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ANALYSIS OF THE GROWTH KINETICS OF Fe₂B LAYERS BY THE INTEGRAL METHOD

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Abstract

In this study, an alternative approach based on the integral method was proposed to estimate the values of boron diffusion coefficients in the Fe₂B layers grown at the surface of Armco iron. The set of differential algebraic equations (DAE) system was obtained to estimate the value of activation energy for boron diffusion when pack-boriding of Armco iron in the range of 1123 to 1273 K taking into account the boride incubation time.

The present model has been validated by making a comparison between the experimental value of Fe_2B layer thickness obtained at 1253 K for 5 h and the predicted results by using two different approaches. A good agreement was observed between these two set of data.

Keywords: Boriding; Iron boride; Kinetic model; Activation energy; Growth kinetics; Integral Method; DAE system.

List of symbols

u(t) is the boride layer thickness (µm).

a(t) and b(t) are the time-dependent parameters

k is the parabolic growth constant of the Fe_2B layer ($\mu ms^{\text{-}0.5}$).

t is the treatment time (s).

 $t_0(T)$ is the boride incubation time (s).

 $C_{up}^{Fe_2B}$ represents the upper boron concentration in the Fe₂B phase (=9 wt.%).

 $C_{low}^{F\bar{e}_{2}B}$ is the lower boron concentration in the Fe₂B phase (=8.83wt.%).

 C_{ads} is the adsorbed boron concentration at the material surface (wt..%).

 C_0 is the solubility limit of boron within the iron matrix ($\gg 0$ wt.%).

 $C_{Fe_2B}[x,t]$ is the distribution of boron concentration in the Fe₂B layer (wt.%).

 $D_{g}^{Fe,B}$ represents the boron diffusion coefficient in the Fe₂B phase (m² s ⁻¹).

1. Introduction

Boriding or boronizing treatment is widely employed in the industries to enhance the surface characteristics of metallic alloys [1]. As a consequence of this thermochemical treatment, the surface hardness, the resistance to wear friction and abrasion are improved for the ferrous alloys like

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steels, cast irons and Armco iron. In this case, the boriding process results in the formation of either Fe₂B layer or a double layer (FeB+Fe₂B) as a function of the process parameters. However, the Fe₂B phase is more desirable than a double layer (FeB+Fe₂B) because high tensile stresses develop in the FeB phase which is harder than Fe₂B [2]. The boriding process can be carried out by using different processes such as: plasma boriding [3], plasma paste boriding [4, 5], gas-boriding [6, 7], liquid boriding [8], laser boriding [9] and solid boriding (paste or powder) [10-12]. Among all boriding methods, only powder packboriding has been widely employed in the industry since it is simpler and more economic. The modeling of boriding kinetics is a relevant tool used to optimize the boriding parameters in order to attain a desired boride layer thickness in accordance with the practical use of treated parts.

For this purpose, several kinds of approaches were cited in the reference works for analyzing the kinetics of formation of Fe_2B layers formed on ferrous materials [13-19] with the presence or not of the boride incubation times. For example, Elias-Espinosa et al.[13] proposed a kinetic model for investigating the kinetics of formation of AISI O1 steel by using the pack-boriding process. The principle of mass conservation at the (Fe₂B/substrate) interface was considered to solve the diffusion problem with the presence of boride incubation times.



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They found a value of activation energy for boron diffusion in AISI O1 steel equal to 197.2 kJ mol⁻¹. Kouba et al. [14] suggested an alternative approach for studying the boriding kinetics of Armco-iron. In their model, the surface boron flux was used as a fitting parameter for reproducing the experimental values of boride incubation times during the formation of Fe₂B layers on Armco iron. Similarly, diffusion models [20-22] were also applied to the Ti-B system for simulating the kinetics of formation of Ti borides on Cp-Ti and Ti6Al4V substrates.

In the current study, an alternative diffusion model [23-25] using the integral method was applied to determine the boron diffusivity in the Fe_2B layers on the Armco iron substrate in the range of 1123 to 1273 K.

The present kinetic approach was firstly used by Cazares et al. [26] to model the growth kinetics of plasma nitrided pure iron. In addition, the value of boron activation energy in Armco-iron was determined by using this model and a comparison was made with the data found in the reference works.

Finally, an experimental validation was done for two different approaches by using the experimental value of Fe_2B layer thickness obtained at 1253 K for 5 h.

2. The integral method

This mathematical approach was developed for describing the growth of Fe_2B layer formed on the iron substrate. A schematic illustration of the boron concentration– profile through the Fe_2B layer is displayed in Figure 1.



