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Original Article

Microstructural alteration and oxidation behavior of boronized stainless steel AISI 440C after heat treatments

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Abstract

The martensitic stainless steel AISI 440C was boronized at a temperature of 950 °C for about 4 hr. Heat treatments at a temperature of 850 °C for about 2-8 hr were performed to eliminate the FeB phase of the boride layer. The oxidation investigations were carried out at a given temperature of 900 °C on different surface conditions. It was found that the FeB phase was completely dissolved and transformed to the Fe_2B phase after a heat treatment at a temperature of 850 °C for about 8 hr. The oxidation behavior of the non-boronized, boronized and boronized with heat treatment of 8 hr conditions exhibit a parabolic manner, while the specimens boronized with heat treatments of 2 – 6 hr show a linear manner. The single-phase (Fe_2B) boride layer on the martensitic stainless steel AISI 440C possesses a lowest oxidation rate as compared to other investigated surface conditions.

Keywords: oxidation resistance, thermochemical surface treatment, heat treatment, martensitic stainless steel, boronizing

1. Introduction

Stainless steel is one of the most widely used engineering materials. It has excellent mechanical strength and good corrosion resistance especially martensitic stainless steels (Davis, 2000). In recent years, not only the complicated and high-loading applications but also high-temperature applications are much more required. Therefore, to enhance a service lifetime, various surface treatments are mentioned for improving wear resistance and oxidation resistance of stainless steels. A boronizing process is a thermochemical surface treatment which provides a boride layer with outstanding properties, i.e., wear, oxidation and corrosion resistances and can be applied on various metallic materials (Atık, Yunker, & Merıç, 2003; Davis, 2002; Lee, Kim, & Kim, 2004; Sen, Sen, & Bindal, 2006; Sinha, 1991). Actually, the main purpose of boronizing process is to improve the wear resistance of various metallic materials. However, other advantageous properties of the boride layers such as high-temperature oxidation resistance and high corrosion resistance were investigated and reported by several researchers (Davis, 2002; Jaques, & Butt, 2015; Kartal, Timur, Sista, Eryilmaz, & Erdemir, 2011; Li, Dai, Cheng, Wang, & Huang, 2012; Santhanakrishnan, Kong, & Kovacevic, 2011; Sinha, 1991). The boride layer formed on high alloying steels is usually a double-phase (FeB and Fe_2B) type. The boron-rich phase, FeB is more brittle than Fe_2B phase and shall be eliminated before use. The conventional heat treatment is a simple method to diminish FeB phase on the double-phase boride layer. The diffusion and phase transformation concepts were used to explain this phenomenon (Davis, 2002; Kartal, Timur, Sista, Eryilmaz, & Erdemir, 2011; Sinha, 1991). During heat treatment as well as phase transformation, the oxidation resistance of the double-phase boride layer shall be altered and is of a particular interest. Therefore, in this paper, the microstructural alteration and oxidation behavior of the boronized stainless steel AISI 440C after heat treatments were addressed and clarified. The microstructural alteration was characterized using an optical microscope and X-ray diffraction technique (XRD). The oxidation rate of the boronized with and without heat treatments were calculated and compared to the non-boronized martensitic stainless steel AISI 440C.

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2. Materials and Experimental Procedures

