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ESTIMATION OF THE BORON DIFFUSION COEFFICIENTS IN FeB AND Fe_2B LAYERS DURING THE PACK-BORIDING OF A HIGH-ALLOY STEEL

DOLOČANJE KOEFICIENTA DIFUZIJE BORA V PLASTEH FeB IN Fe_2B MED BORIRANJEM VISOKO LEGIRANEGA JEKLA V SKRINJI

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In this work we propose a diffusion model to estimate the boron diffusion coefficients in FeB and Fe_2B layers during the pack-boriding of AISI M2 steel in the temperature range 1173–1323 K for a treatment time of 4–8 h.
The proposed model is based on the mass-balance equations at the two growth fronts – FeB/Fe_2B and Fe_2B/substrate – under certain assumptions. The estimated values of the boron activation energies in the FeB and Fe_2B layers were compared with the literature data. The present model was extended to predict the thickness of each boride layer for the borided samples at different temperatures for 10 h. Iso-thickness diagrams were established to be used as a tool for predicting the thickness of each boride layer as a function of the two parameters: temperature and time. Finally, a simple equation was proposed to estimate the required time to obtain a single Fe_2B layer by diffusion annealing.

Keywords: boriding, incubation times, Fick's laws, simulation, growth kinetics, annealing

Predstavljeno delo predlaga model difuzije za določanje koeficienta difuzije bora v plasteh FeB in Fe_2B med boriranjem v skrinji jekla AISI M2 v temperaturnem območju 1173–1323 K pri spreminjanju trajanja postopka od 4 h do 8 h.
Predlagani model temelji na enačbi masne balance na dveh rastočih mejnih ploskvah (FeB/Fe_2B) in (Fe_2B/osnova) pri določenih predpostavkah. Določena vrednost aktivacijske energije bora v FeB- in Fe_2B-plasti je bila primerjana s podatki iz literature. Predstavljeni model je bil razširjen, da bi lahko napovedal debelino vsake od obeh boridnih plasti za borirane vzorce pri različnih temperaturah in trajanju do 10 h. Postavljeni so bili diagrami enake debeline, ki so uporabni kot orodje za napovedovanje debeline vsakega od boriranih slojev v odvisnosti od dveh parametrov (temperature in časa). Predlagana je preprosta enačba za določanje potrebnega časa za nastanek plasti Fe_2B z difuzijskim žarjenjem.

Ključne besede: boriranje, inkubacijski čas, Fickovi zakoni, simulacija, kinetika rasti, žarjenje

1 INTRODUCTION

One of the surface-modification methods for improving the surface properties of ferrous and non-ferrous alloys is boriding. According to the Fe-B binary system, two kinds of iron borides, i.e., FeB and Fe_2B, with a narrow range of composition can be identified.[1] The boriding process applies in the temperature range 1073–1323 K between 1 h to 10 h and it can be carried out in solid, liquid or gaseous media. The possible formation of the FeB and Fe_2B iron borides depends upon various factors, such as the boron activity of the boriding medium, the chemical composition of the substrate, the process temperature and the treatment time. The morphology of the boride layers is influenced by the presence of alloying elements in the matrix. Saw-tooth-shaped layers are obtained in low-alloy steels, whereas in high-alloy steels, the interfaces tend to be flat. The modelling of the boriding kinetics is considered as a suitable tool to match the case depth with the intended industrial applications for this borided steel. So, the modelling of the growth kinetics for boride layers has gained much attention to simulate the boriding kinetics during recent decades.[2–26]

In the present work an original diffusion model is proposed to estimate the boron diffusion coefficients in the FeB and Fe_2B layers grown on AISI M2 steel by considering the boride incubation times. A non-linear boron-concentration profile is assumed through the boride layers. The mass-balance equations were applied to the two diffusion fronts: the FeB/Fe_2B and Fe_2B/substrate interfaces in the temperature range 1173–1323 K. In addition, a simple equation was proposed to estimate the required time to obtain a single Fe_2B layer by diffusion annealing.

2 THE DIFFUSION MODEL

The model takes into account the FeB/Fe_2B bilayer growth on the saturated substrate with boron atoms, as shown in **Figure 1**.

