Research Article

Effect of the Boron Powder on Surface AISI W2 Steel: Experiments and Modelling

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1.Introduction

The AISI W2 type tool grade steels are widely used in cutting tools, punches for cold forming, cutting and punching. The boriding thermochemical treatment can improve the mechanical properties and extend the lifetime of the workpieces made of AISI W2 steel. The boriding process is the saturation of boron surface of ferrous and nonferrous alloy in order to increase the hardness and wear resistance in engineering components where industrial applications require properties [1].

In the boronizing process, boron atoms are introduced into the metal lattice at the surface of the workpiece through thermal energy to form borides with the atoms of the substrate. The boriding process applies in the temperature range 1073–1323 K between 0.5 and 10 h and it can be carried out in solid, liquid, or gaseous media and plasma [1, 2]. According to the iron-boron phase diagram [3], two iron borides can be formed, Fe2B and FeB; the FeB (orthorhombic) phase is more brittle than the Fe2B (tetragonal) phase.

Powder pack boriding is the most widely favoured boriding technique, due to having relative safety and simplicity. This process can be carried out by three boronizing potential of B4C powder: low (10% B4C-90% SiC), intermediate (100% B4C), and high (90% B4C-10% KBF4) [4]. In addition, the low and intermediate boronizing potential can form single Fe2B, and the high boronizing potential can form double Fe2B + FeB phases. The formation of single or double phases depends on the boriding process temperature, the boriding time, the chemical composition of the substrate material, and the boron potential of the boriding medium [5].
The single phase structure (Fe₂B) is generally more desirable for the intended application in the industry than the bilayer structure consisting of FeB + Fe₂B because FeB (2.9 × 10⁻⁸ K⁻¹) and Fe₂B (8.7 × 10⁻⁸ K⁻¹) phases have greatly different coefficients of thermal expansion. Therefore, crack formation is in the region of the FeB/Fe₂B interface of the double layers. The presence of the alloying elements of the substrate influences the morphology of the boride layers, so the smooth shape is obtained in high-alloy steels, whereas in the low alloy steels saw-tooth shape layer is obtained.

The mathematical modeling of the kinetic boriding has gained much attention to simulate the boriding kinetics during these last decades. M. Keddam et al. [6] reported that the modeling of the boriding kinetics is considered as a suitable tool to select the optimum process parameters for obtaining adequate boride layers thicknesses in relation to their practical applications. So, in the literature, there are diffusion models for single phase (Fe₂B) [7–10], for two phases (FeB + Fe₂B) [11, 12], and for the total layer [13, 14] on different substrates. Some parameters to consider are with or without incubation time, linear or nonlinear profile of boron concentration and boriding process. At the moment, there are no studies on the growth kinetics of Fe₂B layer on the surface of AISI W2 steel.

The purpose of this work was forming boride layers with boron powder mixture with low boronizing potential. A mathematical model of growth kinetic is proposed to estimate the boron diffusion coefficient in Fe₂B layer grown on AISI W2 steel considering the boride time incubation; based on the mass balance equation at the (Fe₂B/Fe) interface, the model was used to estimate the boron diffusion coefficient in the temperature range of 1173–1273 K. The estimated boron activation energy was compared with the data available in the literature. The present model was validated by comparing the experimental value of boride layer thickness. Finally, a contour diagram relating the Fe₂B layer thickness with the boriding parameters (temperature and time) was proposed for practical use of this kind of borided steel.

2. Mathematical Model of the Growth Kinetics of the Fe₂B Layer

The model considers a diffusion of boron atoms into a saturated iron substrate by forming the Fe₂B layer. The boron concentration profile through the Fe₂B is displayed in Figure 1. \(c_{\text{up}} = 59.8 \text{ mol m}^{-3}\) and \(C_{\text{low}} = 59.2 \text{ mol m}^{-3}\) are the same limits in the Fe₂B layer, these values are accepted based on the literature data [9, 15, 16]. In this context, the author Massalski [17] reported that Fe₂B iron borides have a narrow range composition of about 1%.

