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# Obtaining and Study of the Properties of the Single-Phase Boride Layer on Different Steels

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#### Abstract

The process for obtaining saturated mediums with powder of the boron oxide basis, of amorphous boron and boron carbide, witch is show, likely, to guarantee the formation of singlephase boride layer in the temperature range (700-1100) °C was proposed. The formation of layer was studied on samples of carbon steels, alloyed steel and of iron Armco. The results obtained showed that the chemical composition of phases in the developed layers is determined by the nature the saturated medium and not by the intensive concentration of boron in the medium. For the same conditions of saturation it is observed that the thickness of the layer increases with the reduction of the carbon content of steel. The mechanical characteristics, the corrosion resistance and the qualitative characteristics of the texture of the single-phase layer were also studied.

**Key words:** Thickness of boride layer, micro hardness, brittleness, pole density, powder, saturating medium, scattering angle, single-phase layer, texture perfection.

#### I. Introduction

Numerous previous works have been reported about the materials with skin boride treatment. Notably, it has been shown that the difficult for obtaining the boride single phase layers with the known mixtures powders was due to the high sensibility of saturation process with temperature and time in one hand, and to the grain characteristics of main constituents in those mixtures as well as to their chemical composition, in the other hand [1-3]. Elsewhere, it has been established that in some cases, boride single phase layers exhibited better working strength than that of the boride diphase layers [4, 5].

In the results previously reported, the use of the iron and nickel borides for iron Armco ( $\approx 99.85$ %) saturation has been mentioned. In these works, it has been shown the chemical composition of the phases in the boride layer depended mainly on the nature of the compound containing boron and not on the mass fraction of boron in the saturated medium [6].

The present paper purposes a new procedure for working out saturated media with compounds such as boron oxide, boron carbide and amorphous boron in order to develop a boride single phase layer. Our method has taken into account:

- thermodynamic equilibrium principle, which can be applied in physicochemical systems case;- phase equilibrium between Fe<sub>2</sub>B and saturated medium containing boron at boride treatment temperatures;

- the tendency towards a uniformity of chemical potential of boron in saturated medium, in the skin zone of the sample being treated as well as in the developing boride layer.

### **II.** Materials and Technical Experimental

The materials used in this investigation are C42 steel, the carbon steel used for mechanical engineering, C80 steel, the carbon steel used for tool making and the X160CrMoV13-8, the steel used for cold forming stamp, and the iron Armco foil of 100  $\mu$ m thickness.

For saturation diffusion process, investigations were bearing upon the mixtures resulted from aluminothermy process. As boron source, the boron anhydride was used, as repeaterregenerator, the aluminum and as ballast additive, the aluminum oxide.

For stabilizing  $Fe_2B$  phase, powders from transition metals group, Fe, Ni, and Cr were used. For the mixtures enhancer, the potassium terafluoboride KBF<sub>4</sub> was used

Boride treatment was carried out in shaft furnaces adjusted at (800–1000) °C for from 2 h to up 8 h, samples being sealed in refractory steel capsules.

The structure and chemical composition of the phases have been investigated using Microscopy and radiocrystallography analysis.

The resistance to corrosion of borides was determinate using gravimetric and electrochemical techniques.

The reference electrode potential was + 0.02 V. Values of all potentials were determinate relatively to hydrogen electrode.

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Wear resistance has been investigated in dry slipping friction conditions on an equipment especially designed for this purpose, according to disc-disc, socket-shaft and surface-socket techniques.

For determining wear rate, relative intensity of wear has been used, which equals to the ratio of linear wear value to the friction distance.

### III. Results and discussion

It has been found that for obtaining a boride single phase layer in iron-carbon alloys case the iron mono boride FeB can be used as a saturate medium (according to the Fe-B phases equilibrium diagram) or any other compound containing boron, which can

Table 1 – Results obtained from boriding processes of C80 steel

be in equilibrium with the iron hemi boride  $F_2B$ , at boride treatment temperatures. As examples, compounds Ni<sub>2</sub>B, MoB and WB could be given.

The appropriateness of such an approach – while working out saturated environments for boride single phase development – was confirmed by the investigation made in the iron Armco foils case. In fact, the use of these foils enables to eliminate mass transfer phenomenon during needle-shaped boride coalescence.