C_{up}^{FeB} and C_{low}^{FeB} (= 16.23 % B) are the upper and lower boron mass concentrations in the FeB, while C_{up}^{FeB} (= 9 % B) and C_{low}^{FeB} (= 8.83 % B) are, respectively, the upper

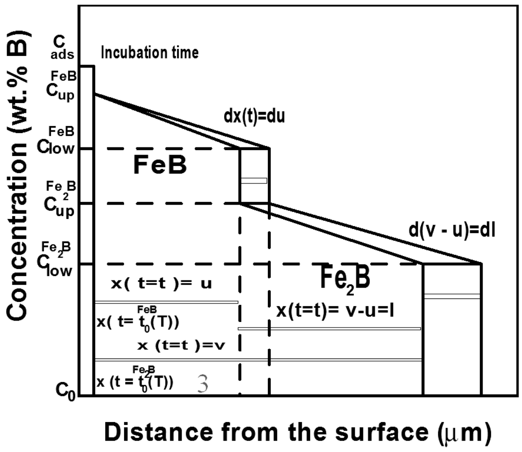

Figure 1: Boron concentration profile through the FeB/Fe$_2$B bilayer
Slika 1: Profil koncentracije bora skozi plasti (FeB/Fe$_2$B)

and lower boron concentrations in the Fe$_2$B. C_{ads} denotes the adsorbed concentration of boron,[15] while u is the position of the FeB/Fe$_2$B interface, and v is the position of the Fe$_2$B/substrate interface. C_0 is the boron solubility in the matrix and is equal to $35 \cdot 10^{-4}$ % B.[2] The upper boron content in the FeB phase (C_{up}^{FeB}), imposed by the boriding medium, gives rise to the two iron borides: FeB and Fe$_2$B. From a thermodynamic point of view, the FeB phase exhibits a narrow composition range (of about the mole fraction x = 1 % B or the mass fraction w = 0.2 % B), as identified by Massalski.[27] The upper boron content in the FeB phase was taken in the composition range of mass fractions 16.25–16.43 % B to obtain a bilayer configuration consisting of the two iron borides, FeB and Fe$_2$B.

The following assumptions are considered during the formulation of the diffusion model:

- The kinetics is dominated by the diffusion-controlled mechanism
- The growth of the boride layers is a consequence of the boron diffusion perpendicular to the sample surface
- The range of homogeneity of the iron borides is about x = 1 % B
- The iron borides nucleate after a certain incubation time
- The boride layer is thin in comparison to the sample thickness
- A local equilibrium occurs at the phase interfaces
- A planar morphology is assumed for the phase interfaces
- The volume change during the phase transformation is ignored
- The diffusion coefficient of boron in each iron boride does not vary with the boron concentration and follows an Arrhenius relationship
- A uniform temperature is assumed throughout the sample
- The alloying elements have no effect on the boron diffusion
- The presence of porosity is neglected during the boron diffusion.

The initial conditions of the diffusion problem are set up as follows:

$$\begin{aligned} &C_{FeB}\{x(t \succ 0)=0\}=0 \\ &C_{Fe_2B}\{x(t \succ 0)=0\}=0 \\ &C_{Fe}\{x(t \succ 0)=0\}=0 \end{aligned} \qquad (1)$$

The boundary conditions are given by the following equations:

$$C_{FeB}\left\{x[t=t_0^{FeB}(T)]=0\right\}=C_{up}^{FeB} \text{ for } C_{ads} \succ 16.23 wt.\%B \qquad (2)$$

$$C_{FeB}\left\{x[t=t_0^{FeB}(T)]=0\right\}=C_{low}^{FeB} \text{ for } C_{ads} \prec 16.23 wt.\%B$$

and with the FeB phase: (3)