The Fe phase is present before boriding, and the initial condition of the diffusion problem is given by the following equation [18]:

\[ C_{\text{Fe}_2\text{B}}[t > 0] = 0. \tag{1} \]

Also, the boundary conditions (see Figure 1) of the diffusion problem are given by the following equations:

\[
C_{\text{Fe}_2\text{B}}[x = t = t_{0\text{PM}}(T)] = m_{0\text{up}}, \quad \text{for } C_{\text{ads}} < 6 \times 10^3 \text{ mol m}^{-3},
\]

\[
C_{\text{Fe}_2\text{B}}[x = t = t_{0\text{PM}}] = m_{0\text{up}}, \quad \text{for } C_{\text{ads}} > 6 \times 10^3 \text{ mol m}^{-3},
\]

\[
C_{\text{Fe}_2\text{B}}[x = t = t_m] = m_{\text{up}},
\]

\[
C_{\text{Fe}_2\text{B}}[x = t = t_m] = m. \tag{2}
\]

where \(C_{\text{ads}}\) is the effective adsorbed boron concentration, \(m\) is the average thickness of the Fe₂B layer, \(t_m\) corresponds to treatment time for the formation of the boride layer with a thickness of \(m\), \(t\) corresponds to treatment time, \(t_{0\text{PM}}(T)\) is the boride incubation time, and \(m_{0\text{up}}\) is the thickness of the layer after the nucleation stage [19]. Thus, \(m_{0\text{up}} \approx 0\) when compared to the thickness \(m\) of the Fe₂B after the boriding process.

With regard to this model, the following assumptions are put forward [10, 16]:

1. The growth kinetics are controlled by the boron diffusion in the Fe₂B layer
2. The growth of the boride layer occurs as a consequence of the boron diffusion perpendicular to the specimen surface
3. Differences in specific volume per solvent atom for Fe₂B phases are accommodated fully in the diffusion direction
4. The iron boride nucleates after a specific incubation time \(t_{0\text{PM}}(T)\)
5. Planar morphology is assumed for the interface Fe₂B/substrate
6. The effect of alloying elements present on the boron diffusion is neglected

The mass balance equation at the interface Fe₂B/Fe [20–22] is described by (3) and shown in Figure 1(a):

\[
a_1(A \cdot \text{dm}) + \frac{1}{2} a_3(A \cdot \text{dm}) = J_{\text{Fe}_2\text{B}}(x = m)(A \cdot \text{dr}_m) - J_{\text{Fe}}(x = m + \text{dm})(A \cdot \text{dr}_m), \tag{3}
\]

where \(A (= 1 \times 1)\) is defined as the unit area, where, from Figure 1(b), \(a_1 = C_{\text{Fe}_2\text{B}} - C_{\text{Fe}_2\text{B}}\), \(a_2 = C_{\text{Fe}_2\text{B}} - C_0\) defines the homogeneity range of the phase designed Fe₂B, \(a_3 = C_{\text{Fe}_2\text{B}} - C_0\) is the miscibility gap [15], and \(C_0\) is the terminal solubility of the interstitial solute. The concentrations \(C_{\text{Fe}_2\text{B}}, C_{\text{Fe}_2\text{B}}\), and \(C_0\) are expressed in mol m⁻³, but \(C_0 = 0\) mol m⁻³ due to the lower solubility of boron [9, 16].

A linear distribution of the boron concentration with the thickness of the Fe₂B layer is described as follows [11, 23, 24]:

\[
C_{\text{Fe}_2\text{B}}[x(t)] = C_{\text{Fe}_2\text{B}} + \frac{C_{\text{Fe}_2\text{B}} - C_{\text{Fe}_2\text{B}}}{m} x(t). \tag{4}
\]

The fluxes \(J_{\text{Fe}_2\text{B}}\) and \(J_{\text{Fe}}\) are then given by Fick’s first law \(J = -D(dC[x(t)]/dx(t))\) as follows:
3. Experimental

