of the specimens at room temperature. The Y₂O₂ film coated specimens were then dried in air. The coating did not peel or dislodge indicating adequate adhesion. The coated specimens were again weighed. All the oxidation measurements were carried out in triplicate. Two sets of oxidation measurements were carried out. The first consisted of heating the bare and Y₂O₂ coated specimens in air in a muffle furnace at 1000 °C for 200 h. The mass gain per unit area of the different specimens was determined. The second set of measurements was cyclic oxidation wherein each cycle consisted of heating the bare and Y2O3 coated specimens for 7 h at 1000 °C followed by cooling to room temperature. This was repeated 15 times. The mass gain per unit area of the bare and Y2O3 coated specimens was recorded after

each cycle. The oxidation products on the specimen surfaces were examined using a scanning electron microscope and micro-regions on the oxidised specimen surfaces analyzed using an energy dispersive spectrometer (EDS). The chemical composition of the oxide scales was determined by x-ray diffraction (XRD) analysis.

RESULTS AND DISCUSSION

The average mass gains of the bare and Y_2O_2 coated Fe-20Cr alloy specimens after oxidation at 1000 °C for 200 h were 1.74 and 0.52 mg/cm² respectively. The mass gains were due to formation of oxides of elements from the alloy and a marked decrease in mass gain of the Y₂O₂ coated specimen is evident. The morphological features of the three bare specimen surfaces after oxidation were identical. The surface of one of these specimens is shown in Fig.1a and it reveals undulating mounds incrusted with fine cuboids of a mixture of oxides. An EDS spectrum of this surface is shown in Fig.1b and it reveals the presence of Cr and Mn, besides oxygen. A cross section of the same specimen is shown in Fig. 1c and it reveals voids at the metal/oxide interface and near the oxide/air interface. EDS analyses at regions 1 and 2 in Fig.1c indicate Fe, Cr and Mn in quantities shown in Table 1. Region 2, close to the oxide/air interface has a high Mn content.

The XRD spectrum of the oxide on the oxidised bare specimen is shown in Fig. 2 and it reveals the oxide phases Cr_2O_3 and $MnCr_2O_4$. The EDS and XRD analyses data indicate that the oxide layer on the oxidised bare specimen consists mainly of Cr_2O_3 with $MnCr_2O_4$ spinel in the oxide near the oxide/air interface. The formation of and the location of the spinel suggests diffusion of Mn through the Cr_2O_3 layer, corroborating Wild's observation based on Auger spectroscopic measurements that the Mn ion diffuses faster than the Cr ion in a Cr_2O_3 lattice. ²² Hua et al., reported the formation of an outer layer of Mn_2O_3 over a layer of a Mn-Cr spinel and this however was not observed in our study, due possibly to the lower Mn content. ¹⁶

The surface features of the three Y_2O_3 coated specimens oxidised for 200 h at 1000 °C were also identical and a micrograph of one of these specimens, shown in Fig.3a, reveals cuboid clusters of the Y_2O_3 coating. The EDS spectrum of this surface is shown in Fig.3b and it reveals the presence of O and Cr besides Y. A cross section of the same specimen is shown in Fig. 3c. This reveals a compact dark grey layer

Table 1: EDS analysis results of regions 1 and 2 in Fig.1c.

Region	Elements (wt%)			
	0	Cr	Mn	Fe
1	27.55	70.18	1.01	1.25
2	20.39	52.84	25.9	0.87



(C)

Figure 1: Scanning electron micrographs and the EDS spectrum of uncoated Fe-20Cr alloy specimen oxidized for 200h at 1000°C. (a) The surface; (b) the EDS spectrum of the surface; (c) cross section of the specimen.

close to the alloy and EDS analyses at regions 1 and 2 revealed the elements shown in the Table 2. Region 1 is rich in Cr suggesting Cr_2O_3 formation by reaction of Cr from the alloy with O in the Y_2O_3 layer. Region 2 revealed Cr and Mn, besides Y, also suggesting diffusion of Cr and Mn through the inner Cr_2O_3 layer.

The XRD spectrum of the oxidised Y_2O_3 coated specimen is shown in Fig. 4 and it reveals YCrO₃ in the scale, a perovskite oxide that formed from the reaction between Cr₂O₃ and Y₂O₃. No Mn containing phase was detected, due



Figure 2: XRD spectrum of uncoated Fe-20Cr alloy specimen oxidized for 200h at 1000 °C.

possibly to the Mn content in region 2 of Fig. 3c being very small. Considering the elements detected in regions 1 and 2 of Fig. 3c, it is evident that the YCrO₃ phase forms at regions between region 1 and region 2. That is, the Cr which diffused through the Cr_2O_3 layer towards the Y_2O_3 layer forms Cr_2O_3 within the Y_3O_3 layer and these in turn react to form YCrO₄.