The investigated materials is a martensitic stainless steel AISI 440C. The chemical compositions are 1.04% C, 0.38% Mn, 0.41% Si, 0.021% P, 0.001% S, 16.59% Cr and 0.43% Mo (all values in wt.%). A cylindrical shape of samples was 13 mm in a diameter and 10 mm in a length. Before boronizing process, all samples were ground up to 600 grit SiC paper for a clean and good surface. The boronizing process was carried out in a solid medium consisting of Ekabor-I powders from BorTec GmbH, Germany. The samples were placed with a distance of 15 mm and packed as well as buried in Ekabor-I powders into a steel container with a lid and cement seal. The steel container was heated in electric furnace under argon atmosphere at a temperature of 950 °C for a boronizing time of 4 hr and then cooled in air to room temperature. After boronizing process, some boronized specimens were heated at a given temperature of 850 °C with soaking times of 2, 4, 6 and 8 hr under argon atmosphere and finally cooled in air to room temperature. Microstructures and kind of the formed, as well as altered layers, were characterized using an optical microscope and XRD technique with Cu Kα radiation source (λ=0.154 nm.), respectively. The thickness values of the formed layer were measured using an optical microscope with an image analyzer program. The hardness depth profile was measured using a Vicker microhardness tester with a load of 50 g. The oxidation tests were conducted in an electric furnace at a temperature of 900 °C with total time of 50 hr. The boronized specimens of both with and without heat treatment process were taken out of the furnace and cooled to room temperature at various intervals for mass measurement using an electronic analytical balance with a sensitivity of 0.01 mg. The total mass of a specimen together with the crucible was recorded.

3. Results

3.1 Microstructure and characterization

After the boronizing process at a temperature of 950 °C for about 4 hr, a double-phase boride layer with a thickness about 41 µm was detected. The cross-sectional microstructure of the boride layer is shown in Figure 1. The boride layer on the martensitic stainless steel AISI 440C has smooth and more compact morphology as compared to low or unalloyed steels (Atık, Yunker, & Merıç, 2003; Campos-Silva *et al.*, 2010; Campos, Palomar, Amador, Ganem, & Martinez, 2006; Lee, Kim, & Kim, 2004; Rie & Broszeit, 1995; Taktak, 2006). The outer dark phase of the boride layer in Figure 1 is the FeB phase with 16.23 wt.% B. The brighter phase is Fe_2B with 8.83 wt.% B (Davis, 2002; Sinha, 1991). The XRD spectrums were measured to verify the phases of the formed boride layer as illustrated in Figure 2. The XRD patterns can confirm that the phases of the formed boride layer are FeB and Fe_2B which are related to the microstructure in Figure 1. Additionally, the near surface hardness of the boride layer could be reached about 2000 HV, whereas the hardness of the substrate of approximately 600 HV was measured. The hardness depth profile of the martensitic stainless steel AISI 440C boronized at 950 °C for about 4 hr was measured and shown in Figure 3.

After heat treatment process at a temperature of 850 °C for 2, 4, 6 and 8 hr, the cross-sectional microstructure of boronized martensitic stainless steels are shown in Figure 4a-d.

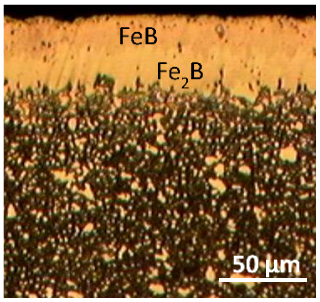

Figure 1. Cross-sectional microstructure of the boronized martensitic stainless steel AISI 440C.

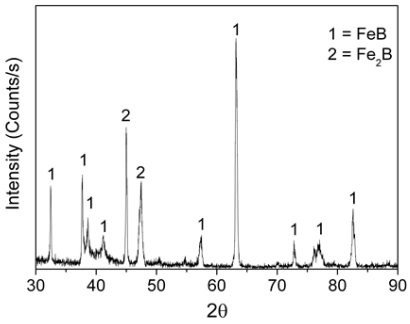

Figure 2. XRD spectrums of the boronized martensitic stainless steel AISI 440C.

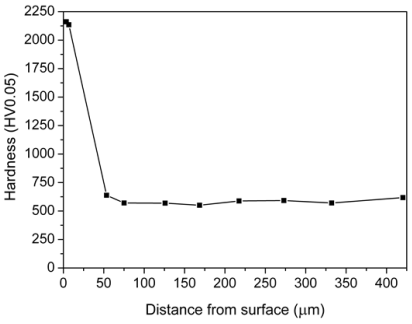

Figure 3. Hardness depth profile of the boronized martensitic stainless steel AISI 440C.

