

## Boriding Kinetics of FeB and Fe<sub>2</sub>B Layers on AISI M2 Steel by the Integral Diffusion Model

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### **ABSTRACT**

The purpose of this work is to investigate the boronizing kinetics of AISI M2 steel by using the integral diffusion model with consideration of boride incubation periods. This simulation model was established by solving the differential algebraic equations (DAE) resulting from the integral method in the temperature range of 1173 to 1323 K. By using a particular solution of the obtained DAE system, the values of boron diffusivities in the FeB and Fe<sub>2</sub>B layers were estimated. The estimated values of activation energies for boron diffusion in AISI M2 steel were respectively 228.06 kJ mol<sup>-1</sup> and 212.10 for FeB and Fe<sub>2</sub>B. Finally, a comparison was made between the simulated thicknesses of FeB and Fe<sub>2</sub>B layers and the experimental values obtained at 1173, 1223, 1273 and 1323 K for 10 h. The findings of this research work may serve as a tool to simulate the boronizing kinetics of any steel with a microstructure consisting of FeB and Fe<sub>2</sub>B layers versus the boriding parameters (the time and the temperature).

## 1. INTRODUCTION

The boriding treatment is used to harden the surfaces of workpieces by forming hard borides that result in an extremely high surface hardness, anti-wear and anti-corrosion resistance [1]. It is a thermochemical process involving the diffusion of boron atoms into the workpiece surface in the temperature range of 800 to 1050 °C for a time duration between 0.5 and 10 h. For ferrous alloys (steels and cast irons), two kinds of iron borides besides the metallic borides could be formed at the surface of treated parts. The simulation of boriding kinetics constitutes a tool for the optimization of the borided layer thickness in relation with the industrial applications of the treated steels. For this reason, the modeling of boriding kinetics using different approaches [2-12] received much interest from several researchers to predict the borided layer thickness at a given process temperature.

Among these diffusion models applied to borided steels, the diffusion model proposed by Campos-Silva et al. [12] can be evoked. This model was capable of analyzing the growth kinetics of FeB and Fe<sub>2</sub>B layers besides the diffusion zone on AISI 316 steel by incorporating the influence of boride incubation periods. Another diffusion model by Nait Abdellah and Keddam [4] was adopted to investigate the kinetics of formation of FeB and Fe<sub>2</sub>B layers on AISI M2 steel. This model was formulated by the use of the continuity equations at the two interfaces (FeB/Fe<sub>2</sub>B) and (Fe<sub>2</sub>B/substrate). Two temperature- dependent parameters were incorporated into this model to take into account the presence of boride incubation times during the formation of FeB and Fe<sub>2</sub>B layers. Recently, a diffusion model proposed by Keddam and Kulka [2] and called the integral method was applied to the boronizing kinetics of AISI D2 steel. This diffusion model required a numerical resolution of

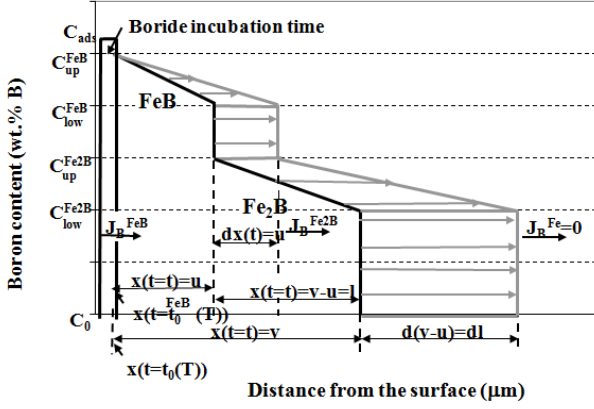
Differential Algebraic Equations (DAE) for its experimental validation.

In the current work, an integral diffusion model was proposed in order to predict the layers' thicknesses of FeB and Fe<sub>2</sub>B on AISI M2 steel for specified boriding conditions. A particular solution of differential algebraic equations (DAE) was used to evaluate the boron diffusivities in the FeB and Fe<sub>2</sub>B layers grown on AISI M2 steel. The values of activation energies for boron diffusion in the FeB and Fe<sub>2</sub>B layers were also estimated. Furthermore, a numerical solution of the resulting DAE system was obtained for predicting the thicknesses of FeB and Fe<sub>2</sub>B layers obtained at 1173, 1223, 1273 and 1323 K for 10 h. The predicted results in terms of layers' thicknesses were in good concordance with the experimental data.

