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# The effect of boron-pack refreshment on the boriding of mild steel by the spark plasma sintering (SPS) process

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**Abstract**

Mild steel samples were borided with and without boron pack refreshment using the spark plasma sintering (SPS) process. Results show that the borided samples with boron pack refreshment developed a significantly thicker boride layer compared to that without boron pack refreshment for the same boriding duration. When boriding duration is t < 120 min, the boriding growth in the samples borided with boron pack refreshment followed a parabolic growth pattern. In contrast, the boride growth in samples processed without boron-pack refreshment deviates from parabolic at t=60 min. Computer simulation shows that the boron concentration change in the boriding media during the boriding process is an important factor affecting the composition and final thickness of the boride layer.

***Keywords:*** *Spark plasma sintering (SPS), Boriding, Growth kinetics, Non-steady-state diffusion, Microhardness*

## 1. Introduction

Boriding is a thermochemical treatment technique used in various metal surface engineering processes [1-3], in which boron atoms diffuse into the surface of a workpiece to form borides with the base metal. Owing to the nature of the diffusion process, the boride layers formed shows excellent adhesion to the base metal when compared to prevalent physical coating processes or various forms of advanced plating techniques. It offers a superior alternative to conventional surface hardening processes, such as carburizing, nitriding or carbonitriding [1, 4, 5], due to the superior hardness of borides (between 1500 to 2000 HV for ferrous borides). Besides its good wear resistance [2, 6, 7], the boride layer also has excellent corrosion and oxidation resistance, and a low coefficient of friction that greatly reduces the wear arising from adhesion, abrasion and surface fatigue [6].

The boriding process can be carried out in solid, liquid or gaseous medium [4, 6, 8-11]. Among these methods, only pack boriding has been widely used on a commercial basis. Yet the pack boriding process has the disadvantages of relatively high processing temperature (between 950 – 1100 °C) and long process duration (typically 4 – 15 h) for getting an effective boride layer thickness (~ 60 μm or more). Spark plasma sintering (SPS) boriding is introduced by Yu et al. to activate the pack boriding media as well as the workpiece with a high current discharge. [4, 12, 13] Previous studies [4, 13] showed that SPS boriding technique significantly lowers activation energy for accomplishing the boriding operation.

Several investigations have been carried out on the boriding kinetics of various metallic materials [1, 4, 9, 11, 14-23]. The thickness of the boride layer is determined by a number of factors. It is determined by the applied boriding method as well as the temperature and the treatment duration. Other factors like pack thickness of the boriding mixture [8] and chemical composition of the substrate material [14] can also affect the thickness of the boride layer. Jain et al. [8] observed that, for boriding treatment carried out at 940$^{o}$C for 2 h, a boriding mixture pack thickness less than 10 mm will results in a thin boriding layer thickness of no more than 60 μm. Martini et al. reported that the starting boronizing potential of the boriding powder mixture also plays an important role on the phase composition and growth kinetics of the borided layer [15]. It is expected that the boron potential change during the boriding process is also an importance factor affecting the final thickness of the boride layer. But in most reports on the boride layer

growth kinetics, this factor is not taken into account. By assuming a non-changing surface and interface B-concentrations, the boride layer thickness is only the function of boriding temperature and boriding duration.

In this study, mild steel samples are borided by the SPS technique, and the effect of refreshing the boriding pack mixture after certain boriding duration on the boriding layer thickness is examined. Scanning electron microscopy (SEM) is used to characterize the microstructure of the boriding layer. X-Ray Diffractometry (XRD) is used to identify the phase composition of the boriding layer. A theoretical model is proposed to describe the phenomena, taking account the depletion of the B-concentration of the boriding pack during the boriding process.

## 2. Experimental Setup

Mild steel of grade AISI 1018, with a carbon content of 0.2%, was used in this study. The mild steel rods, with diameter of 10 mm and length of 5 mm, were ground, polished and cleaned with acetone before boriding. The boriding processes were carried out in the Sumitomo Coal Mining (SCM) SPS system (DR SINTER Model 1050) at a fixed temperature of 850$^{o}$C. The boriding media contains $B_4C$ as the boron source and SiC as the diluent. The effects of boriding durations and refreshment of boron pack on the boriding growth kinetics were investigated in this study.

The main parameter investigated in this study is the boriding duration. The boriding parameters for the samples are tabulated in Table I. There were three sets of samples reserved for the boriding duration study. The first set (Set A) involved the once-through boriding of the mild steel for a full predetermined duration. The second set (Set B) of samples was borided for a total duration corresponding to the first set, except that the boriding was done with boron pack refreshment every 30 minutes. Other than the refreshing of the boron pack, all other conditions were kept constant. The third set (Set C) of the samples was first borided at 850°C for 240 minutes, and then borided again with refreshed boriding media mixture at intervals of 30 and 60 minutes.

After SPS boriding, the borided mild steel rods were sectioned, polished with Grade 4000 sandpaper and finally etched in 10% $HNO_3$ + 90% ethanol for 10 minutes to highlight the FeB and $Fe_2B$ phases. The X-ray diffraction (Philips 7198W XRD) was used to identify the different phases contained in the sample. The scanning electron

microscopy (SEM, JEOL JSM-5600LV, Japan) was employed to observe the morphology of the boriding layer. Since the boriding layer has a finger-like appearance, the definition of coating thickness is not obvious. A standard for the defining of the thickness of boriding layer was developed in a previous study [13], which considers both the height and the width of the boride fingers.