The chemical composition of the saturating mixture used and the results obtained from saturation process on the C80 steel are given in table 1

N⁰	Chemical Composition of medium of	Ratio	Thickness of layers (µm)	
	saturated medium (%)	B <sub>2</sub> O <sub>3</sub> /Fe	Fe <sub>2</sub> B	FeB
 1	39B <sub>2</sub> O <sub>3</sub> +30Al+30Al <sub>2</sub> O <sub>3</sub>	- 10	120	110
2	36B <sub>2</sub> O <sub>3</sub> +29Al+30Al <sub>2</sub> O <sub>3</sub> +5Fe	7.3	60	140
3	25B <sub>2</sub> O <sub>3</sub> +20Al+30Al <sub>2</sub> O <sub>3</sub> +25Fe	1.0	125	75
4	21B <sub>2</sub> O <sub>3</sub> +29A1+30A1 <sub>2</sub> O <sub>3</sub> +30Fe	0,7	150	
5	18B <sub>2</sub> O <sub>3</sub> +11+30Al <sub>2</sub> O <sub>3</sub> +35Fe	0,6	75	-
6	$14B_2O_3 + 51 + 30Al_2O_3 + 45Fe$	0,3	20	

In this table, it can be easily observed that the increase in iron content of the medium result in the diminution of the overall thickness of the diphase boride layer as well as that of the FeB phase.

For the ratio  $B_2O_3/Fe = 0.70$ , only the layer of the single-phase  $Fe_2B$  formation is observed On the figure 1, one can observe that the thickness of borides layer increases with increasing in the treatment temperature as well as in the duration of

saturation process, witch is confirmed by laws of the diffusion. It is obvious, in some that for the same conditions of saturation, the reduction of the carbon content of the steel leads to the increase in thickness of the layer of boride made up.

The microstructures of the single-phase layers of boride obtained on the steels C45, C80, and X160CrMoV13-8 are represented on figure 2. The boride layers exhibit a needle-shaped aspect.



(a) (b) (c) Figure 1- Influence of the temperature and the duration of saturation on the thickness of borides layers produced on different steels from the Mixture of  $(11\% \text{ Al} + 18\% \text{ B}_2\text{O}_3 + 35\% \text{ Fe} + 30\% \text{ Al}_2\text{O}_3) +$ 

 $1 \% \text{ KBF}_4 + 2 \% \text{ AlF}_3$ ).

(1) 
$$t = 2 h$$
; (2)  $t = 4 h$ ; (3)  $t = 6 h$ ; (4)  $t = 8 h$ .

(a) C45; (b) C80; (c) X160CrMoV13-8

With the increase in the carbon concentration in steel the needles of boride phase in

formation are accentuated with the ends swelled. In X160CrMoV13-8 steel under the boride layer of  $Fe_2B$  combined, one meets boron carbides particles.

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(a) Figure 2 - Microstructures of a single-phase layer produced on different steels from the Mixture of (11% Al +  $18 \% B_2O_3 + 35 \% Fe + 30 \% Al_2O_3) + 1 \% KBF_4 + 2 \% AlF_3).$ (1) t = 2 h; (2) t = 4 h; (3) t = 6 h; (4) t = 8 h.

(*a*) C45; (*b*) C80; (*c*) X160CrMoV13-8

Figure 3 shows the distribution of the elements entering in the composition of steels and saturating mixtures, following the thickness of the layer of borides, for C45 and X160CrMoV13-8 steels. During the saturation of C45, from a mixture containing Cr, one obtains a weak alloy  $Fe_2B$  boride with chromium (fig.3a). It is well known, that the allying elements of steels, do not

give clean borides, but take part rather in the development of iron boride alloy. According to the character of the interaction between the elements of alloy and the boride  $Fe_2B$ , their redistribution occurs during the process of constitution of the boride layer.



Figure 3 - Distribution of elements in single-phase boride layer in the conditions of saturation T = 950 °C, t = 4 h for two steels.

(a) - steel X160CrMoV13-8, in the saturated medium of boron oxide and iron oxide.

(b) C45, in the saturated medium of iron and chrome.

The results of the analysis carried out on steel X160CrMoV13-8 (fig.3b) show a carbide zone under the allied layer of chromium molybdenum

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boride. They are present also in the layer itself. The zone of the solid boron solution in iron reaches a few millimetres and contains (0.002-0.005) % of boron.