The present research paper is organized in 5 sections. Section 1 presents an overview of existing research background and significance of the studied problem. In Section 2 is formulated the integral diffusion model. The simulation results are grouped in Section 3. A comparison was made in terms of boron activation energies with existing literature data in Section 4. This research work ends with a conclusion containing the main simulation results and prospects.

## 2. INTEGRAL DIFFUSION MODEL

In this approach, the continuity equations for the two interfaces (FeB/Fe<sub>2</sub>B) and (Fe<sub>2</sub>B/substrate) are considered with the inclusion of boride incubation periods. Figure 1 gives a schematic illustration of the boron concentration profiles through the FeB and Fe<sub>2</sub>B layers.



**Figure 1.** A schematic illustration of concentration profile of boron in each boride layer

$C_{up}^{FeB}$  and  $C_{low}^{FeB}$  (=16.23 wt.%B) denote the values of upper and lower boron contents in FeB.  $C_{up}^{Fe_2B}$  (=9 wt.%B) and  $C_{low}^{Fe_2B}$  (=8.83 wt.%B) represent the values of upper and lower boron contents in Fe<sub>2</sub>B.  $C_{ads}$  is the boron quantity adsorbed at the material surface [11]. The variable  $u$  is the FeB layer thickness whereas  $v$  represents the total boride layer (FeB + Fe<sub>2</sub>B).  $C_0$  is the solubility limit of boron in the substrate whose value is  $35 \times 10^{-4}$  wt.% B [13-14]. The assumptions assumed for the integral method can be found elsewhere [2]. The distance  $u$  changes with treatment time according to Equation (1), with  $k_{FeB}$  the value of parabolic growth constant of FeB.

$$u = k_{FeB} [t - t_0^{FeB}(T)]^{1/2} \quad (1)$$

The distance  $v$  varies with the process time according to Equation (2).

$$v = k [t - t_0(T)]^{1/2} \quad (2)$$

where,  $t_0(T)$  is the boride incubation time of the total boride layer and  $t_0^{FeB}(T)$  is the boride incubation time of FeB layer. Equation (1) can be rewritten in the following form by considering the boride incubation period of (FeB+Fe<sub>2</sub>B) layer:

$$u = k' [t - t_0(T)]^{1/2} \quad (3)$$

where,  $k'$  is the new value of parabolic growth constant at the (FeB/Fe<sub>2</sub>B) interface for a boride incubation time of  $t_0(T)$ .

The initial conditions are established as:

$$\begin{aligned} C_{FeB} \{x(t > 0) = 0\} &= 0 \\ C_{Fe_2B} \{x(t > 0) = 0\} &= 0 \\ C_{Fe} \{x(t > 0) = 0\} &= 0 \end{aligned} \quad (4)$$

The boundary conditions are expressed by:

$$C_{FeB} \{x[t = t_0^{FeB}(T)] = 0\} = C_{up}^{FeB} \quad \text{if } C_{ads} > 16.23 \text{ wt.\% B} \quad (5)$$

$$C_{FeB} \{x[t = t_0^{FeB}(T)] = 0\} = C_{low}^{FeB}$$

$$\text{if } C_{ads} < 16.23 \text{ wt.\% B} \text{ and with FeB phase} \quad (6)$$

$$C_{Fe_2B} \{x[t = t_0(T)] = 0\} = C_{up}^{Fe_2B}$$

$$\text{if } 8.83 \text{ wt.\% B} < C_{ads} < 16.23 \text{ wt.\% B} \text{ and without FeB phase} \quad (7)$$

$$C_{Fe_2B} \{x[t = t_0(T)] = 0\} = C_{low}^{Fe_2B}$$

$$\text{if } C_{ads} < 8.83 \text{ wt.\% B} \text{ and without FeB phase} \quad (8)$$