## 3. Results & Discussion

Fig. 1 shows the morphology of the mild steel samples borided at 850°C without boron pack refreshment (once-through boriding). Well-formed finger-like structures can be clearly observed from the SEM pictures. When the boriding duration is shorter than 90 minutes, boride layers with thin FeB layer on the top of $Fe_2B$ layer can be found. The thickness of the $Fe_2B$ phase is around three times that of the FeB phase. However, the FeB phase gradually vanishes with the further increase of the boriding duration, which is also confirmed by the XRD results [13]. The morphology of the borided samples with boron pack refreshment is shown in Fig. 2. The SEM pictures show that FeB phase is present in the boride layers even though the total boriding duration is as long as 150 min, indicating that different boriding processes were involved for the samples with and without boron pack refreshment.

The thickness of the boride layers vs. $t^{1/2}$ ($t$: total boriding duration) for the samples with and without boron pack refreshment is summarized in Fig. 3. The results in Fig. 1 and Fig. 3 indicate that, without boron pack refreshment, the total boriding growth (FeB+$Fe_2B$) does not satisfy parabolic growth after 60 minutes boriding duration. In a previous study, a mathematic model was developed to understand the abnormal growth phenomena [13] for the once-through borided samples. Those phenomena could be ascribed to the competition between two processes: the formation of the FeB phase and the phase transformation for FeB to $Fe_2B$ phase at the boriding temperature. In order to further investigate the boriding kinetics, boron pack refreshment was carried out every 30 minutes. As compared to Fig. 3, except for the total boriding duration of 60 minutes, it is found that the boride layers of the samples with boron pack refreshment are much thicker than that of the samples without boron pack refreshment for the same total boriding duration.

It can also be seen that, even if the boron pack refreshment method is used, the boriding layer does not fit the parabolic growth kinetics after a total boriding duration of

120 min (30 min × 4). This can be explained as following: Parabolic growth can be insured only when the boron concentration on the surface is enough to support a steady stat diffusion of boron in the boride layer. For the subsequent pack refreshment boriding, the boron atoms need time to diffuse to the FeB/$Fe_2B$ and $Fe_2B$/Fe interlayers, the thicker the boride layer from the previous boriding process, the longer the diffusion time. During this time, the boron concentration on the surface might already not enough for the steady state boron diffusion for prolonged boron pack refreshment boriding, results in the non-parabolic growth.

The microhardness vs. distance from the surface of the borided samples is shown in Fig. 4. For the samples without boron pack refreshment, the hardness profiles along the cross-sectional surface are almost the same. This means although the appearance boride layer thickness slightly increases with time for the once-through samples, as shown in Figure 3, the effective layer thickness for hardness test is not increased with time. The sample with boriding duration of 60 minutes reaches a low hardness of 200 HV at around 75 to 80 μm away from the top surface, while the rest of the samples maintained such hardness level at around 100 μm. And for a fixed hardness of 1000 HV, the distances from the surface for all once-through borided samples are around 60 μm with an error of ±10 μm. This result can be explained by the depletion of boron concentration in the boriding pack with the prolonged boriding time in the once-through boriding process. Although the boriding time is prolonged from 60, 80, 120 to 150 min, due to the insufficient boron atoms provided by the boriding pack, the effective boride layer thickness is not grown accordingly. However, for the samples with boron pack refreshment, different hardness profiles along the cross-sectional surface can be found from Fig. 4b. For a fixed hardness value, the longer the boriding duration, the deeper the distance from the top surface will be to reach such hardness value. For a fixed hardness value of 1000 HV, the distances from the top surface (the effective boriding thickness) are 35 μm, 70 μm, 100 μm and 120 μm for boriding duration of 30min×2, 30min×3, 30min×4 and 30min×5, respectively. This result can be attributed to the recovery of high boron concentration in the refreshment boriding process. With each refreshing cycle, the boron concentration in the boridng pack is maintained at a relatively high level, and a sustainable boron diffusion process can be maintained, resulting in a much thicker effective boriding layer thickness for the same prolonged boriding time. Because the FeB phase has much higher hardness than the $Fe_2B$ phase, the hardness results found in Fig.

4b could be attributed to the coexistence of FeB and $Fe_2B$ phases for all of the samples borided with boron pack refreshment.

## 4. Analysis

To understand the difference of the microstructure, phase composition and microhardness of the borided samples with and without boron pack refreshment, a special boriding case was investigated, e.g. a sample with once-through boriding duration of 240 minutes was adopted as the starting sample and borided again for 30 and 60 minutes in a refreshed boron pack (e.g. 240 min+30 min and 240 min+60 min). The XRD results of the three samples are compared in Fig. 5. It is found that the top sublayer transforms from almost pure $Fe_2B$ phase for 240 min boriding to combination of $Fe_2B$ and FeB for 240 min+30 min boriding and finally to pure FeB phase for 240 min+60 min boriding. During the once-through boriding process, when the boron concentration in the boriding media mixture ($B_4C$+SiC) depletes to a certain point, the chemical driving force is not high enough to incorporate the boron atoms into the FeB lattice, resulting in the consumption of FeB phase to form $Fe_2B$ phase. Thus, if the once-through boriding duration is long enough, such as 240 min in the study case, all of the FeB phase will be consumed to form the $Fe_2B$ phase. However, after boron pack refreshment, formation of the FeB phase occurs again due to the high boron concentration in the refreshed boriding media mixture.