The double-phase boride layer is still observed after heat treatment for about 2 – 6 hr. However, the FeB phase thickness decreases with the soaking time increases till 6 hr as illustrated in Figure 4a-c. Finally, only single-phase Fe_2B boride layer of about 53 µm is detected after heat treatment for about 8 hr (Figure 4d). The XRD patterns in Figure 5 are used to confirm above mention. The altered thickness values of the FeB and Fe_2B with different heat treatment conditions are given in Figure 6.

3.2 High-temperature oxidation behavior

After the boronizing, the high-temperature oxidation tests were performed at a given temperature of 900 °C for a total time of 50 hr. Two characteristics of the oxidation behavior are detected. First, the parabolic manner was observed for three conditions, e.g., non-boronized, boronized and boronized with heat treatment for about 8 hr as shown in Figure 7a. For the

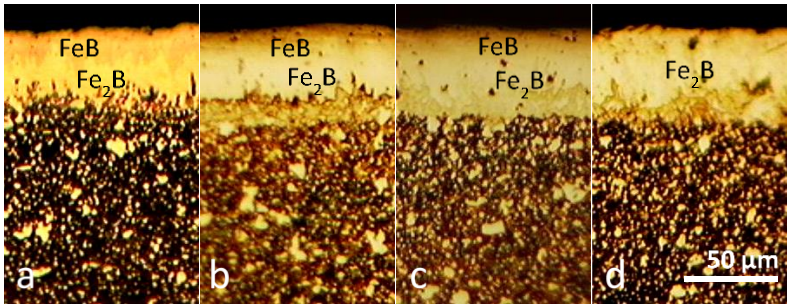

Figure 4. Cross-sectional microstructure of the boronized martensitic stainless steel AISI 440C after heat treatment at a temperatures of 850 °C for (a) 2 hr, (b) 4 hr, (c) 6 hr and (d) 8 hr.

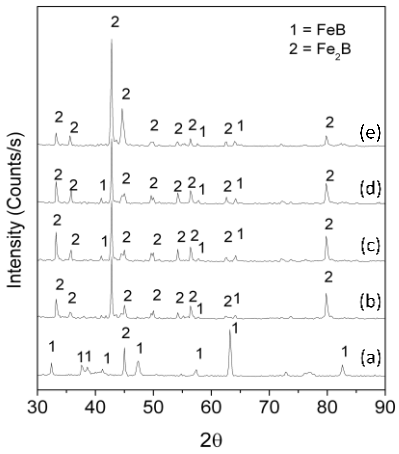

Figure 5. XRD patterns of various surface of the boronized martensitic stainless steel AISI 440C; (a) without heat treatment, with heat treatment at a temperature of 850 °C for (b) 2 hr, (c) 4 hr, (d) 6 hr and (e) 8 hr.

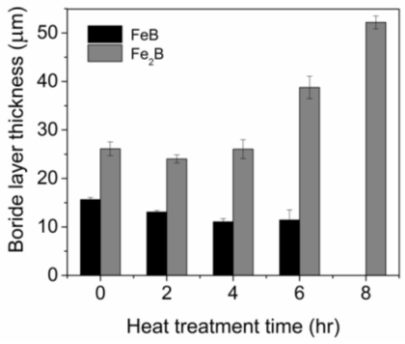

Figure 6. Alteration of the FeB and Fe2B thickness during heat treatments at a temperature of 850 °C.

parabolic manner, the squared weight grain of the oxidized sample as a function of time can be described in Equation (1) as follows (Fontana, 1986):

$$m^2 = k_p t + c \quad (1)$$

where m is the weight gain per unit area (mg/cm^2), t is the oxidation time (hr), c is a constant, k_p is the oxidation rate at a constant oxidation temperature. Second, the linear manner was seen for other conditions, e.g., boronized with heat treatment for about 2, 4 and 6 hr as illustrated in Figure 7b. For linear manner, the weight gain of the oxidized sample as a function of time can be described in Equation (2) as follows (Fontana, 1986):

$$m = k_l t \quad (2)$$

where m is the weight gain per unit area (mg/cm^2), t is the oxidation time (hr), k_l is the oxidation rate at a constant oxidation temperature. After oxidation test at a temperature of 900 °C for about 50 hr, XRD was performed to verify the oxidized surfaces as shown in Figure 8. The iron oxide, Fe_2O_3 pattern is observed in all conditions after the high-temperature oxidation tests.