Figure 5 shows the average mass gain after each oxidation cycle of the bare and Y_2O_3 coated specimens. Every point in this figure represents the average value of mass gained by three specimens at the end of a specific cycle of oxidation. The average mass gains of the bare and the coated specimens were very high during the first four cycles and thereafter remained almost unchanged. None of the specimens spalled, even after 15 cycles. The higher mass gains during the first four cycles of oxidation is indicative of a transient stage and formation of MnCr₂O₄, besides Cr₂O₃. Evidence of this is shown henceforth in the EDS and XRD analyses results of these specimens. After the 7th cycle the mass gains are almost constant indicating steady growth of the Cr₂O₃ layer.

Table 2: EDS analysis results of regions 1 and 2 in Fig.3c.

Region	Elements (wt%)				
	0	Cr	Mn	Fe	Y
1	32.4	66.6	-	1.0	-
2	23.6	8.3	0.5	-	67.6



Figure 3: Scanning electron micrographs and the EDS spectrum of Y_2O_3 coated Fe-20Cr alloy specimen oxidized for 200h at 1000°C. (a) The surface; (b) the EDS spectrum of the surface; (c) cross-section.

(c)

3 µm ⊢

The surface features of the three bare specimens after 15 cycles of oxidation were identical and Fig 6a shows the features of one of the specimens. Some thermal stress induced cracks can be seen but there is no evidence of spalling. The EDS spectrum of this surface is shown in Fig. 6b and it reveals that the oxide is rich in Cr and contains Mn, similar to that observed on specimens that were oxidised for 200 h at 1000 °C. Fig. 6c shows the cross section of this specimen and it reveals voids at the metal/oxide interface. The results of EDS analyses at regions 1 and 2 are shown in Table 3. Region 1 is a Cr rich oxide with traces of Fe and region 2, close to the oxide/gas interface reveals Mn, besides Fe and Cr. XRD analysis of the bare specimen surface after 15 cycles of oxidation revealed Cr₂O₃ and MnCr₂O₄, similar to that observed on the surface of the specimen that was oxidised for 200 h at 1000 °C.

The surface features of the three Y_2O_3 coated specimens after 15 cycles of oxidation were identical and as shown in Fig. 7a. No spalling was observed and the morphology of the surface oxide was similar to that of the specimen oxidised for 200 h at 1000 °C (Fig. 3a). The EDS spectrum of the surface in Fig 7a is shown in Fig. 7b. The elements Y and Cr were detected, indicating Cr diffusion to the outer layer. The cross section of the cyclically oxidised Y_2O_3 coated specimen is



Figure 4: XRD spectrum of Y_2O_3 coated Fe20Cr alloy specimen oxidized for 200 h at 1000 °C.



Figure 5: Mass gain per unit area after each cycle of 7 h at 1000 °C followed by cooling to room temperature of uncoated and Y_2O_3 coated Fe-20Cr specimens.

shown in Fig. 7c. The results of EDS analysis at regions 1, 2 and 3 are shown in Table 4. Region 1 revealed Cr with some Fe, while regions 2 and 3 in the Y_2O_3 coating revealed Y and Cr as foreseen. The Cr content in region 3 is less than that in region 2, indicating its diffusion from the substrate through the Y_2O_3 layer. The results of XRD analysis of the oxide scale on the Y_2O_3 coated specimen oxidised cyclically revealed the phases Cr_2O_3 , Y_2O_3 and $YCrO_3$, quite similar to that on the specimen that was oxidised for 200 h at 1000 °C.

Macroscopic observations of the Y_2O_3 coated specimens oxidised cyclically revealed that after each cycle, increasing areas of the coating became dark and eventually green. This colour change is due to chromium diffusion through the Y_2O_3 coating, evidence of which can be seen in Table 4.

GENERAL DISCUSSION

Oxidation of iron-chromium alloys at temperatures above 950 °C and under high oxygen pressures leads not only to formation of Cr₂O₃ on the surface of the alloy but reaction of the Cr_2O_3 with O_2 to form volatile CrO_3 . ²³ At 1000 °C and at constant oxygen pressure, mass loss caused by conversion of Cr₂O₂ to CrO₂ is constant with time. Hence the observed mass gains of the bare and the Y_2O_3 coated specimens oxidised for 200h at 1000 °C indicate that the rate of Cr₂O₃ formation is higher than the rate of conversion of Cr₂O₃ to CrO₃. The mass gain of the bare specimens oxidised at 1000 °C for 200 h was significantly higher than that of the Y₂O₃ coated specimens oxidised under the same conditions. This indicates that the higher mass gain of the bare specimen compared with the Y2O3 coated specimen is due to formation of significantly more Cr₂O₃ in the former and that the Y₂O₂ coating affects markedly the oxidation rate of the Fe-20Cr alloy. The micrographs, the EDS spectra and XRD data of the bare specimens oxidised for 200 h at 1000 °C reveal that the oxide formed was intact and its growth due to predominant alloy cation diffusion, demonstrating thus parabolic growth behaviour. The growth of oxide on the Y₂O₃ coated specimens oxidized for 200 h at 1000 °C was also parabolic, but due to predominant oxygen ion diffusion as shown below.

The structure and composition of the oxide formed on specimens oxidised for 200 h at 1000 °C and cyclically were identical. The scale on the bare and coated specimens

Table 3: EDS analysis results of regions 1 and 2 in Fig.6c.