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

$8.83 wt.\%B \prec C_{ads} \prec 16.23 wt.\%B$ and without the FeB phase: (4)

$$C_{Fe_2B}\left\{x[t=t_0^{Fe_2B}(T)]=0\right\}=C_{low}^{Fe_2B} \text{ for } C_{ads} \prec 8.83 wt.\%B$$

and without the FeB phase: (5)

$$C_{FeB}(x(t=t)=u)=C_{low}^{FeB} \qquad (6)$$

$$C_{Fe_2B}(x(t=t)=u)=C_{up}^{Fe_2B} \qquad (7)$$

$$C_{Fe_2B}(x(t=t)=v)=C_{low}^{Fe_2B} \qquad (8)$$

$$C_{Fe}(x(t=t)=v)=C_0 \qquad (9)$$

The mass-balance equations[28] are given by the equations (10) and (11):

$$w_{FeB}\left(\frac{du}{dt}\right)=\left[J_B^{FeB}-J_B^{Fe_2B}\right]_{x=u} \qquad (10)$$

$$w_{Fe_2B}\left(\frac{dv}{dt}\right)+w'\left(\frac{du}{dt}\right)=\left[J_B^{Fe_2B}\right]_{x=v} \qquad (11)$$

with

$$\begin{aligned} w_{FeB} &= \left[0.5\times(C_{up}^{FeB}-C_{low}^{FeB})+(C_{low}^{FeB}-C_{up}^{Fe_2B})\right] \\ w_{Fe_2B} &= \left[0.5\times(C_{up}^{Fe_2B}-C_{low}^{Fe_2B})+(C_{low}^{Fe_2B}-C_0)\right] \\ w' &= 0.5\times(C_{up}^{Fe_2B}-C_{low}^{Fe_2B}) \end{aligned}$$

The boron flux through a given boride layer is obtained from Fick's first law as follows:

$$J_B^i=-D_B^i\frac{\partial C_i(x,t)}{\partial x} \text{ with } i = (\text{FeB or Fe}_2\text{B}) \qquad (12)$$

D_B^{FeB} and $D_B^{Fe_2B}$ are, respectively, the diffusion coefficients of boron in the FeB and Fe$_2$B phases. The boron concentration profile in the FeB layer is given by:

$$C_{FeB}(x,t)=C_{up}^{FeB}+\frac{(C_{low}^{FeB}-C_{up}^{FeB})}{erf\left(\dfrac{u}{2\sqrt{D_B^{FeB}t}}\right)}\cdot erf\left(\frac{x}{2\sqrt{D_B^{FeB}t}}\right)$$

For $0 \le x \le u$ (13)

In the same way, the boron concentration profile in the Fe$_2$B layer can be obtained as follows:

$$C_{Fe_2B}(x,t)=C_{up}^{Fe_2B}+\frac{(C_{low}^{Fe_2B}-C_{up}^{Fe_2B})}{\left[erf\left(\frac{u}{2\sqrt{D_B^{Fe_2B}t}}\right)-erf\left(\frac{v}{2\sqrt{D_B^{Fe_2B}t}}\right)\right]}\cdot\left[erf\left(\frac{u}{2\sqrt{D_B^{Fe_2B}t}}\right)-erf\left(\frac{x}{2\sqrt{D_B^{Fe_2B}t}}\right)\right]$$

For $u \le x \le v$ (14)

The FeB layer thickness u grows parabolically according to equation (15), where k_{FeB} represents the parabolic growth constant at the FeB/Fe_2B interface:

$$u=k_{FeB}\left[t-t_0^{FeB}(T)\right]^{1/2} \quad (15)$$

The distance v is the location of the Fe_2B/substrate interface and k its parabolic growth constant (equation (16)) and the difference ($l = v - u$) denotes the layer thickness of the Fe_2B (equation 17):

$$v=k\left[t-t_0(T)\right]^{1/2} \quad (16)$$

$$l=v-u=k\left[t-t_0(T)\right]^{1/2}-k_{FeB}\left[t-t_0^{FeB}(T)\right]^{1/2} \quad (17)$$

with $t_0^{FeB}(T)\succ t_0(T)$ and $k\succ k_{FeB}$

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 the FeB layer. To take into account the effect of the boride incubation times when solving the mass-balance equations, it is necessary to define the two parameters $\beta_{FeB}(T)$ and $\beta(T)$:

$$\beta_{FeB}(T)=\left[1-\frac{t_0^{FeB}(T)}{t}\right]^{0.5} \quad (18)$$

and

$$\beta(T)=\left[1-\frac{t_0(T)}{t}\right]^{0.5} \quad (19)$$

The layer thickness of the FeB (u) is related to the $\beta_{FeB}(T)$ parameter by equation (20):