Distance from the surface (µm)

Figure 1. Boron concentration profile through the Fe_2B layer

In contact with the boriding agent, the boron atoms penetrate into the iron lattice bv thermodiffusion to form a saturated solid solution. After a time surpassing the boride incubation time $t_0(T)$, the Fe₂B layer starts to form and becomes continuous and more compact after a prolonged treatment time. $C_{up}^{Fe_2B}$ denotes the upper boron concentration in Fe₂B (=9 wt.%), $C_{low}^{Fe_2B}$ is the lower boron concentration in Fe₂B (=8.83 wt.%) and x(t)=u represents the Fe₂B layer thickness. The Fe₂B phase exhibited a narrow composition range (of about 1 at. % B) as reported by Brakman et al.[27]. The term C_{ads} denotes the adsorbed boron concentration at the material surface [28]. C_0 is the solubility limit of boron within the material substrate which can be neglected (≈0 wt.%) [29, 30]. The following assumptions considered during the establishment of diffusion model can be found elsewhere [23-25].

Initial condition :

 $t = 0, x \succ 0$, with $C_{Fe_2B}[x(t), t = 0] = C_0 \approx 0$ wt.% (1) Boundary conditions:

 $C_{Fe_{2}B}[x(t=t_{0})=0,t=t_{0}] = C_{up}^{Fe_{2}B} \text{ for } C_{ads} \succ 8.83 \text{ wt.}\% (2)$ $C_{Fe_{2}B}[x(t=t)=u(t),t=t] = C_{low}^{Fe_{2}B} \text{ for } C_{ads} \prec 8.83 \text{ wt.}\% (3)$ The boron concentration profile is described by the Second Fick's law as follows:

$$D_B^{Fe_2B} \frac{\partial^2 C_{Fe_2B}[x,t]}{\partial x^2} = \frac{\partial C_{Fe_2B}[x,t]}{\partial t}$$
(4)

where the boron diffusion coefficient is only dependent on the boriding temperature. It is possible to obtain the expression of boron-concentration profile through the Fe_2B layer using the Goodman's method also called the heat balance integral method (HBIM) [31].

$$C_{Fe_{2}B}[x,t] = C_{low}^{Fe_{2}B} + a(t)(u(t) - x) + b(t)(u(t) - x)^{2}$$

for $0 \le x \le u$ (5)

By adopting such a shape, the distribution of boron concentration inside the Fe_2B layer depends on the three time-dependent unknowns a(t), b(t) and u(t). It is also noted that the two parameters a(t) and b(t) must be positive because of a decreasing nature of the boron-concentration profile. Equation (6) was deduced for a concentration value at x=u(t) equal to the lower boron concentration in the Fe_2B phase to get the first algebraic constraint:

$$a(t)u(t) + b(t)u(t)^{2} = (C_{uv}^{Fe_{2}B} - C_{low}^{Fe_{2}B})$$
(6)

Equation (7) was obtained by integrating the second Fick's law between the two limits 0 and u(t):

$$\frac{u(t)^{2}}{2}\frac{da(t)}{dt} + a(t)u(t)\frac{du(t)}{dt} + \frac{u(t)^{3}}{3}\frac{db(t)}{dt} + b(t)u(t)^{2}\frac{du(t)}{dt} = 2D_{B}^{Fe_{2}B}b(t)u(t)$$
(7)



Equation (8) based on the principle of mass conservation at the (Fe_2B /substrate) interface was used to get the second algebraic constraint:

$$W \frac{dx}{dt}\Big|_{x=u} = -D_B^{Fe_2B} \frac{\partial C_{Fe_2B}[x,t]}{\partial x}\Big|_{x=u}$$
(8)
with $W = \left[\frac{(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}{2} + (C_{low}^{Fe_2B} - C_0)\right]$

Equation (8) can be re-written in the following form:

$$W(-\frac{\frac{\partial C_{Fe_2B}[\mathbf{x},t]}{\partial t}\Big|_{x=u}}{\frac{\partial C_{Fe_2B}[\mathbf{x},t]}{\partial \mathbf{x}}\Big|_{x=u}}) = -D_B^{Fe_2B} \left.\frac{\partial C_{Fe_2B}[\mathbf{x},t]}{\partial \mathbf{x}}\right|_{x=u}$$
(9)

Finally, Equation (10) was obtained after derivation and some mathematical manipulations:

$$(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})b(t) = a(t)^2$$
(10)

Equations (6), (7) and (10) form the DAE system whose the unknowns are a(t), b(t) and u(t).