A numerical study is carried out to understand the different growth kinetics for the borided samples with and without boron pack refreshment. The growth of FeB and $Fe_2B$ layers vs. d$t$ occurs when the simultaneous "consumption" of $Fe_2B$ and Fe at FeB/ $Fe_2B$ and $Fe_2B$/Fe interfaces respectively. Thus, the B should be conserved at the phase interface.

$$\left(C_{\text{low}}^{\text{FeB}} - C_{\text{up}}^{\text{Fe}_2\text{B}}\right)\mathrm{d}u = \left[-D_1\left(\frac{\partial C}{\partial x}\right)_{u-0} + D_2\left(\frac{\partial C}{\partial x}\right)_{u+0}\right]\mathrm{d}t \tag{1}$$

$$C_{\text{low}}^{\text{Fe}_2\text{B}}\mathrm{d}v = \left[-D_2\left(\frac{\partial C}{\partial x}\right)_{v-0}\right]\mathrm{d}t \tag{2}$$

where $u$ and $v$ are the location of Fe/ $Fe_2B$ and $Fe_2B$/Fe interfaces, respectively. Since only Fe phase is present before boriding, the initial condition can be written as:

$$C_{Fe_2B} = 0; \quad C_{FeB} = 0 \quad \text{at } x = 0 \text{ and } t > 0 \tag{3}$$

Assuming that $C_{up}^{FeB}$, $C_{low}^{FeB}$, $C_{up}^{Fe_2B}$ and $C_{low}^{Fe_2B}$ are the upper and lower limit of boron concentration in FeB and $Fe_2B$ phases respectively, the boundary conditions are:

$$\left.\begin{array}{ll} C = C_{up}^{FeB} & \text{for } C_{ads}^{B} > 0.5 \\ C = C_{low}^{FeB} & \text{for } C_{ads}^{B} < 0.5 \text{ and with FeB phase} \\ C = C_{up}^{Fe_2B} & \text{for } 0.33 < C_{ads}^{B} < 0.5 \text{ and without FeB phase} \\ C = C_{low}^{Fe_2B} & \text{for } C_{ads}^{B} < 0.33 \text{ and without FeB phase} \end{array}\right\} \text{at } x = 0 \tag{4}$$

$$C = C_{low}^{FeB} \quad \text{at } x = u - 0 \tag{5}$$

$$C = C_{up}^{Fe_2B} \quad \text{at } x = u + 0 \tag{6}$$

$$C = C_{low}^{Fe_2B} \quad \text{at } x = v - 0 \tag{7}$$

$$C = 0 \quad \text{at } x = v + 0 \tag{8}$$

The results in Fig. 6 simulate the growth behavior of the samples with boron pack refreshment after 240 minutes once-through boriding. The detailed derivative and assumptions for the mathematic model can be found in our previous study [13]. The simulation results are in good agreement with the experimental results, indicating that the proposed boriding mechanisms are reasonable in this study case. Different growth behaviors for the FeB and $Fe_2B$ phases can be found from the simulation results in Fig. 6b and 6c. The growth of FeB phase increase from 0 μm for sample without refreshment after 240 minutes once-through boriding to 15 μm for 60 minutes refreshment, satisfying the parabolic growth pattern. However, the thickness of the $Fe_2B$ phase continuously decreases during the first 20 minutes refreshment and then grows almost linearly with the further elongation of the boriding duration. It should also be noted that the total boride layer thickness (FeB+$Fe_2B$) almost has no changes during the first 10 minutes of refreshment, meaning that the formation of the FeB phase is the result of consuming the $Fe_2B$ phase.

Fig. 7 shows the normalized boron atom concentration profile along the FeB and $Fe_2B$ phases as a function of refreshment duration for the sample after 240 minutes once-through boriding. The normalized bon concentrations refer to the boron concentration in FeB and $Fe_2B$ phases with the up limit $C_{up}^{FeB}$ and $C_{up}^{Fe_2B}$ as the maxima and the lower limit $C_{low}^{FeB}$ and $C_{low}^{Fe_2B}$ as the minima in FeB and $Fe_2B$ phases. And the normalized boride

layer thickness is the relative boride layer thickness with that for the sample after 240 min once-through boriding as 1.0. The concentration profiles show that it costs about 10 minutes for the boron atoms diffusing from FeB/$Fe_2B$ interface to the $Fe_2B$/Fe interface. The abnormal growth behavior of the boride layer with boron pack refreshment is due to the non-steady-state diffusion of the boron atoms through the boride layers formed during once-through boriding. Generally, the effective boron concentration in the boriding media depletes exponentially with the boriding duration, and it will be below 0.5 after certain boriding duration in the boriding system, such as 80 minutes in our SPS boriding system, so that the FeB phase stops growing. For the samples borided with boron pack refreshment, the boriding media is refreshed periodically every 30 minutes so that the effective boron concentration is always high enough for the formation of the FeB phase.

Based on the above results and analysis, it can be concluded that the boron pack refreshment can maintain high effective boron concentration, resulting in much thicker boride layers than the samples borided without boron pack refreshment with the same total boriding duration. However, the boride growth of the samples with boron pack refreshment deviates from parabolic growth, which is due to the nonsteady-state diffusion of boron atoms within the boride layer (FeB or $Fe_2B$) after refreshment. The time required for the boron diffusion to reach steady-state condition depends on the thickness of the boride layers and the boron diffusion coefficient within the boride layer. If the steady-state diffusion is reached and the effective boron concentration in the boriding media is high enough, the parabolic growth of the boride layers could be expected.