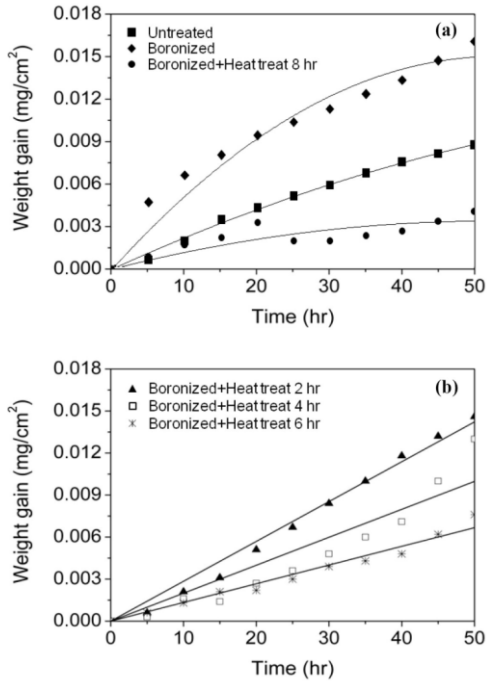

Figure 7. Evolution of the weight gains over soaking time during the oxidation tests of the various surface conditions; (a) non-boronized, boronized, boronized with heat treatment of 8 hr, (b) boronized with heat treatment of 2, 4 and 6 hr.

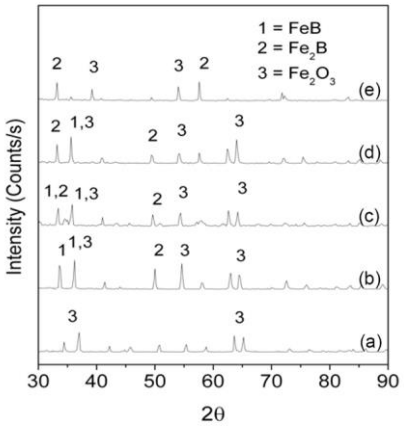

Figure 8. XRD patterns of various surfaces: (a) non-boronized, boronized with heat treatment at a temperature of 850 °C for (b) 2 hr, (c) 4 hr, (d) 6 hr and (e) 8 hr oxidized at a temperature of 900 °C for about 50 hr.

4. Discussion

After boronizing process at a temperature of 950 °C for 4 hr, the formed boride layers on the martensitic stainless steel AISI 440C have smooth and more compact morphology due to high alloying elements. The development of a jagged boride/substrate interface is suppressed in consequence of an increase of alloying elements and carbon content of the substrate (Davis, 2002; Sinha, 1991). Alloying elements in the martensitic stainless steel AISI 440C obstructed the diffusion of boron atoms into the steel, as a consequence of a smooth boride/substrate interface and double-phase boride layer (Figure 1). The double-phase boride layer is affected by applied heat treatment at a temperature of 850 °C. The phase transformation was observed due to the chemical concentration gradient between the FeB (16.23 wt.% B) and Fe_2B (8.83 wt.% B) phases, and then a diffusion process occurred. The boron atoms can diffuse from high chemical potential side to low chemical potential side at the interface in the boride layer and steel substrate during the heat treatment process at a high temperature (Davis, 2002; Kartal, Timur, Sista, Eryilmaz, & Erdemir, 2011; Naemchanthara & Juijerm, submitted; Sinha, 1991). Thus, the FeB phase was slowly dissolved at the FeB and Fe_2B interface during the heat treatment in argon atmosphere determined by Equation (3). Afterwards, the Fe_2B phase increased at the interface of the Fe_2B and Fe (substrate) determined by Equation (4) (Figure 4 and 6) (Naemchanthara & Juijerm, submitted). Finally, the double-phase boride layer was changed entirely to the single-phase boride layer by heat treatment at a temperature of 850 °C for 8 hr (Figure 4 and 5) (Davis, 2002; Kartal, Timur, Sista, Eryilmaz, & Erdemir, 2011; Sinha, 1991).