Region	Elements (wt%)				
	0	Cr	Mn	Fe	
1	32.58	66.73	-	0.69	
2	14.89	54.31	29.59	1.21	

Table 4: EDS analysis results of regions 1, 2 and 3 in Fig.7c.

Region	Elements (wt%)			
	О	Cr	Fe	Y
1	29.92	66.58	3.5	-
2	23.88	25.14	-	50.98
3	24.00	13.59	-	62.41





Figure 6: Scanning electron micrographs and the EDS spectrum of Fe-20Cr alloy specimen after 15 cycles of oxidation for 7 h at 1000 °C. (a) The surface; (b) the EDS spectrum of the surface; (c) the cross-section.

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Figure 7: Scanning electron micrographs and the EDS spectrum of Y_2O_3 coated Fe-20Cr alloy specimen after 15 cycles of oxidation for 7 h at 1000 °C. (a) The surface; (b) the EDS spectrum of the surface; (c) the cross-section.

did not spall even after 15 cycles of oxidation. Oxidation of Y₂O₂ coated Fe-20Cr specimens can be considered to consist of an initial transient stage during which a thin layer of Cr₂O₃ forms on the alloy surface. Formation of this layer has been reported to be aided by Y₂O₂ acting as nucleation sites. ^{24,25} This stage is followed by oxidation at a much lower rate with Cr diffusing through the Cr₂O₂ layer to form Cr₂O₃ in the Y₂O₃ coating. These two oxides react to form perovskite-type oxide YCrO, which increases in quantity with time at temperature. In this study the scale on the Y_2O_2 coated Fe-20Cr specimen oxidised for 200 h at 1000 °C consisted of Cr₂O₂, Y₂O₂ and YCrO₂. Chevalier and Larpin also detected Y_2O_3 , Cr_2O_3 and $YCrO_3$ in the scale on Y_2O_3 coated Fe-30Cr alloy oxidised at 1000 °C for 100 h and also in an equimolar powder mixture of Y₂O₃ and Cr₂O₃ heated for 288 h at 1000 °C, the latter indicating that formation of YCrO₃ phase is a very slow process. ²⁶

The lower oxidation rate of the Y2O3 coated alloy compared to the bare alloy is due to formation of YCrO₃ which brings about an increase in oxygen ion vacancies in the chromia scale and consequent increase in diffusion of oxygen ions to the scale/alloy interface. 25,27 Other studies have also reported the reduced oxidation rates of Y containing or Y₂O₂ coated Fe-Cr alloys to formation of YCrO₃ and/or YCrO₄. ^{26, 28-34} It has also been reported that below 1100 °C oxidation is dominated by short circuit diffusion. ³⁵ That is, the oxide scale grain boundaries are fast diffusion paths for migrating ions. In Y containing or Y2O3 coated alloys, the Y ion in the oxide scale segregates to the grain boundaries and its radius (1.02 Å) being larger than the Cr and Fe ions (0.615 Å and 0.78 Å respectively) blocks the path of the diffusing alloy cations, decreasing thereby the oxidation rate of the alloy. 6,36 Hence, the influence of the Y₂O₃ coating in reducing the oxidation rate of chromia forming alloys is twofold. It increases oxygen ion vacancies and thereby oxygen ion diffusion in the YCrO, compound. It also decreases Cr ion diffusion rate along Cr₂O₂ grain boundaries due to the presence of the segregated Y ions. Indirect proof of segregation of Y ions to the Cr₂O₃ grain boundaries is evident from the marked

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reduction in the rate of Cr_2O_3 formation on the Y_2O_3 coated specimen compared to that on the bare specimen oxidised at 1000 °C. Increase in oxidation resistance of chromia forming alloys in the presence of Y has been also explained by the interface segregation mechanism. ³⁷ Seo et al., showed using depth profiling studies on Y containing Fe-22Cr-0.5Mn alloy specimens oxidised at 800 °C that Y segregated at the alloy/oxide interface and suppressed outward Cr ion diffusion.¹² This mechanism may be valid to a greater extent when the reactive element is added to the alloy.

CONCLUSIONS

- The oxide layer formed on Fe-20Cr alloy oxidised isothermally at 1000 °C for 200 h and cyclically between 1000 °C and room temperature consisted of mainly Cr₂O₃. This oxide layer contained MnCr₂O₄ near the oxide/air interface.
- The oxide layer formed onY₂O₃ coated Fe-20Cr alloy oxidised at 1000 °C for 200 h and cyclically contained YCrO₃.
- 3. Compared to the uncoated alloy a marked decrease in mass gain of the coated alloy was observed and attributed to formation of the YCrO₃ phase.
- 4. The oxide on the specimen surfaces did not spall during the cyclic oxidation tests.
- During the cyclic oxidation tests, the Y₂O₃ coating remained adhered to the substrate and its colour changed gradually from white to dark green with increase in duration of oxidation.
- The effect of Y₂O₃ coating in reducing the oxidation rate is twofold. It increases oxygen ion diffusion in the YCrO₃ compound and segregation of Y ion to scale grain boundaries with resultant reduction in diffusion rate of alloy cations.
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