## 5. Conclusions

The microstructure, phase composition and microhardness of the borided samples with and without boron pack refreshment in SPS boriding system were investigated in this study. The borided samples with boron pack refreshment were found to have much thicker boride layer than the samples without boron pack refreshment when the total boriding duration is longer than 60 minutes. The FeB phase is present in the borided layers for all of the samples with boron pack refreshment. However, for the samples without boron pack refreshment, the FeB phase gradually vanishes and almost disappears after 150 min once-through boriding duration. Modeling was carried out to understand the growth kinetics for the borided samples with and without boron pack refreshment. It

was found that the effective boron concentration in the boriding media plays important role on the once-through boriding, which depletes exponentially with the boriding duration, leading to insufficient boron concentration in the boriding media to form the FeB phase. For the samples with boron pack refreshment, the boron concentration in the boriding media is refreshed periodically to maintain high boron concentration for the formation of the FeB phase, which results in thicker boride layers. At the same time, non-steady-state diffusion of the boron atoms within the boride layers is involved, causing deviation of the boriding growth from parabolic growth.

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Table 1: Boriding parameters in this study

Table 2: Parameters for XRD analysis

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Fig. 2. Morphology of boride layer with boron pack refreshment sintered at 850$^{o}$C. (a) 30 mins×2; (b) 30 mins×3; (c) 30 mins×4; (d) 30 mins×5.

Fig. 3. Boride layer thickness at different boriding durations with and without boron pack refreshment.

Fig. 4 Layer microhardness from surface down (a) Set A in Table 1; (b) Set B in Table 1.

Fig. 5 Phase change of the top sublayer of the boride layer after refreshment

Fig. 6 Comparison between the simulation and experimental results with the boron pack refreshment after 240 minutes once-through boriding. (a) thickness of FeB+$Fe_2B$; (b) thickness of $Fe_2B$; (c) thickness of FeB.

Fig. 7 Concentration profile of boron atoms within the FeB and $Fe_2B$ layers as a function of refreshment duration.

| Total boriding duration | Temperature | Boriding Duration | | |
|---|---|---|---|---|
| | | Set A | Set B | Set C |
| 30 min | 850°C | 30 min | - | - |
| 60 min | 850°C | 60 min | 30 min × 2 | - |
| 90 min | 850°C | 90 min | 30 min × 3 | - |
| 120 min | 850°C | 120 min | 30 min × 4 | - |
| 150 min | 850°C | 150 min | 30 min × 5 | - |
| 180 min | 850°C | - | 30 min × 6 | - |
| 240 min | 850°C | 240 min | - | - |
| 270 min | 850°C | - | - | 240 mins + 30 mins |
| 300 min | 850°C | - | - | 240 mins + 60 mins |

Table 1

| Parameter | Setting |
|---|---|
| Anode | Cu |
| Generator voltage | 40 kV |
| Tube current | 30 mA |
| Alpha 1 & 2 wavelength | 1.54056 Å, 1.54439 Å |
| Intensity ratio | 0.5 |
| Divergence slit | Fixed, 2 |
| Receiving slit | 0.2 |
| $2\theta$ range | 20.0000 to 90.0000 |
| Scan step time (s) | 2.00 |
| Scan step size | 0.040 |
| Type of scan | Step |
| Monochromator used | Yes |