3.1. The Powder Pack Boriding. AISI W2 steels with a nominal composition in weight: % C 0.85–1.50, Mn 0.10–0.40, Si 0.10–0.40, Cr 0.15, Ni 0.20, Mo 0.10, W 0.15, and V 0.15–0.35. The samples were sectioned in cubic shape of 500 mm$^3$. Before the boriding, the samples were sanded with silicon carbide abrasive paper up to 600 grit and cleaned with alcohol. The samples were embedded with cylindrical container (AISI 316) having Durborid fresh boron powder mixture inside with an average particle size of 50 µm as shown in Figure 2. Boron powder mixture contains of B$_4$C (active source of boron), Na$_3$AlF$_6$ (activator), SiC (inert filler), and SiC$_{6}H_{12}O_{4}$, which is used to protect surfaces; the surface of the sample is covered with a layer of approximately 10 mm thick boron powder mixture with a lid that does not act as a closing device. Boriding was accomplished by placing the container in an electrical resistance furnace model Millennium 2500 from Mexico without the use of inert gases at temperatures of 1173, 1223, and 1273 K with four exposure times of 2, 4, 6, and 8 h for each temperature. After the boriding treatment, the container was removed from the furnace and slowly cooled to room temperature.

3.2. Experimental Techniques. The samples were sectioned by diamond saw and resin embedded, polished using standard metallographic techniques, and then etched with Vilella’s reagent to reveal their cross-sectional microstructure. The microstructure, morphology, and thickness of boride layers were observed in clear-field through optical microscopy with the aid of a CARL ZEISS AXI0 equipment.
4. Results and Discussion

4.1. Boron Powder Mixture (Boriding Process). The microstructures of the cross-sectional of AISI W2 borided steel with the condition at 1173 K for 2, 4, 6, and 8 h are shown in Figure 4. According to the chemical etching in the cross section of the specimen, clear zones are observed in the image obtained by optical microscopy (OM). These zones have boride layer with Fe₂B and substrate with carbides. In Figure 4, single Fe₂B layer with smooth and compact morphology was observed in all samples. The comparison of Figures 4(a)–4(d) shows that the thickness of Fe₂B layer increases with the exposure time boriding.

The carbides present in AISI W2 steel reduce the boron flux into the substrate, and the interface between boride layer and substrate takes smooth morphology; this morphology is a characteristic feature for boride layers in high-alloyed steel [7, 10, 14]. Therefore, the borided layers depend on the concentration of alloying elements of the substrate, the temperature, and time exposure.

The average thickness values of Fe₂B layers were calculated using the equation as set in Figure 3. In this way, Figure 5 shows the influence of the boriding time and temperature of the depth of Fe₂B layers and represents a parabolic relation between the thickness of the Fe₂B layer and boriding time. The average value of the largest Fe₂B layer formed is about 45.86 ± 4.13 μm at condition at 1273 K for 8 h and the average value of the smallest Fe₂B layer formed is about 9.96 ± 2.61 μm at condition at 1123 K for 2 h. The Fe₂B layer thickness increased with treatment time and temperature, so the growth of continuous layers of iron borides is generally recognized as a diffusion-controlled process [25].

If the adsorbed boron concentration is on the value of upper limit 59.8 mol·m⁻³ and lower limit 60 × 10² mol·m⁻³ of boron content, the iron atoms react with active boron atoms to form the Fe₂B phase according to the following reaction [26]:

\[
B(\text{ads}) + 2\text{Fe} \rightarrow \text{Fe}_2\text{B}
\]  (10)

The mechanism of nucleation and growth during the boriding treatment can be described according to reports by Espinoza et al. [7] and Keddam et al. [10] during the powder pack boriding. At the beginning, there is a saturation of matrix by boron element for a time duration corresponding to \( t_0^{\text{Fe}_2\text{B}} \) (time incubation), after exceeding this period of incubation, the Fe₂B crystal starts nucleation on the sites energetically favorable. In addition, the growth mechanism involves three subsequence stages described by Martini et al. [4]: (i) acircular crystals of Fe₂B preferentially growing on the substrate surface with their [001] axis mainly oriented parallel to the surface, (ii) several Fe₂B crystals growing inside along different direction, and (iii) Fe₂B crystals cause the formation of orientation [002] and growth along the direction of minimum resistance.