$$u=k_{FeB}\beta_{FeB}(T)\sqrt{t} \quad (20)$$

In the same way, the layer thickness of the Fe_2B (l) is expressed using equation (21):

$$l=\left[k\beta(T)-k_{FeB}\beta_{FeB}(T)\right]\sqrt{t} \quad (21)$$

3 ESTIMATION OF THE BORON DIFFUSION COEFFICIENTS IN THE FeB AND Fe_2B LAYERS

To estimate the boron diffusion coefficients in the FeB and Fe_2B layers, the experimental results published by Campos-Silva et al.[29] on borided AISI M2 steel were used. In this reference work, the powder-pack boriding was carried out at four temperatures, (1173, 1223, 1273 and 1323) K, for three exposure times, (4, 6 and 8) h, using the B_4C Durborid as a boriding medium. Eighty measurements were performed on different cross-sections of the borided samples from the AISI M2 steel to determine the thickness of each boride layer.

Tables 1 and **2** list the experimental parabolic growth constants for each phase interface with the corresponding incubation times. The experimental values of the parabolic growth constants at each phase interface were obtained from the slopes of the curves relating the squared boride layer thickness to the boriding time. The boride incubation times were deduced for a null boride layer thickness.

Table 1: Experimental values of the parabolic growth constants at the FeB/Fe_2B interface in the temperature range 1173–1323 K with the corresponding boride incubation times

Tabela 1: Eksperimentalne vrednosti konstant parabolične rasti na stiku (FeB/Fe_2B) v temperaturnem območju 1173–1323 K, z ustreznim inkubacijskim časom borida

T/K	Experimental growth Constants: k_{FeB}/(μm $s^{-1/2}$)	$t_0^{FeB}(T)$/s
1173	0.065	10131
1223	0.121	6085.7
1273	0.179	4347.8
1323	0.238	3815.5

Table 2: Experimental values of the parabolic growth constants at the Fe_2B/substrate interface in the temperature range 1173–1323 K with the corresponding boride incubation times

Tabela 2: Eksperimentalne vrednosti konstant parabolične rasti na stiku (Fe_2B/podlaga) v temperaturnem območju 1173–1323 K, z ustreznim inkubacijskim časom borida

T/K	Experimental growth Constants: k/(μm $s^{-1/2}$)	$t_0(T)$/s
1173	0.168	8806.2
1223	0.305	4729
1273	0.448	4323
1323	0.589	3742.7

It was demonstrated that the higher boriding temperatures involve the shorter incubation times,[24] as shown in **Tables 1** and **2**. The two respective parameters $\beta_{FeB}(T)$ and $\beta(T)$ are linearly dependent on the boriding temperature and can be approximated by equations (22) and (23) from a linear fitting of the experimental data displayed in **Figure 2**:

$$\beta_{FeB}(T)=(1\cdot 39\times 10^{-3}\,T-0.8579) \quad (22)$$

and

$$\beta(T)=(1\cdot 40\times 10^{-3}\,T-0.8470) \quad (23)$$

For this purpose, a computer code written in Matlab (version 6.5) was used to estimate the boron diffusivity in each boride layer. This program requires the following input data: the time, the temperature, the lower and upper boron concentrations at each phase interface as well as the two parameters $\beta_{FeB}(T)$ and $\beta(T)$. By solving the mass-balance equations (equations (10) and (11)) via the Newton-Raphson method,[30] it is possible to determine the boron diffusion coefficients in the FeB and Fe_2B layers. **Table 3** summarizes the estimated values of the boron diffusion coefficients in the FeB and Fe_2B layers for an upper boron content equal to w = 16.40 % in the FeB phase.

Figure 3 depicts the temperature dependence of the boron diffusion coefficients in the FeB and Fe_2B layers according to the Arrhenius equation. The value of the