$$C_{FeB}(x(t) = u) = C_{low}^{FeB} \quad (9)$$

$$C_{Fe_2B}(x(t) = u) = C_{up}^{Fe_2B} \quad (10)$$

$$C_{Fe_2B}(x(t) = v) = C_{low}^{Fe_2B} \quad (11)$$

$$C_{Fe}(x(t) = v) = C_0 \quad (12)$$

In this approach [2], the change in the boron concentration with time and diffusion distance in each boride layer is not linear and satisfies the Second Fick's law given by Equation (13):

$$D_B^i \frac{\partial^2 C_i(x,t)}{\partial x^2} = \frac{\partial C_i(x,t)}{\partial t} \quad (13)$$

where,  $i$ =(FeB or Fe<sub>2</sub>B) and  $D_B^i$  represents the diffusion coefficient of boron in the  $i$  phase. In the integral method, the profile of boron content in each boride layer has a parabolic form as suggested by the Goodman's method [15]. Such a shape of the boron concentration profile through each boride layer is required for the applicability of the integral method.

$$\begin{aligned} C_{FeB}(x,t) &= C_{low}^{FeB} + a_1(t)(u(t) - x) + b_1(t)(u(t) - x)^2 \\ &\quad \text{if } 0 \leq x \leq u \end{aligned} \quad (14)$$

$$\begin{aligned} C_{Fe_2B}(x,t) &= C_{low}^{Fe_2B} + a_2(t)(v(t) - x) + b_2(t)(v(t) - x)^2 \\ &\quad \text{if } u \leq x \leq v \end{aligned} \quad (15)$$

The parameters  $a_1(t)$ ,  $b_1(t)$ ,  $a_2(t)$ ,  $b_2(t)$ ,  $u(t)$  and  $v(t)$  should verify the boundary conditions given by Equations (5), (6), (7) and (8). By using the boundary condition on the surface, Equation (16) was derived:

$$a_1(t)u(t) + b_1(t)u(t)^2 = (C_{up}^{FeB} - C_{low}^{FeB}) \quad (16)$$

Equation (17) was deduced for the boundary condition at the (FeB/Fe<sub>2</sub>B) interface as follows:

$$a_2(t)[v(t) - u(t)] + b_2(t)[v(t) - u(t)]^2 = (C_{up}^{Fe_2B} - C_{low}^{Fe_2B}) \quad (17)$$

The two ordinary differential equations given by Equations (18) and (19) are obtained from the integration of Second

Fick's law between the two limits 0 and u(t) for FeB, and between u(t) and v(t) for Fe<sub>2</sub>B.

$$\frac{d}{dt} \left[ \frac{u(t)^2}{2} a_1(t) + \frac{u(t)^3}{3} b_1(t) \right] = 2D_B^{FeB} b_1(t) u(t) \quad (18)$$

$$2w_{12} \frac{dv(t)}{dt} + \frac{[v(t)-u(t)]^2}{2} \frac{da_2(t)}{dt} + \frac{[v(t)-u(t)]^3}{3} \frac{db_2(t)}{dt} \quad (19)$$

$$= 2D_B^{Fe_2B} b_2(t) [v(t)-u(t)]$$

The first algebraic constraint of this diffusion problem can be derived from the continuity equation at the (Fe<sub>2</sub>B/substrate) interface as follows [2]:

$$2w_1 b_1(t) D_B^{FeB} = D_B^{FeB} a_1^2(t) - D_B^{Fe_2B} a_1(t) (a_2(t) + 2b_2(t) [v(t) - u(t)])$$

$$\text{with } w_1 = \left[ \frac{(C_{up}^{FeB} + C_{low}^{FeB})}{2} - C_{up}^{Fe_2B} \right] \quad (20)$$

In the same manner, the second algebraic constraint can be obtained from the continuity equation at the (Fe<sub>2</sub>B/substrate) interface as follows:

$$2w_{12} b_1(t) D_B^{FeB} a_2(t) + 2w_2 b_2(t) D_B^{Fe_2B} a_1(t) \quad (21)$$

$$= D_B^{Fe_2B} a_2^2(t) a_1(t)$$

$$\text{with } w_2 = \left[ \frac{(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})}{2} - C_0 \right] \text{ and } w_{12} = \frac{(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}{2}$$