$$2FeB \rightarrow Fe_2B + B \quad (3)$$

$$2Fe + B \rightarrow Fe_2B \quad (4)$$

The oxidation behavior of the investigated martensitic stainless steel AISI 440C at a temperature of 900 °C exhibits a parabolic manner (Figure 7a). At a given temperature of 900 °C, the passive film of the chromium oxide on the surface of the martensitic stainless steel AISI 440C should be unstable (Fontana, 1986) and then oxygen atoms could react with iron atoms to produce iron oxide on the surface as confirmed by the XRD result in Figure 8e. In the early stage of the oxidation process, oxygen atoms directly contacted to the surface. The oxidation rate was controlled by chemical reaction, as a consequence of a high oxidation rate. Afterwards, the oxidation rate seems to be decreased due to a formation of an oxide film at the surface. Therefore, the oxidation rate was controlled by the solid-state diffusion of alloying elements through the oxide film (Cairo, Florian, Graça, & Bressiani, 2003; Campos, Palomar, Amador, Ganem, & Martinez, 2006; Sumathi, Sundaram, & Chandramohan, 2011; Sundararajan, Kuroda, Kawakita, & Seal, 2006). The parabolic manner was also detected for the boronized and boronized with heat treatment for about 8 hr (Figure 7a) which the phases on the surface are FeB and Fe_2B, respectively. For the FeB phase at the surface, the boron atoms constituting the FeB phase were preferentially oxidized on the surface, and then Fe_2O_3 and B_2O_3 layer were formed according to the reaction in Equation (5) (Carbucichio, Reverberi, Palobarini, & Sambogna, 1989). The oxidation reaction of the Fe_2B phase was described by Equation (6). The oxidation products of the FeB and Fe_2B phases are similar. Moreover, the boron oxide, B_2O_3 was thermodynamically stable as a liquid phase at the oxidation temperature of 900 °C and became to a non-crystalline structure at room temperature, which could not be detected by XRD (Parthasarathy, Rapp, Opeka, & Kerans, 2007; Rizzo, 1960).

$$2FeB + 3O_2 \rightarrow Fe_2O_3 + B_2O_3 \quad (5)$$

$$4Fe_2B + 9O_2 \rightarrow 4Fe_2O_3 + 2B_2O_3 \quad (6)$$

The oxidation rate, k_p in Equation (1) can be determined using a slope of a straight line in linear plots of the squared weight gain versus oxidation time in Figure 9. By fitting the measured data of the three conditions, e.g., untreated (non-boronized), boronized and boronized with the heat treatment of 8 hr, oxidation rates of 0.0138×10^{-4}, 0.0466×10^{-4} and 0.0025×10^{-4} $mg^2\cdot cm^{-4}\cdot hr^{-1}$ were determined, respectively. The oxidation rate of the boronized condition (FeB phase at the surface) is higher than that of the non-boronized and boronized with heat treatment of 8 hr conditions (Fe_2B phase at the surface) due to the more thermodynamic stability of the Fe_2B phase at a high temperature as compared to the FeB phase (Jiang, Wang, Zhong, Zhou, & Zhang, 2011; Liu *et al.*, 2010). Therefore, free boron atoms of the FeB phase more easily released at a high temperature as compared to the Fe_2B phase. New free iron atoms were generated directly if the boron atoms released from FeB because of a one-by-one bonding. The generated iron atoms were reacted by oxygen atoms to form an iron oxide, while the released boron atoms were also reacted by oxygen atoms to form a boron oxide at the surface. Accordingly, the boronized one which has the Fe_2B at the surface as well as single-phase boride layer exhibits the lowest oxidation rate as compared to the other ones.