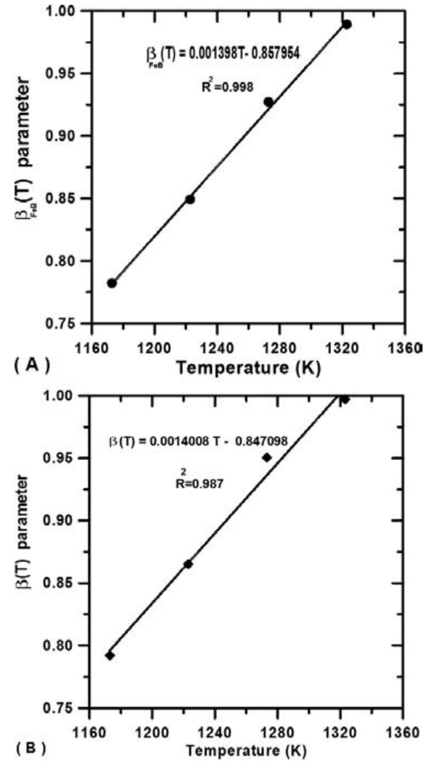

Figure 2: Evolution of the two parameters as a function of the boriding temperature: a) $\beta_{FeB}(T)$ and b) $\beta(T)$

Slika 2: Razvoj dveh parametrov v odvisnosti od temperature boriranja: a) $\beta_{FeB}(T)$ in b) $\beta(T)$

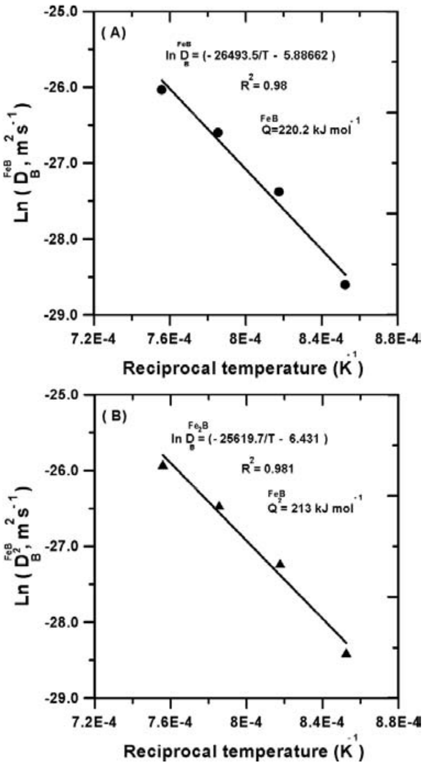

Figure 3: An Arrhenius relationship between the boron diffusion coefficient and the temperature: a) FeB layer, b) Fe_2B layer

Slika 3: Arrheniusova odvisnost med koeficientom difuzije bora in temperaturo: a) FeB-plast, b) Fe_2B-plast

Table 3: Determination of the boron diffusion coefficient in each boride layer for an upper boron mass fraction content of w =16.40 % in the FeB layer

Tabela 3: Določanje koeficienta difuzije bora v vsaki boridni plasti za zgornji masni delež vsebnosti bora 16,40 % v plasti FeB

T/K	$D_B^{FeB}(m^2s^{-1})\times10^{-12}$	$D_B^{Fe_2B}(m^2s^{-1})\times10^{-12}$
1173	0.376	0.462
1223	1.282	1.502
1273	2.797	3.227
1323	4.915	5.539

boron activation energy in each boride layer can be easily obtained from the slopes of the corresponding curves. So, the boron diffusion coefficients in the FeB and Fe_2B layers are, respectively, given by equations (24) and (25):

$$D_B^{FeB} = 2.8\times10^{-3} \exp\frac{-220.2\ kJ/mol}{RT} (m^2 s^{-1}) \quad (24)$$

$$D_B^{Fe_2B} = 1.6\times10^{-3} \exp\frac{-213\ kJ/mol}{RT} (m^2 s^{-1}) \quad (25)$$

where R is the universal gas constant (= 8.314 J/(mol K)), and T represents the absolute temperature in Kelvin.

The reported values of the activation energies[24,29,31–33] of the borided steels are listed in **Table 4** together with the values from this work. The obtained values of the activation energies are found to be dependent on the boriding method and on the chemical composition of the substrates.

In **Table 5**, a comparison was achieved between the experimental boride layer thicknesses and the simulated ones at different temperatures for 10 h of treatment. The simulated results were obtained from equations (20) and (21). The present model was able to predict the boride layer thickness (FeB or Fe_2B) for the given boriding conditions.