Equations (16), (17), (18), (19), (20) and (21) constitute a DAE system to be solved. The variables a<sub>1</sub>(t), a<sub>2</sub>(t), b<sub>1</sub>(t), b<sub>2</sub>(t), u(t) and v(t) should verify the algebraic constraints given by Equation (16), (17), (20) and (21) at boride incubation time t<sub>0</sub>(T) if the values of boron diffusion coefficients in FeB and Fe<sub>2</sub>B are known. A particular solution of this type of diffusion problem was reported in a recent reference work [2] (see this publication for further details) to determine the boron diffusion coefficients in the FeB and Fe<sub>2</sub>B layers by putting the following new change of variables given by:

$$a_1(t) = \frac{\alpha_1}{u(t)}, b_1(t) = \frac{\beta_1}{u(t)^2}, a_2(t) = \frac{\alpha_2}{[v(t) - u(t)]}$$

$$b_2(t) = \frac{\beta_2}{[v(t) - u(t)]^2} \quad (22)$$

where, the constants α<sub>1</sub>, β<sub>1</sub>, α<sub>2</sub> and β<sub>2</sub> should satisfy the boundary conditions. By applying this new change of variables, the expression of boron diffusion coefficient in the FeB layer was found.

$$D_B^{FeB} = k'^2 \left[ \frac{(C_{up}^{FeB} - C_{low}^{FeB})}{8\beta_1} - \frac{1}{24} \right] \text{ for } \beta_1 < 3(C_{up}^{FeB} - C_{low}^{FeB}) \quad (23)$$

The expression of boron diffusion coefficient in the Fe<sub>2</sub>B layer was expressed by Equation (24):

$$D_B^{Fe_2B} = \frac{k(k-k')(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}{4\beta_2} - (k-k')^2 \left[ \frac{(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}{8\beta_2} + \frac{1}{24} \right] \quad (24)$$

After determining the boron diffusivity in each boride layer, a computer program was created by using the Petzold's DAE solver DASPK [16] with the purpose of evaluating the output variables u(t) and v(t) for a given treatment time and temperature.

### 3. RESULTS

The integral method was adapted to determine the values of diffusion coefficients of boron in the FeB and Fe<sub>2</sub>B layers. The experimental results provided by Campos-Silva et al. [5] on the pack-boriding of AISI M2 steel were employed for this purpose. In their experiments, the B<sub>4</sub>C Durborid was used as a boriding agent in the temperature range of 1173 to 1323 K with a holding time from 4 to 8 h. To guarantee the accuracy of layer thickness measurements, an average of eighty measurements was taken on different locations of cross-sections of the boronized specimens.

Table 1 provides the experimental values of parabolic growth constants at the two growing interfaces (FeB/Fe<sub>2</sub>B) and (Fe<sub>2</sub>B/substrate) along with the associated boride incubation times by fitting the experimental data [5] according to Equations (1) and (2).

**Table 1.** Experimentally determined values of the parabolic growth constants at each interface with the associated boride incubation periods

T (K)	k <sub>FeB</sub> (μm s <sup>-0.5</sup> )	t <sub>0</sub> <sup>FeB</sup> (T) (s)	k (μm s <sup>-0.5</sup> )	t <sub>0</sub> (T) (s)
1173	0.065	10131	0.168	8806.2
1223	0.121	6085.7	0.305	4729
1273	0.179	4347.8	0.448	4323
1323	0.238	3815.5	0.589	3742.7

To evaluate the values of boron diffusion coefficient in each boride layer by the integral method with Equations (23) and (24), it is necessary to estimate the new values of experimental parabolic growth constants at the (FeB/Fe<sub>2</sub>B) interface with respect to the incubation time t<sub>0</sub>(T) at each boriding temperature (see Table 2).

**Table 2.** New Experimental values of the parabolic growth constants at the (Fe<sub>2</sub>B/ substrate) interface related to the boride incubation times of the total boride layer

T (K)	k' (μm s <sup>-0.5</sup> )	t <sub>0</sub> (T) (s)
1173	0.0615	8806.2
1223	0.116016	4729
1273	0.17887	4323
1323	0.238	3742.7

Table 3 provides the computed values of boron diffusion coefficients in the FeB and Fe<sub>2</sub>B phases by the integral diffusion model for an upper boron concentration of 16.40 wt. % in FeB.

