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## Effect of Diffusion Annealing on Borides Layers Produced on XC38 Steel

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In this work, we present a study of the effect of diffusion annealing at 700 °C for 1 h on the nature and properties of boride layers obtained on XC38 steel through a molten salt consisting of borax  $(Na_2B_4O_7)$  and boron carbide  $(B_4C)$ . We evaluated the changes brought by the diffusion annealing on the morphology of the boride layer, the thickness of this layer, the distribution of elements in the steel, and the hardness. Comparing the results obtained allowed concluding that the diffusion annealing will completely transform the two-phase layer formed of FeB and Fe<sub>2</sub>B borides in a single-phase layer consisting of single boride Fe<sub>2</sub>B. The transformation of the two-phase boride into a single-phase boride is done with an increase in thickness of about 30% compared to the initial thickness of the sample. The values of Si concentration obtained in the underlying zone after the diffusion annealing treatment are more important than those obtained in the same underlying zone of samples borided directly by immersion in molten salt consisting of borax and silicon carbide (SiC).

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### 1. Introduction

Compared to conventional surface treatments such as carburizing, nitriding, or carbonitriding, boriding treatment leads to surface hardness much higher than those obtained by conventional treatments. Indeed, the hardness of borides obtained on steels is generally higher than 1600 HV and it remains at high temperature, while the hardness obtained on case-hardened steel, nitrided or carbonitrided does not exceed 1000 HV in the best conditions [1]. Thus, the boride layers have excellent resistance to abrasive wear [2].

The main advantage of boriding treatment applied to steels is that it allows combining a high surface hardness at a low friction coefficient, which promotes good wear resistance. Similarly, borided layers have a longer lifetime service and good performance under oxidizing and corrosive atmospheres.

Like all thermochemical surface treatments, boriding can be achieved by various methods and techniques:

- in a solid medium consisting of powders or paste [3, 4];
- in a liquid medium molten salt with or without electrolysis [5–7],
- or, finally, in a gaseous medium [8, 9].

Each technique or method has advantages and disadvantages.

The diffusion annealing is performed on borided steels (in most cases) to obtain a single-boride (Fe<sub>2</sub>B) coating and to improve the mechanical properties at the surface of steels. Recently, two methods consisting of boriding followed by a diffusion annealing process (DAP) have been proposed to allow the phase transformation from the FeB coating to the Fe2B coating [9, 10]. A number of works for different purposes were also performed on different types of steel [11–13].

In this work, we have achieved an annealing diffusion treatment at 700 [°C] for two hours in objective to convert the dual-phase boride layer (FeB+Fe<sub>2</sub>B) on XC38 steel obtained by boriding in liquid medium consisting of borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) added to boron carbide (B<sub>4</sub>C), to a single-phase boride layer consisting of only Fe<sub>2</sub>B boride. Morphology, thickness and mechanical characteristics of this layer of single phase obtained after annealing diffusion will be compared with those measured on a single phase boride layer formed directly from a boriding treatment in a bath consisting of borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) and silicon carbide (SiC).

#### 2. Experimental procedures

The chemical composition of XC38 steel samples selected for boriding treatment determined by spectrometric analysis is given in Table I.

TABLE I
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Chemical composition [wt%] of the XC38 steel.

С	Mn	Si	Р	S	Cu	Cr	Ni	Fe
0.39	0.68	0.34	0.026	0.025	0.18	0.19	0.26	bal.

Before boriding treatment, all samples have undergone a surface preparation with silicon carbide paper up to grade 1000 to facilitate the boron diffusion through the surface.

The boriding treatment was carried out in molten salts consisting of 70% borax, completed with 30% of a reducing agent. The reducing agents used in this work are

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boron carbide  $(B_4C)$  and silicon carbide (SiC). Treatments of 4 h at 900 °C were applied in order to have a thick layer. After boriding, all samples are cooled in air. The annealing diffusion treatment is performed at 720 °C for one hour.

The identification of borided layers was made by X-ray diffraction using the  $K_{\alpha 1}$  of cobalt. The samples' chemical microanalysis of elements before and after boriding was determined using electron microprobe. Microhardness of boride layers and the underlying zone were determined with loads of 25 and 50 g, while that of the substrate was done with a load of 1 kg.

#### 3. Results and discussions

#### 3.1. Boriding treatment

The boriding treatment in molten salts consisting of 70% borax, completed with 30% of  $B_4C$  as reducing agent has yielded on XC38 steel surface a dual-phase borided layer of about 180  $\mu$ m thickness. In Fig. 1a, we can easily distinguish the FeB boride of Fe<sub>2</sub>B boride by its darker color, and in Fig. 1b we can note the presence representative peaks of both borides FeB and Fe<sub>2</sub>B on the diffractogram.



Fig. 1. Microstructure (a) and diffractogram (b) of boride layers on XC38 steel before diffusion annealing treatment with dual phase borides.

#### 3.2. Diffusion annealing treatment

The diffusion annealing of previously produced dualphase borided layer should be performed in order to form the single-phase boride layer. Holding at 700 °C for one hour led to the complete transformation of the dual-phase borided layer consisting of FeB and Fe<sub>2</sub>B borides into a single-phase borided layer consisting only of Fe<sub>2</sub>B boride.

In Fig. 2a and b, the total absence of FeB boride (dark zones) in the surface layer formed on XC38 steel can easily be seen after diffusion annealing of one hour at 700 °C. In the diffractogram, in Fig. 2c, one can note the total absence of the representative peaks of the FeB boride.

During annealing diffusion process, carried out in furnace at ambient atmosphere, the transformations take place at the interfaces as follows:

at the interface  $FeB/Fe_2B$ :  $2FeB \rightarrow Fe_2B + B$ ; at the interface  $Fe_2B/substrate$ :  $B + 2Fe \rightarrow Fe_2B$ .



Fig. 2. Microstructures and diffractogram of boride layer on XC38 steel after diffusion annealing treatment.

# 3.3. Effect of diffusion annealing on Si distribution in XC38 steel

In Table II, the distribution of Si in XC38 steel after boriding and diffusion annealing treatment is given.

TABLE II

Distribution of Si in XC38 steel directly after boriding in 70%Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> + 30%SiC (A), before (B) and after (C) diffusion annealing treatment.

	Silicium [wt%]				
	А	В	С		
FeB	-	0.05	-		
$Fe_2B$	0.03	0.02	0.01		
underlying zone	0.58	0.49	0.96		
substrate	0.31	0.31	0.20		

According to values of Si distribution in Table II, we can say that the diffusion annealing instead of homogenizing the Si distribution in the different zones has accentuated its concentration in the underlying zone. The presence of high concentration of Si under the borided layer can be harmful because of the formation of a very soft zone under the very hard borided layer.

#### 4. Conclusion

Towards the end of this work, we can advance the following conclusions:

- 1. The annealing diffusion at 700 °C for one hour is sufficient to transform completely the dual-phase borided layer in a single-phase borided layer consisting only in the Fe<sub>2</sub>B boride.
- 2. The transformation of the dual-phase borided layer in a single-phase borided layer is accompanied by an increase in thickness of about 20% compared to the initial thickness.

3. The annealing diffusion caused a significant segregation of silicon in the area underlying the  $Fe_2B$  boride.

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