Table 2

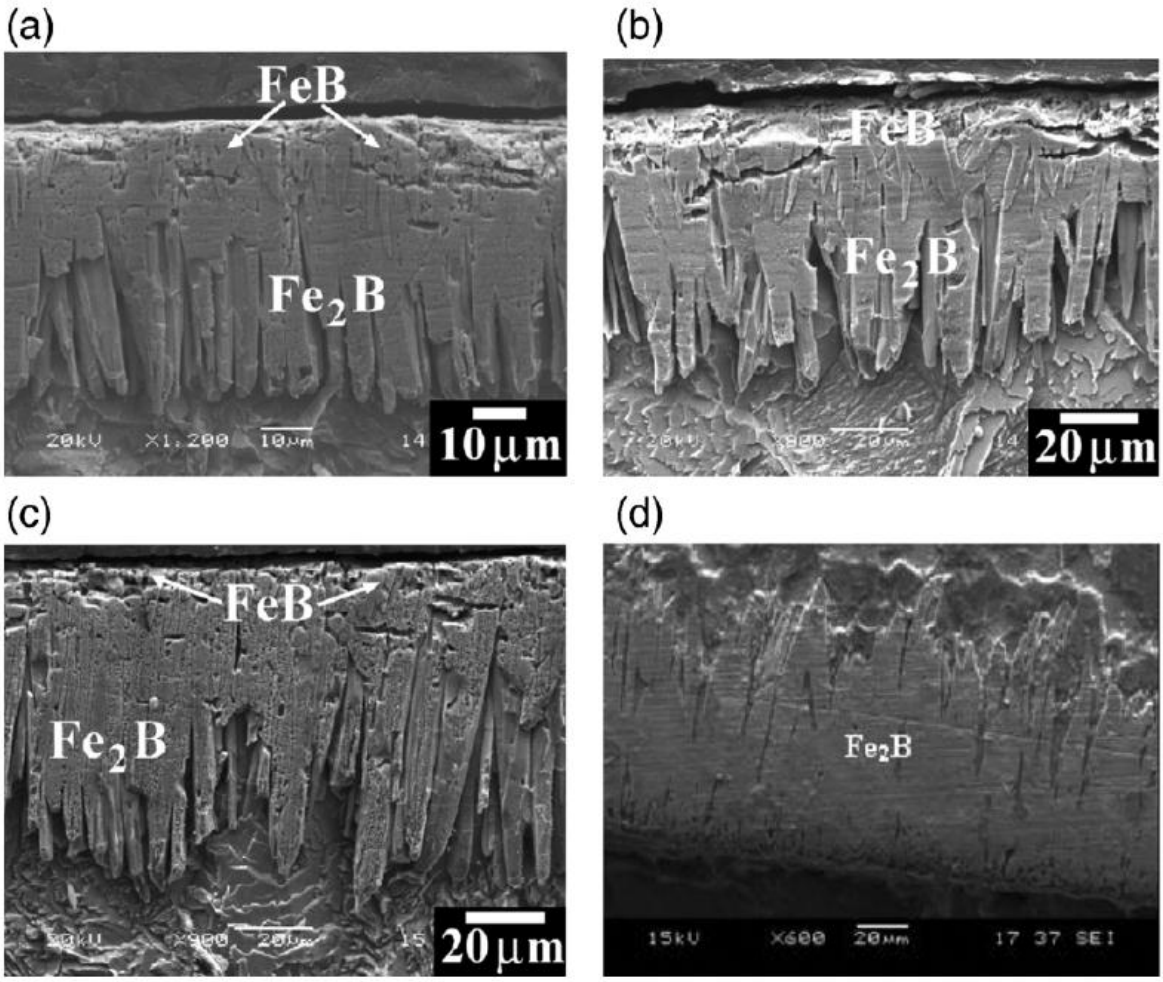

(a)
FeB
Fe2B
20kV X1,200 10μm 14
10 μm
(b)
FeB
Fe2B
20 μm
(c)
FeB
Fe2B
20 μm
(d)
Fe2B
15kV X600 20μm 17 37 SEI

Fig. 1

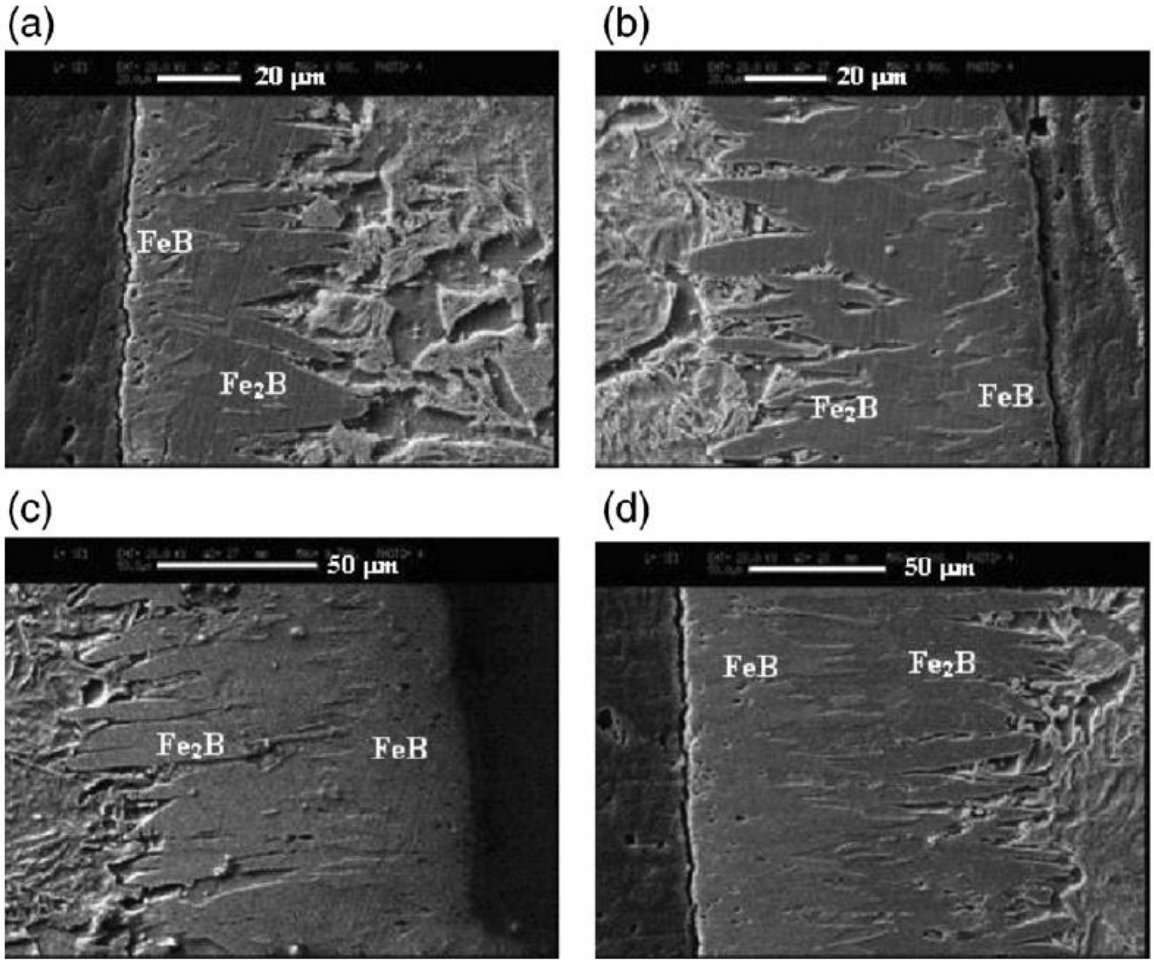



Fig. 2.