**Table 3.** Computed values of diffusion coefficients of boron in the FeB and Fe<sub>2</sub>B layers by the integral diffusion model for an upper boron concentration of 16.40 wt.% in FeB

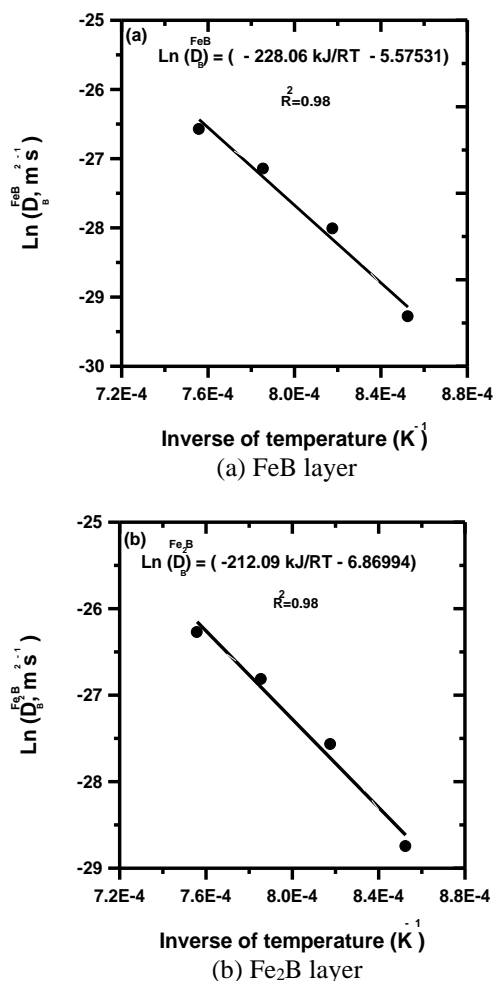
T (K)	$D_B^{FeB}$ ( $\times 10^{-12} m^2/s$ )	$D_B^{Fe_2B}$ ( $\times 10^{-12} m^2/s$ )
1173	0.231	0.327
1223	0.806	1.064
1273	1.862	2.257
1323	3.274	3.884

Figure 2 describes the change in the temperature of computed values of boron diffusivities in the FeB and Fe<sub>2</sub>B layers by using the integral diffusion model. The expressions of boron diffusivities in the FeB and Fe<sub>2</sub>B layers (expressed in m<sup>2</sup>/s) were given by Equations (25) and (26):

$$D_B^{FeB} = 3.79 \times 10^{-3} \exp\left(\frac{-228.06 kJ/mol}{RT}\right) \quad (25)$$

$$D_B^{Fe_2B} = 1.04 \times 10^{-3} \exp\left(\frac{-212.10 kJ/mol}{RT}\right) \quad (26)$$

with  $R=8.314 \text{ J mol}^{-1} \text{ K}^{-1}$  and T the absolute temperature in Kelvin.



**Figure 2.** Temperature dependence of simulated values of boron diffusivity in each boride layer

In Table 4 are gathered the reported values of activation energies for boron diffusion in different steels [3-5, 9, 12] with the values of activation energies obtained in this work.

Tables 5 and 6 list the predicted thicknesses of FeB and Fe<sub>2</sub>B layers using the integral method and the experimental thicknesses of FeB and Fe<sub>2</sub>B layers obtained at different temperatures for 10 h. In the integral method, the obtained DAE system was solved by using the Petzold's DAE solver DASPK [16]. For example, the parameters that fulfil the initial conditions for this DAE system are the following:  $a_1(t_0)=0.4449$ ,  $a_2(t_0)=0.27474$ ,  $b_1(t_0)=0.002316$ ,  $b_2(t_0)=0.004223$ ,  $u_0=0.38 \mu m$  and  $v_0=0.99 \mu m$  at 1323 K for a boride incubation time of 3742.7 s. A good concordance was observed between the predicted thicknesses and the experimental values.