During the phase transformation as well as heat treatment for about 2, 4 and 6 hr, the oxidation behavior of the boronized martensitic stainless steel AISI 440C shows a linear function (Figure 7b). That means that the oxidation rate remains constant with the oxidation time followed by Equation (2). It is possibly due to the microcracks on the double-phase boride

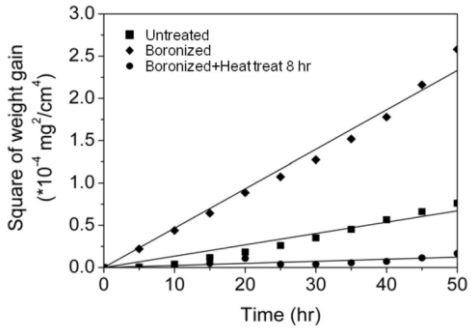

Figure 9. Evolution of the square weight gain over time during the oxidation tests of the various surface conditions; non-boronized, boronized and boronized with heat treatment of 8 hr.

layer formed during the heat treatment process. At the microcracks, there are a preferred diffusion path for oxygen atoms from environment into the boride layer. The oxidation rate could be probably enhanced by local stresses at the microcracks. Consequently, a linear characteristic was observed for the oxidation rates (Fontana, 1986; Trethewey, & Chamberlain, 1995). By fitting the measured data, the oxidation rates of the boronized specimens with heat treatment of 2, 4 and 6 hr, the oxidation rates of 0.00028, 0.00020 and 0.00013 $mg{\cdot}cm^{-2}{\cdot}hr^{-1}$ were determined, respectively. The oxidation test of the boronized condition illustrates that the FeB phase exhibits a high and parabolic oxidation rate (Figure 7a). If the FeB phase decreases with existed microcracks after heat treatment process, a lower oxidation rate with linear characteristic shall be expected. Therefore, the lower oxidation rates with increasing soaking time of the heat treatment process were detected (Figure 7b). The oxidation rates of all investigations are summarized in Table 1.

Table 1. Summary of the oxidation rates of all investigations.

Surface conditions	Oxidation rate	
	Parabolic ($x\ 10^{-4}\ mg^2{\cdot}cm^{-4}{\cdot}hr^{-1}$)	Linear ($mg{\cdot}cm^{-2}{\cdot}hr^{-1}$)
Non-boronized	0.0138	–
Boronized	0.0466	–
Boronized + Heat treatment 2 hr	–	0.00028
Boronized + Heat treatment 4 hr	–	0.00020
Boronized + Heat treatment 6 hr	–	0.00013
Boronized + Heat treatment 8 hr	0.0025	–

5. Conclusions

In this research, the microstructural alteration and oxidation behavior of boronized stainless steel AISI 440C after heat treatments were addressed and investigated. The main obtained results are summarized as follows:

(1) The double-phase (FeB and Fe_2B) boride layer formed on the martensitic stainless steel AISI 440C has a smooth and compact morphology. The hardness values of the boride layer can be reached approximately 2000 HV.

(2) The phase transformation occurred during the heat treatment process at a temperature of 850 °C. The FeB phase decreases with increasing soaking time. For about 8 hr at a temperature of 850 °C, the FeB phase was completely dissolved and transformed to the Fe_2B phase (single-phase boride layer) on the martensitic stainless steel AISI 440C.

(3) The oxidation behavior of the non-boronized, boronized (FeB) and boronized with heat treatment of 8 hr (Fe_2B) conditions exhibit a parabolic manner, while the specimens boronized with heat treatment of 2 – 6 hr show a linear manner.

(4) At a temperature of 900 °C, the single-phase (Fe_2B) boride layer on the martensitic stainless steel AISI 440C possesses a lowest oxidation rate as compared to the double-phase boride layer or non-boronized surface.

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