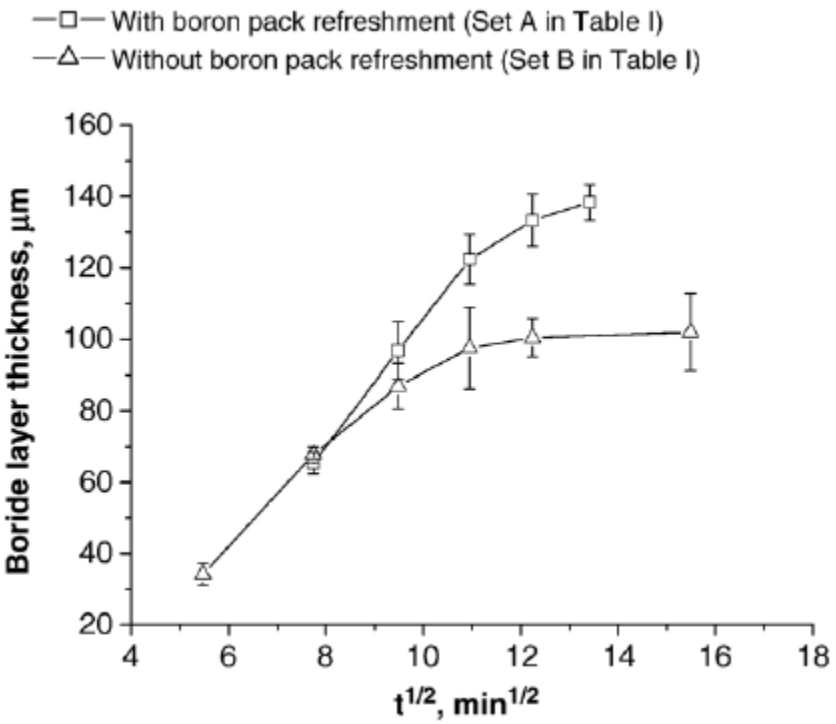
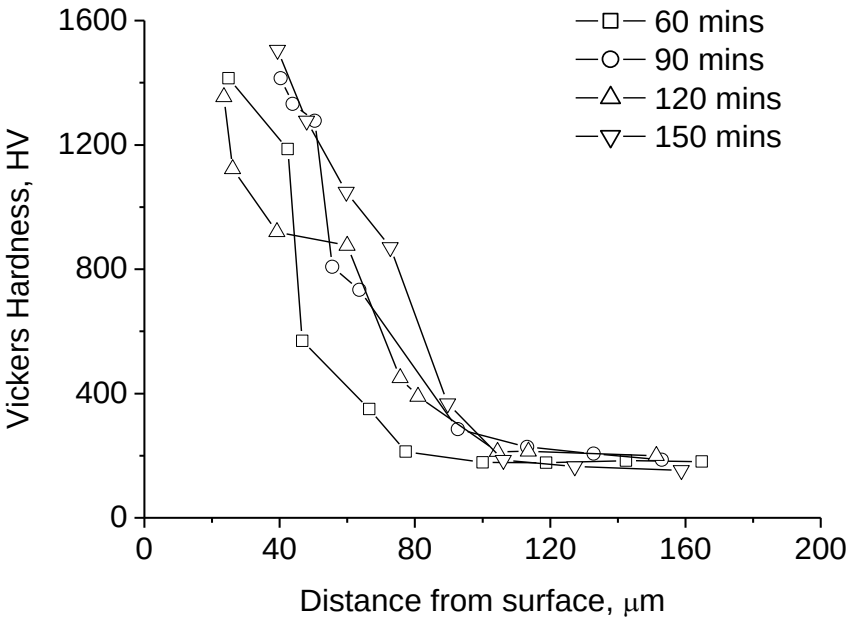
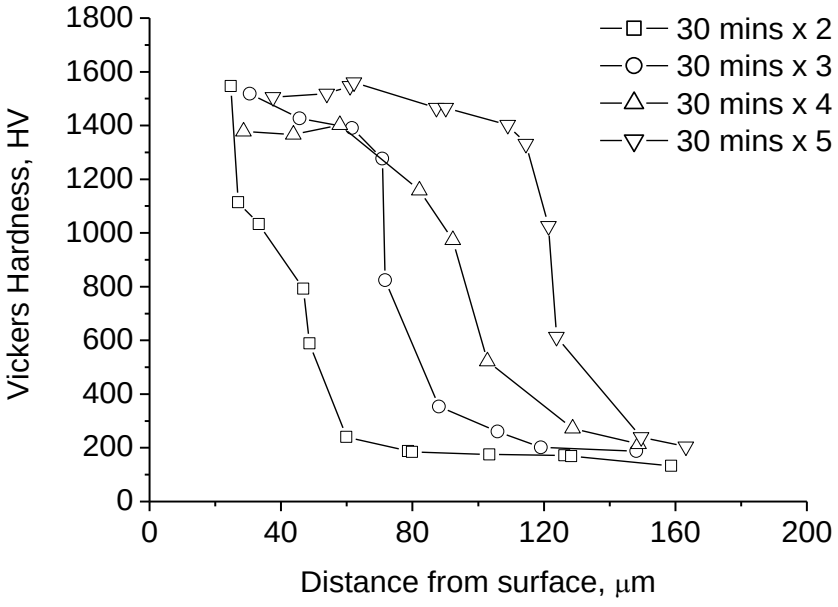