**Table 4.** Reported values of boron activation energies in high alloy steels treated by solid boriding

Material	$Q_{FeB}$ (kJ mol <sup>-1</sup> )	$Q_{Fe_2B}$ (kJ mol <sup>-1</sup> )	Refs.
AISI M2	283.02±7.90	239.4±19.77	[9]
AISI 316	204	198	[12]
AISI D2	215 9	206 9	[3]
AISI M2	223	207	[5]
AISI M2	220.3	213	[4]
AISI M2	228.06	212.1	This work

**Table 5.** Comparison between the simulated FeB layers' thicknesses by integral method and experimental FeB layers' thicknesses obtained at increasing temperatures for 10 h

FeB layer thickness (μm)	1173 K	1223 K	1273 K	1323 K
Estimated values	10.82	18.94	29.91	45.77
Experimental values	10.17	20.98	28.3	40.24

**Table 6.** Comparison between the simulated Fe<sub>2</sub>B layers' thicknesses by integral method and experimental Fe<sub>2</sub>B layers' thicknesses obtained at increasing temperatures for 10 h

Fe <sub>2</sub> B layer thickness (μm)	1173 K	1223 K	1273 K	1323 K
Estimated values	18.77	30.87	46.05	66.84
Experimental values	19.66	32.81	51.83	72.28

#### 4. DISCUSSIONS

A particular solution of the resulting DAE system from the integral method was developed to estimate the values of boron diffusion coefficients in the FeB and Fe<sub>2</sub>B layers on AISI M2 steel in the temperature range 1173-1323 K. Recently, the integral model was successfully applied to analyze the kinetics of formation of Fe<sub>2</sub>B layers on different substrates [17-22]. The value of boron diffusivity in Fe<sub>2</sub>B was found to be directly proportional to the square of parabolic growth constant at the considered interface. In our study, the procedure used for determining the boron diffusivity in each boride layer was explained in detail in the reference work [2]. Such a procedure allowed us to perform the required computations. In Table 4 are listed the reported values of activation energies for boron diffusion in some high alloy steels. The obtained values of the activation energies are very comparable for AISI M2 steels treated by the powder-pack boriding or the paste-boriding method [4-5, 9]. In the reference work by Campos et al. [9], a diffusion model inspired from the Brakman's approach [23] and considered

the specific molar volume ratio between the two phases FeB and Fe<sub>2</sub>B as well as the specific molar volume ratio between Fe<sub>2</sub>B and substrate was applied for the pack-borided AISI M2 steel. A comprehensive diffusion model was also applied to AISI 316 steel for analyzing the kinetics of formation of the (FeB/Fe<sub>2</sub>B) bilayer and the diffusion zone [12].

It is seen that the reported values of boron activation energies for both layers (FeB and Fe<sub>2</sub>B) are high due to the presence of high contents of alloying elements in different substrates (i.e. AISI M2, AISI D2 and AISI 316 steels) which slows down the diffusion rate of boron atoms into the substrate. The present diffusion model was validated experimentally for four additional boriding conditions (1173, 1223, 1273 and 1323 K with a treatment time of 10 h). It is found the predicted values of FeB and Fe<sub>2</sub>B layers' thicknesses agreed with the experimental results displayed in Tables 5 and 6 by using a computer program written in Octave free software.

## 5. CONCLUSIONS

In this work, diffusion based on the integral method and applied to the boronizing kinetics of a high alloy steel (AISI M2) was presented. This kinetic approach considered the influence of boride incubation times on the kinetics of formation of FeB and Fe<sub>2</sub>B layers on AISI M2 steel. The validity of integral method was verified experimentally for other boriding conditions. Finally, the experimental values of FeB and Fe<sub>2</sub>B layers' thicknesses obtained at different temperatures for a treatment time of 10 h were compared to the predicted values. A good agreement was then observed between the experimental results and the simulated values.

As a prospect, the present kinetic approach can be adopted for any borided steel to simulate the kinetics of formation of FeB and Fe<sub>2</sub>B layers. It can also be extended to study the growth kinetics of FeB and Fe<sub>2</sub>B layers as well as the diffusion zone in steels. In general, this present integral diffusion model can be adapted for the study of diffusion phenomenon of an interstitial element in a system of three phases. Particularly, this kinetic approach can be used to simulate the nitriding kinetics of Armco iron with a double-phase compound layer (with  $\epsilon$  and  $\gamma'$  as iron nitrides).

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