Table 4: Values of the boron activation energies obtained for different borided steels

Tabela 4: Vrednosti aktivacijske energije bora, dobljene iz različnih boriranih jekel

Material	Boriding method	Activation energy of FeB E/(kJ mol^{-1})	Activation energy of Fe_2B E/(kJ mol^{-1})	Reference
AISI M2	Paste	283	239.4	32
AISI4140	Paste	–	168.5	33
AISI H13	Powder-pack	–	186.2	31
AISI 316L	Powder-pack	204	198	24
AISI M2	Powder-pack	223	207	29
AISI M2	Powder-pack	220.2	213	Present study

Table 5: Experimental (exp.) and simulated (sim.) values of the boride layer thickness in the temperature range 1173–1323 K for 10 h of treatment, with an upper boron mass fraction of content of w = 16.40 % in the FeB phase

Tabela 5: Eksperimentalne (exp.) in simulirane (sim.) vrednosti za debelino plasti borida v temperaturnem območju 1173–1323 K, za 10 h obdelave pri gornjem masnem deležu vsebnosti bora 16,40 %, v FeB plasti

T/K	FeB (μm) exp.	FeB (μm) sim.	Fe_2B (μm) exp.	Fe_2B (μm) sim.
1173	10.17	10.30	19.66	16.70
1223	20.98	17.89	32.81	28.23
1273	28.30	29.75	51.83	45.81
1323	40.24	47.60	72.28	71.67

In **Table 6**, the predicted values of the boride layer thicknesses are compared with the experimentally determined values in the temperature range 1173–1323 K for a treatment time varying from 4 h to 8 h. Good agreement was observed between the experimental data and the simulation results for an upper boron content equal to w = 16.40 % in the FeB phase.

Table 6: Experimental (exp.) and simulated values (sim.) of the boride layer thickness in the temperature range 1173–1323 K for different treatment times with an upper boron content w = 16.40 % in the FeB phase

Tabela 6: Eksperimentalne (exp.) in simulirane (sim.) vrednosti debeline plasti borida pri temperaturah 1173–1323 K za različne čase obdelave in zgornjo vsebnostjo bora w = 16,40 % v FeB-fazi

T/K	Time (h)	FeB (μm) exp.	FeB (μm) sim.	Fe_2B (μm) exp.	Fe_2B (μm) sim.
1173	4	4.24	6.47	8.31	10.53
	6	6.96	7.92	12.04	12.89
	8	8.88	9.15	14.87	14.89
1223	4	11.03	11.32	18.96	17.85
	6	15.07	13.86	24.54	21.86
	8	18.23	16.00	29.08	25.24
1273	4	17.94	18.81	27.02	28.97
	6	23.51	23.04	35.37	35.48
	8	28.00	26.60	42.09	40.97
1323	4	24.48	24.89	36.31	35.90
	6	31.73	32.27	46.97	46.43
	8	37.61	38.25	55.61	54.98

Figure 4 displays the iso-thickness diagrams describing the evolution of the boride layer thickness as a function of the time and the boriding temperature. The results derived from **Figure 4** can be used as a tool to predict the boride layer thickness in relation with its practical use in an industrial area.

4 OBTAINING OF A SINGLE LAYER OF Fe_2B BY DIFFUSION ANNEALING

In industrial practice it is possible to reduce the brittleness of boride layers by controlling their microstructure. It is known that a single Fe_2B boride layer is more desirable than a dual FeB-Fe_2B layer.[34] This makes it possible to reduce the FeB layer thickness by applying a diffusion annealing in a hydrogen atmosphere. During this stage, the supply of boron is stopped since the concentration gradient of boron in the FeB is null (i.e., $C_{up}^{FeB} = C_{low}^{FeB}$ = 16.23 %), the FeB layer will be converted into an Fe_2B layer. The time required to eliminate the FeB layer during the diffusion annealing can be obtained from equation (26):

$$t_{u_{FeB}=0} = \frac{u \times l \times (C_{low}^{FeB} - C_{up}^{Fe_2B})}{D_B^{Fe_2B}(C_{up}^{Fe_2B} - C_{low}^{Fe_2B})} \tag{26}$$

where u is the FeB layer thickness (μm), l the Fe_2B layer thickness (μm) and $D_B^{Fe_2B}$ represents the boron diffusion coefficient in Fe_2B. It is clear that the annealing time depends on the boron diffusion coefficient in Fe_2B, and also on the thickness of each boride layer. During the diffusion annealing, an infinitesimal reduction of the FeB layer is related to the infinitesimal growth of the Fe_2B layer by equation (27):

$$\Delta u = -\left(\frac{w_{Fe_2B}}{w_{FeB} + w_{Fe_2B} + w'}\right)\Delta l = -0.5493\Delta l \tag{27}$$