—□— With boron pack refreshment (Set A in Table I)
—△— Without boron pack refreshment (Set B in Table I)
Boride layer thickness, µm
160
140
120
100
80
60
40
20
4
6
8
10
12
14
16
18
t^1/2, min^1/2

Fig. 3.


60 mins
90 mins
120 mins
150 mins
Vickers Hardness, HV
Distance from surface, μm
0
400
800
1200
1600
0
40
80
120
160
200

(a)


30 mins x 2
30 mins x 3
30 mins x 4
30 mins x 5
Vickers Hardness, HV
Distance from surface, μm
0
200
400
600
800
1000
1200
1400
1600
1800
0
40
80
120
160
200

(b)

Fig. 4

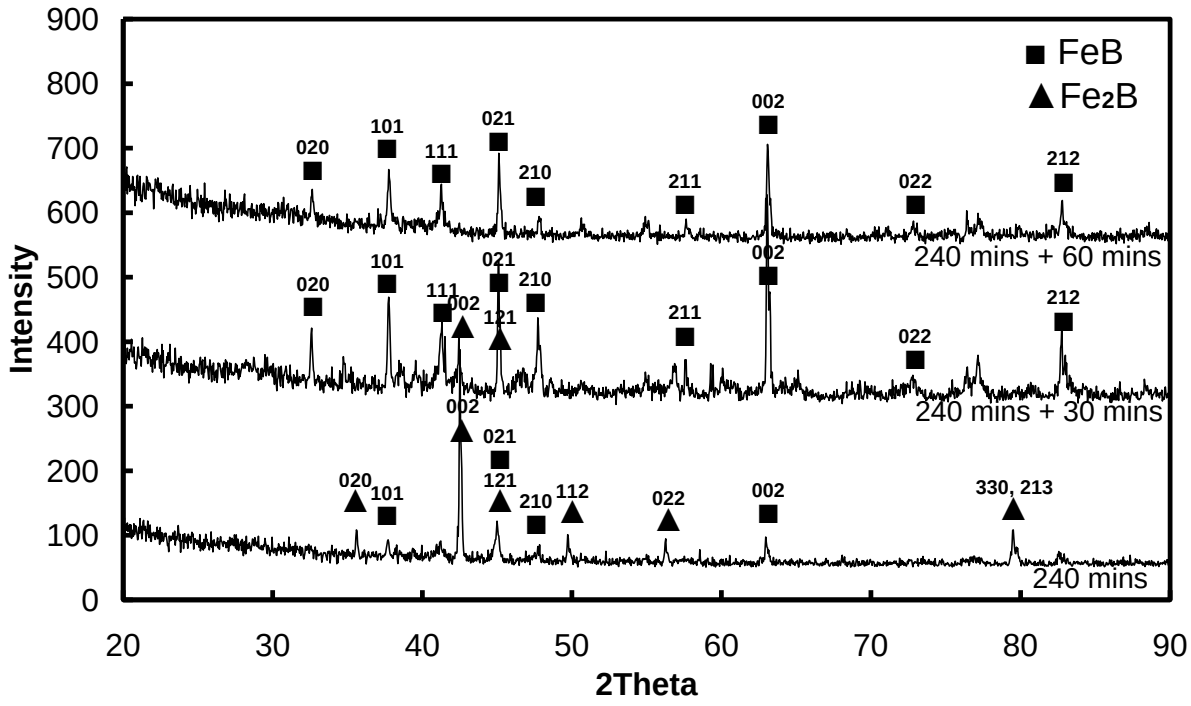
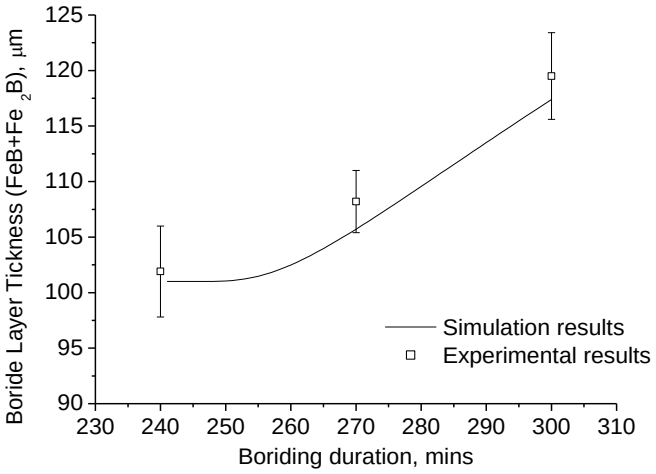
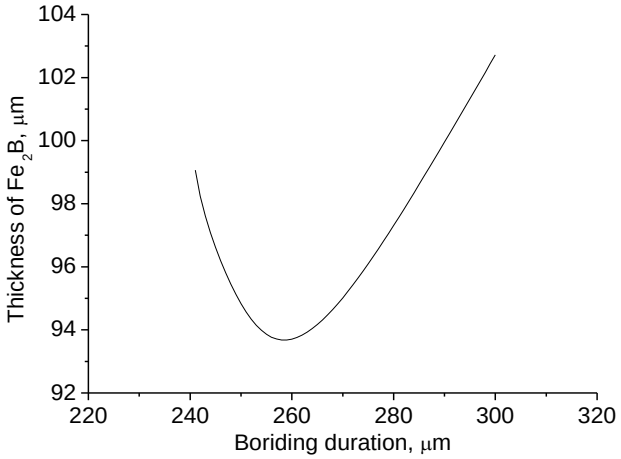