This resulting DAE system can be solved either analytically [23-25] or numerically [32]. To get the expression of boron diffusion coefficients in Fe₂B, an analytic solution exists for this diffusion problem by setting:

$$u(t) = k[t - t_0(T)]^{1/2}$$
(11)

and by choosing the expressions for a(t) and b(t) as :

$$a(t) = \frac{\alpha}{u(t)} \tag{12}$$

$$b(t) = \frac{p}{u(t)^2} \tag{13}$$

u(t) represents the thickness of Fe₂B layer, $t_0(T)$ the associated incubation time and k the parabolic growth constant of the Fe₂B layer. The incubation time is the required period to get a continuous and compact Fe₂B layer at the surface of iron substrate. It is noticed that that the use of Equation (11) is valid from a practical point of view since it has been observed in many experiments.

Mathematically, Equation (11) can also be rewritten as follows:

$$u(t) = k_1 \sqrt{t} \tag{14}$$

where k_1 is the new parabolic growth constant at th (Fe₂B/Fe) interface. The two unknown parameters α and β involved in Equations (12) and (13) have to be determined by substituting the expressions of timedependent parameters a(t) and b(t) into Equations (6) and (10). The two following algebraic equations were then obtained:

$$\alpha + \beta = (C_{up}^{Fe_2B} - C_{low}^{Fe_2B})$$
(15)

$$(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})\beta = \alpha^2$$
(16)

The possible solutions of algebraic system formed by Equations (15) and (16) are the following:

$$\alpha = \frac{(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})[-1 + \sqrt{1 + 4(\frac{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}})]}}{2} \quad (17)$$

$$\beta = (C_{up}^{Fe_2B} + C_{low}^{Fe_2B}) \left[\frac{\frac{2 + 4(C_{up}^{Fe_2B} - C_{low}^{Fe_2B}) - C_{low}^{Fe_2B} - C_{low}^{Fe_2B})}{4}}{\frac{2}{\sqrt{1 + 4(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}}{\frac{2}{\sqrt{1 + 4(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}}}{4}} \right]$$
(18)

After substitution of Equations (11), (12) and (13) into Equation (7) and derivation with respect to the time, Equation (19) was deduced [23-25]. This last equation allows us to calculate the value of boron diffusion coefficient in Fe₂B at the considered boriding temperature.

$$D_B^{Fe_2B} = \eta k^2 \tag{19}$$

with

$$\eta = \left[\left(\frac{1}{16}\right) \left(\frac{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}\right) \left(1 + \sqrt{1 + 4\left(\frac{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}}\right)}\right) + \left(\frac{1}{12}\right) \right]$$

where n=13 3175

where $\eta = 13.3175$.

The two dependent-parameters a(t) and b(t) are finally expressed by Equations (20) and (21):

$$a(t) = \frac{(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})[-1 + \sqrt{1 + 4(\frac{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}})]}}{2k[t - t_0(T)]^{1/2}} \quad (20)$$

$$b(t) = (C_{up}^{Fe_2B} + C_{low}^{Fe_2B}) \left[\frac{\frac{2 + 4(\frac{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}})}{4k^2[t - t_0(T)]} - \frac{2\sqrt{1 + 4(\frac{C_{up}^{Fe_2B} - C_{low}^{Fe_2B}}{C_{up}^{Fe_2B} + C_{low}^{Fe_2B}})}}{4k^2[t - t_0(T)]} \right] \quad (21)$$

It is seen that the parameters a(t) and b(t) are found to be positive.

3. Results

3.1 Estimation of activation energy for boron diffusion

The experimental data published in the paper [33] about the boriding kinetics of Armco were employed to calculate the boron diffusivities in the Fe₂B layers.



For information, the pack-boriding treatment was carried out to get the Fe₂B layers on Armco iron in the range of 1123 to 1273 K for 2, 4, 6 and 8 h. A container placed in the furnace and filled with a Durborid powder mixture with a particle size of 50 μ m was used for this thermochemical treatment. Fifty measurements were made in different locations of the cross-sections of borided specimens to determine the Fe₂B layers' thicknesses. The square of boride layer thickness was plotted against the boriding time according to Equation (11) as shown in Figure 2.



Figure 2. Square of Fe_2B layer thickness against the treatment time at different boriding temperatures.

The slopes of the obtained straight lines provide the square of parabolic growth constants. The boride incubation time can be obtained at each boriding temperature for a null boride layer thickness. The experimental parabolic growth constants at the (Fe₂B/substrate) interface and the boride incubation times [33] by using Equation (11) are displayed in Table 1.