The value of the Fe_2B layer thickness l' (μm) after diffusion annealing becomes:

$$l' = l + \frac{\Delta u}{0.5493} \tag{28}$$

Table 7 presents the simulation results obtained from equations (26) and (28) to estimate the Fe_2B layer thickness after diffusion annealing and the time required

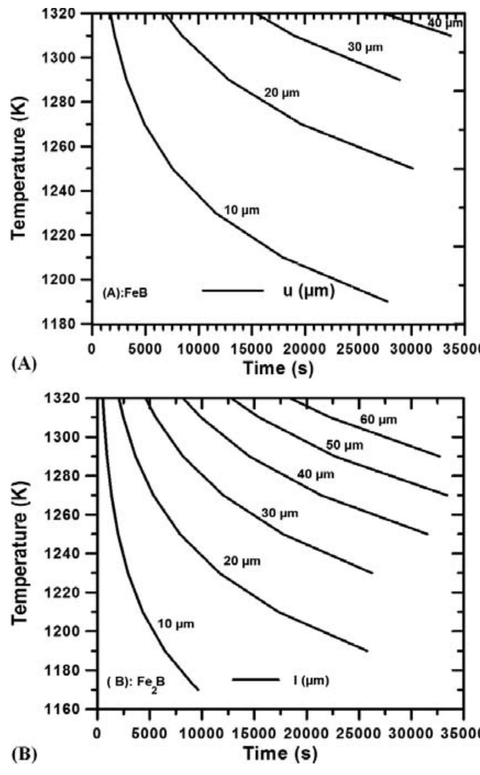

Figure 4: Iso-thickness diagrams describing the evolution of the boride layers: a) FeB, b) Fe_2B

Slika 4: Diagram enakih debelin opisuje razvoj boridnih plasti: a) FeB, b) Fe_2B

to eliminate the FeB layer in the case of the borided samples treated at different temperatures for 10 h. The obtained annealing times are increased with an increase of the boriding temperature since the boride layer becomes thicker. In this context, Kulka et al.[26] have experimentally determined the annealing time using a hydrogen atmosphere to obtain a single Fe_2B layer on gas borided Armco Fe at 1173 K for 2 h in a gas mixture (H_2–BCl_3). They found that the total elimination of the FeB layer took about 1 h.

Furthermore, it was shown by Dybkov et al.[35] that annealing of a borided Fe–Cr sample for 6 h resulted in the disappearance of the FeB layer.

Table 7: Estimation of the Fe_2B layer thickness and the time required to eliminate the FeB layer for the borided samples at different temperatures for 10 h

Tabela 7: Določanje debeline plasti Fe_2B in čas, potreben za odpravo FeB plasti, za vzorce, borirane 10 h pri različnih temperaturah

T/K	FeB (μm) sim.	Fe_2B (μm) sim.	Fe_2B (μm) After diffusion annealing Equation (28)	Annealing time $t_{u_{FeB}=0}$/h Equation (26)
1173	10.30	16.70	35.45	4.15
1223	17.89	28.23	60.79	4.99
1273	29.75	45.81	99.96	5.92
1323	47.60	71.67	158.32	6.93

5 CONCLUSIONS

In this work an original diffusion model was proposed to estimate the boron diffusion coefficients in the FeB and Fe_2B layers grown on AISI M2 steel. To determine the boron activation energy in each boride layer, the mass-balance equations were formulated, including the effect of the boride incubation times. The estimated boron activation energies were compared with the literature data. The present model was extended to predict the thickness of each boride layer for the borided samples at different temperatures for 10 h. Iso-thickness diagrams were established to be used as a tool to predict the thickness of each boride layer as a function of the two parameters (temperature and time). The required time to obtain a single Fe_2B layer by diffusion annealing was estimated on the basis of a simple equation. The formation of a single Fe_2B layer on AISI M2 steel depended on the boriding parameters.

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