FeB
Fe2B
240 mins + 60 mins
240 mins + 30 mins
240 mins
Intensity
2Theta
020
101
111
021
210
211
002
022
212
121
112
330, 213

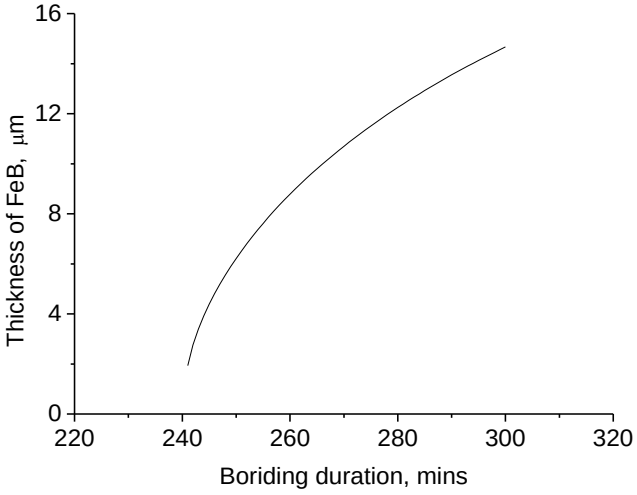
Fig. 5


Boride Layer Tickness (FeB+Fe 2B), μm
Boriding duration, mins
Simulation results
Experimental results

(a)

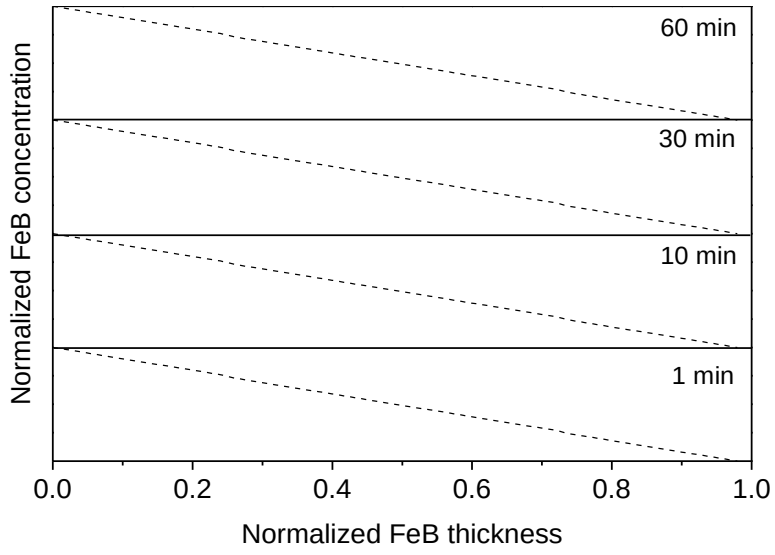

Thickness of Fe2B, μm
Boriding duration, μm

(b)

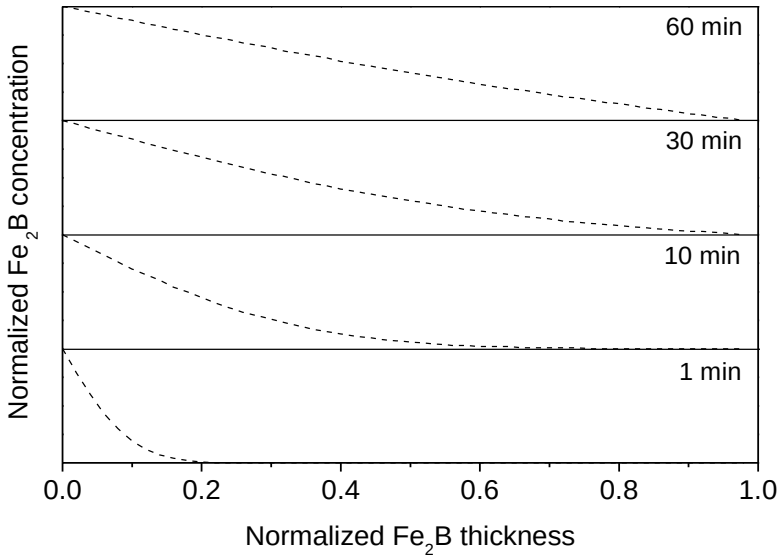

Thickness of FeB, μm
Boriding duration, mins

(c)

Fig. 6


60 min
30 min
10 min
1 min
Normalized FeB concentration
0.0
0.2
0.4
0.6
0.8
1.0
Normalized FeB thickness

(a)


60 min
30 min
10 min
1 min
Normalized Fe2B concentration
0.0
0.2
0.4
0.6
0.8
1.0
Normalized Fe2B thickness

(b)

Fig. 7