The EDS analysis was used to examine the element distribution of the boride layer. Figure 6 shows the SEM image and EDS line scan analysis on the cross section of the AISI W2 steels boride at 1273 K for 8 h. The EDS analysis was performed in two areas: boride layers and substrate. Figure 6(a) shows that the area of boride layer is rich in iron and boron; due to the atomic radius of B (=0.09 nm), the boron atoms can diffuse easily into ferrous alloys (atomic radius of 0.124 nm). The result is the formation of a single layer of Fe₂B, and other elements such as vanadium, chromium, and molybdenum are present. The results also show the possible dissolution of chromium in the Fe sublattice of Fe₂B. The elements carbon and silicon are not dissolved in Fe₂B; these elements tend to displace towards...
the substrate [14, 27]. Figure 6(b) shows the area of the substrate (AISI W2) indicating the presence of the following elements: carbon, vanadium, chromium, silicon, molybdenum, and iron. In the case of vanadium, chromium, and molybdenum, spheroidal-shaped carbides are present in the AISI W2 steel matrix, and the boron is not present in the substrate.

Figure 7 shows the XRD patterns of the surface AISI W2 boride steels at a temperature of 1223 K for 8 h. The predominant phases formed in boride layers were Fe$_2$B, and the interstitial compounds such as CrB and Cr$_2$B phases were identified. The element Cr tends to dissolve in the Fe$_2$B phases than molybdenum. In contrast, the XRD results reported by Genel et al. [14] and Ozbek et al. [27] indicated the existence of iron boride (FeB + Fe$_2$B) for the pack boride AISI W1 and AISI W4, respectively. The formation of a single phase (Fe$_2$B) and bilayers (FeB + Fe$_2$B) in the steel substrate depends on the boron potential, and in the case of Fe$_2$B phase formation a low boron potential is required according to reported work [4, 28].

4.2. Growth Kinetics of Fe$_2$B Layers. As mentioned before, the boriding time evolution of the Fe$_2$B thickness is shown in Figure 5. The growth rate constants $k^2$ are reflected by the slope of the straight lines in Figure 5. The intersection in the abscissa is considered as the incubation time $t_{0,Fe_2B}$. The experimental values of $k^2$ and $t_{0,Fe_2B}$ are shown in Table 1. By
combining equation (8) with the experimental results demonstrated in Figure 5, the boron diffusion coefficient of boron in the Fe2B layers is estimated for each treatment time at a constant treatment temperature.

The Arrhenius equation relating the boron diffusion coefficient to the boriding temperature, with boron activation energy $Q_{Fe2B}$ and preexponential $D_0$ factor, can be calculated from the slope and intercepts of the strapline on $Q_{Fe2B}$ of $\ln D_{Fe2B}$ versus $1/T$, as shown in Figure 8.

The boron diffusion coefficients in the Fe2B ($D_{Fe2B}$) layer was expressed as a function of boriding temperature (1173K to 1273K) as follows:

$$D_{Fe2B} = 1.07 \times 10^{-5} \exp \left(-\frac{187.696 \text{kJ mol}^{-1}}{RT}\right),$$

where $R = 8.314462$ (J/mol) is the ideal gas constant and $T$ absolute temperature (K). In this study, the calculated activation energy for the formation of Fe2B, CrB, and Cr2B phases in the boride coatings; the found values of boron activation energy for AISI W2 (187.696 kJ mol$^{-1}$) are different in comparison with borided tool steels which are attributed to the formation of phases type (Fe)$_x$B, (Cr)$_x$B, and (Mo)$_x$B. Therefore, the activation energy varies of the chemical composition of the substrate material, the boriding process, and the boron potential of the medium. In the case of boron potential, using Durborid powder mixture allows us to form a Fe2B layer on the surface; this is obtained in works of AISI O1 [7], AISI D2 [8], AISI S1 [29], and AISI W2 steels boriding by powder pack, due to lowering the boronizing potential (B$_4$C-base powder) [4].