 Table 1. The experimental parabolic growth constants at the (Fe₃B/substrate) interface and the corresponding boride incubation times according to Equation (11)[33].

	Experimental values of	Boride
T(K)	parabolic growth constant	Incubation time
	$k \; (\mu m \; s^{-0.5})$	$t_0(T)$ (s)
1123	0.57	1757.9
1173	0.8665	1790.6
1223	1.1886	1758.1
1273	1.547	1758.1

From Table 1, the boride incubation times are nearly constant in the range of 1123 to 1273 K.The values of diffusion coefficients of boron in Fe₂B can be estimated by using Equation (14) after considering the new values of experimental parabolic growth constants k_1 displayed in Table 2.

Table 2. Experimental values of new parabolic growth constants at the (Fe₂B/substrate) interface according to Equation (14).

T(K)	Experimental values of new parabolic growth constant $k_1 (\mu m \ s^{-0.5})$
1123	0.5468
1173	0.8305
1223	1.1401
1273	1.4839

So, the value of boron activation energy for Armco iron was deduced by plotting the natural logarithm of boron diffusion coefficient in Fe₂B versus the inverse of temperature. Figure 3 describes the variation of calculated values of boron diffusion coefficients in the Fe₂B layers as a function of reciprocal temperature and expressed by Equation (22):

$$D_B^{Fe_2B} = 9.2752 \times 10^{-5} \exp(\frac{-157.9 k J mol^{-1}}{RT})$$
(22)

where R=8.314 Jmol⁻¹K⁻¹ and T the absolute temperature in Kelvin.



Figure 3. Evolution of diffusion coefficient of boron in the Fe_2B layer as a function of inverse temperature.

Table 3 lists the reported values of activation energies for boron diffusion [10, 16, 27, 33-36] in



Armco iron by using different boriding methods.

Table 3. Values of activation energies for boron diffusion in the Fe₂B layers for Armco iron depending on the boriding method.

Boriding method	Activation energy for boron diffusion in Fe ₂ B (kJ mol ⁻¹)	References
Gaseous	117.5	[34]
Gaseous	120.65	[35]
Gaseous	105.5	[36]
Paste	157	[16]
Paste	151	[10]
Powder	157	[27]
Powder	157.5	[33]
Powder	157.9	Present study



In order to check the experimental validation of the integral method, the experimental value of boride layer thickness obtained at 1253 K for 5 h was used. Table 4 shows a comparison between the experimental Fe₂B layer thickness obtained at 1253 K during 5 h and the predicted value by solving numerically the DAE system for an upper boron concentration in Fe₂B of 9%. A good concordance was obtained when comparing the experimental Fe₂B layer thickness [33] with the predicted values by using these two approaches. A computer program written in Octave free software was created by using Petzold's DAE solver DASPK [37] to get the numerical solution of DAE system.

Therefore, the numerical solution by the integral method was obtained without assuming a priori a parabolic growth law for the Fe₂B layer and considering the boride incubation time of 1758.1 s with the initial values of $u(t_0)=0.01 \ \mu m$, $a(t_0)=16.8409$ and $b(t_0)=15.9067$.

For the diffusion model [13], the simulated value of Fe_2B layer thickness was estimated from Equation (23):

$$u(t) = \frac{4}{\sqrt{\pi}} \frac{(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})}{(C_{up}^{Fe_2B} + C_{low}^{Fe_2B})} \frac{e^{-\varepsilon^2}}{erf(\varepsilon)} \sqrt{D_B^{Fe_2B}t}$$
(23)

with
$$D_B^{Fe_2B} = 1.68 \times 10^{-4} \exp(-\frac{157.94kJ}{RT})$$

and ϵ =0.0973

where $D_B^{Fe_2B}$ is given in (m² s⁻¹) for an upper boron concentration in the Fe₂B phase equal to 9 wt.%. It is also noticed that the determined value of boron activation energy for Armco iron from the diffusion model [13] is very comparable to the result given by the integral method.

Table 4 The experimental Fe₂B layer thickness obtained at 1253 K for 5 h and the values of Fe₂B layer thickness estimated numerically by the integral method and from the diffusion model [13] for an upper boron content in the Fe₂B phase equal to 9 wt.%.

Boriding conditions	Experimental Fe ₂ B layer thickness (µm) [33]	Simulated Fe ₂ B layer thickness (μ m) by the integral method	Simulated Fe ₂ B layer thickness (µm) by the diffusion model [13]
1253 K for 5 h	175.25 ±12.15	171.95 μm	172.79 μm

Table 5 compares the experimental values of Fe_2B layer thickness [33] and the predicted values obtained from the integral method at 1123 and 1273 K for variable treatments times. It is seen that the integral method reproduces in a satisfactory manner the experimental results obtained at 1123 K and 1273 K for 2, 4, 6 and 8 h.

Table 5 The experimental values of Fe₂B layer thickness [33] and the predicted values obtained from the integral method at 1123 and 1273 K for variable treatments times.

Time (h)	Predicted Fe ₂ B layer thickness by the integral methodat 1123 K	Experimenta 1 Fe ₂ B layer thickness obtained at 1123 K	Predicted Fe ₂ B layer thickness by the integral method at 1273 K	Experimental Fe ₂ B layer thickness obtained at 1273 K
2	41.39	40.34 ±6.7	112.1	109.47 ± 8.6
4	63.09	61.48 ± 5.6	170.88	166.84 ±4.1
6	79.04	77.02 ±3.8	214.08	209.03 ±5.3
8	92.27	89.92 ±4.6	249.93	244.02 ±5.1

4. Discussions

The experimental results on the pack-borided Armco iron was used to calculate the boron diffusivities in the Fe_2B layers in the range of 1123-1273 K by using Equation (19) derived from the integral method. Afterwards, the value of activation energy for boron diffusion in Armco iron was calculated by assuming the Arrhenius relationship. Hence, the value of the boron activation energy was estimated as equal to 157.9 kJ mol⁻¹ for Armco iron.

The obtained value of activation energy for boron diffusion in Armco iron can be interpreted as the required amount of energy that stimulates the diffusion of boron atoms [10] along the preferred crystallographic direction [001]. From Table 3, it is seen that the published results [10, 16, 27, 33-36] in terms of activation energies depend on the following



factors: the boriding method, the boriding parameters, the procedure for the measurement of boride layer thickness, the nature of boriding agent, the mechanism of boron diffusion and the considered temperature ranges. The reported values of activation energies for boron diffusion in paste or pack-borided Armco iron are higher in comparison with those obtained from the gas-boriding process since the activity of boron depends on its concentration in the boriding agent and on the diffusion mechanism of boron atoms involved in the corresponding chemical reactions. The obtained values of activation energies for boron diffusion displayed in Table 3 are very comparable for the solid boriding (powder and paste) [10, 16, 27, 33-36] but different from the gas boriding process [34-36]. The discrepancy observed in the values of boron activation energies points out that the rate determining steps in powder and paste boriding deviate from that for gas boriding [33]. Finally, two different approaches (the integral method and the diffusion model taken from the reference work [13]) have been experimentally validated by comparing the predicted values with the experimental value of Fe₂B layer thickness obtained at 1253 K for 5 h. A good agreement was observed between the experiment and the simulation results.

5. Conclusions

- In the present study, an alternative diffusion model based on the integral model was suggested to analyze the kinetics of formation of Fe_2B layers on Armco iron.

- A set of differential algebraic equations (DAE) were obtained to calculate the boron diffusivities in the Fe₂B layers in the range of 1123 to 1273 K considering the occurrence of of boride incubation time which is nearly constant.

-The value of activation energy for boron diffusion was determined as equal to 157.9 kJ mol⁻¹ in Armco iron by assuming the Arrhenius relationship. This value of energy was in good agreement with the results published in the reference works.

- The validity of the two different approaches have been experimentally verified for the boriding condition (1253 K for 5 h).

- The present model can be extended for the determination of boron diffusivities in the FeB and Fe₂B layers formed on ferrous alloys.

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ANALIZA KINETIKE RASTA Fe, B SLOJEVA UZ POMOĆ INTEGRALNE METODE

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Abstract

U ovom radu je za procenu vrednosti koeficijenta difuzije borona u Fe₂B slojevima na površini Armco gvožđa predložen alternativni pristup zasnovan na integralnoj metodi. Sistem diferencijalnih algebarskih jednačina (DAE) je upotrebljen da bi se procenile vrednosti aktivacione energije za difuziju borona prilikom postupka pack boriranja Armco gvožđa u opsegu od 1123 do 1273 K uzimajući u obzir vreme inkubacije boride.

 $Ovaj model je potvrđen poređenjem eksperimentalnih vrednosti debljine Fe_2B sloja dobijenih pri temperature od 1253 K u trajanju od 5 sati i predviđenih rezultata korišćenjem dva različita pristupa. Primećeno je dobro slaganje između ova dva skupa podataka.$

Ključne reči: Boriranje; Borid gvožša; Kinetički model; Energija aktivacije; Kinetika rasta; Integralna metoda; DAE sistem.