In addition, the formation kinetics of Fe2B boride described by the diffusion model is verified by estimation of the Fe2B boride thickness as a function of the temperature and exposure time. Figure 9 shows the parameter $\beta(T)$ depends only on the temperature; this parameter has been used by several studies to estimate the thickness of the boride layers [32] and can be approximated by equation (11) using a linear fit:

$$
\begin{array}{c|c|c}
\text{Temperature (K)} & \text{Experimental parabolic growth constant } k^2 (\mu m^2/s) & \text{Time incubation } t_{Fe2B} (s) \\
\hline
1123 & 0.0749 & 4735 \\
1223 & 0.0534 & 3205 \\
1273 & 0.0352 & 1996 \\
\end{array}
$$

Table 1: Experimental values of parabolic growth rate constants with the corresponding boride incubation time on AISI W2 by power pack boriding.
Equation (7) can be modified as follows:

$$m = 2\beta(T) \left( \frac{c_{Fe_2B}^{up} - c_{Fe_2B}^{low}}{2c_{Fe_2B}^{up}} \right)^{1/2} \left( \frac{c_{Fe_2B}^{low} D_{Fe_2B}^{B} t}{c_{Fe_2B}^{up}} \right) (m).$$

Results obtained in equation (12) are compared with experimental values. Figure 10 shows the optical images of the boride layer formed at 1259 K for 3 h and 1148 K for 1 h, and a good concordance was obtained between the experimental values of the Fe$_2$B layer thickness and the predicted results by equation (12) as shown in Table 3. Furthermore, the thickness of Fe$_2$B is presented in contour plots as a function of the
exposure time and temperature for powder pack boride on AISI W2. The contour diagram presented in Figure 11 can be used as a tool to select the optimal value of Fe₂B layer thickness in relation to the industrial application on W2 steel. In this way, the optimal boride layer thickness on the surface of high-alloy steels ranges from 15 to 20 μm, and the Fe₂B layer can be applied on tool steels for chipless forming of metals [33].

5. Conclusions

The Fe₂B layers formed on the surface of AISI W2 steels with boron powder mixture and the growth kinetic of the boride layer were analyzed. The morphology in all the conditions was smooth and the thickness of the Fe₂B layers ranged from 9.96 to 45.86 μm. The activation energy is 187.69 kJ·mol⁻¹, and this result was compared with the literature data on tool steel borided. In addition, a mathematical model is established to estimate the growth kinetics of Fe₂B layers. This model takes the test parameters (temperature, treatment time, and incubation time) of the boriding process and the boron concentration profile in the Fe₂B/substrate. The effectiveness of the mathematical model is verified with both experimental measurement Fe₂B layer thicknesses and mathematical model and shows a strong agreement at 1259 K for 3 h and 1148 K for 1 h conditions. Finally, contour plots can be used to select the optimum boride layer thickness for industrial applications on AISI W2 tool steels.

Table 3: Prediction and estimate values of the Fe₂B layer thickness.

| Temperature (K) | Time (h) | Boride layer thickness (μm) estimated by equation (10) | Experimental boride layer thickness (μm) |
|----------------|----------|------------------------------------------------------|----------------------------------------|
| 1259           | 3        | 26.20                                                | 25.99 ± 2.61                           |
| 1148           | 1        | 9.25                                                 | 9.41 ± 1.14                            |

Figure 10: Cross-sectional views of AISI W2 steel borided by optical micrograph: (a) 1259 for 3 h and (b) 1148 K for 1 h.

Figure 11: Contour diagram of boride layer thickness Fe₂B phase of AISI W2 borided steel, as a function of temperature and time.

Data Availability

The paper includes experimental and numerical data on AISI W2 boriding steel used to evidence the conclusions and results of this study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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