One observes a tendency to the determination of optimum thickness of layers according to the conditions of operating. For the parts requested under the conditions of slip, it is sufficient, in theory, to have a fine thickness of layer of boride from (20 to 50)  $\mu$ m. For a heavy wear conditions by abrasion, it is necessary to have a thickness of layer equalizes at least to 250  $\mu$ m. The powder saturating media we have developed for this work were shown to have a very strong capacity of saturation. For example, in the case of the C80 steel, the thickness of the boride single-phase layer can reach 200  $\mu$ m.

Our investigations showed that the hardness of the boride single-phase layer decreased with the increase in the carbon content of steel. The micro hardness of the Fe<sub>2</sub>B phase in the case of the steels used for tools proved to be lower than that of the same phase on iron Armco, and was seen to vary in the range (10 000–11 000) MPa.

In addition, the micro brittleness of the boride layers was found to decrease with the increase in the steel carbon content.

By contrast, the micro brittleness of the layer increases with the increase in hardness. The presence of a significant quantity of chromium in the X160CrMoV13-8 steel was shown result in the development of more brittle single phase boride layer, compared to that obtained in the case of the carbon steels (fig. 4).

The study of the influence of the saturation conditions on the brittleness of the single-phase  $Fe_2B$  layer did not establish a clear tendency.





However, it should be mentioned that, for obtaining required thickness of layer, the prevalence is conferred on the high temperature with a minimal term of treatment.

The tendency of the layer of boride to shearing for various rates of plastic deformation was studied. It rises from the results obtained that the single-phase layers of boride exhibit better shear strength than the diphase layers. In general, shearing was found to occur at boundary area of the  $F_2B$  phase.

The study of the corrosion resistance of layers of single-phase and diphase of boride in the case of C80 steel revealed that the layers of boride significant anti-corrosion have properties, respectively in the salt NaCl and in the hydrochloric and sulphuric acid solutions also. Stationary measurements of potentials of electrodes revealed that the potentials of boride layers in the solutions made up from (1-30) % of HCl and H<sub>2</sub>SO<sub>4</sub> were found to be higher than those of C80 untreated steel and these potentials were seen to increase with the increase in the time of maintaining. The increase in the potential confirms the reinforcement of the protection capacity of films made up of products of corrosion, which is checked by the data of analysis by gravimetric techniques.

However, in the case of nitric acid, boride layers afford low corrosion resistance. The corrosion resistance of the single-phase layers of boride in the solutions of HCl was seen to exceed from (2 to 8) times that of the diphase layers: 8, 5, 4, 2.5 and 2 times in the solutions of 1 %, 5 %, 10 %, 20 % and 30 %, respectively. The ennoblement of potential in the solutions above mentioned begins after 130 h of test for the boride single-phase layer while in the case of the boride diphase layer it appears only in the solution of 30 %.

The study carried out reveals that the wear resistance of the layers of boride, under the conditions of dry sliding friction, is determined mainly by the diagram of contact and the characteristic of the relative displacement of the samples one upon another and, also, by the hardness of surface and the brittleness of the layers obtained by diffusion. Wear resistance of steel couple treated with boron, studied by the diagram disc - disc proves to be lower by (2.3-3.1)times than that of thermally treated couple 42 Cr4 steel and 100Cr6 bearing steel and that, respectively for the single-phase and diphase layers. In the case of the contact by sleeve-shaft diagram, the value of layers wear resistance is controlled by the surface hardness, essentially; this is why it is 6.8 times more significant in the boride diphase layers than a C80 steel having undergone low hardening then aged, and 1.5 times only compared to the single phase layers. The wear resistance of the couple for surface-sleeve diagram is determined primarily by

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the brittleness of the coating. This is why, that contact diagram is more convenient in the case of the boride single phase layers.

### IV. Conclusion

The boride single phase layer formation depends not only on the boron quantity in the mixture but equally on the chemical composition of the compounds present in the mixture.

For obtaining a boride single phase layer in the case of carbon steels and iron Armco one can use as saturated medium the iron mono boride FeB or another compound containing boron and being in equilibrium with the iron boride Fe<sub>2</sub>B such as FeB,  $Ni_2B$ , MoB and WB. The presence of these compounds in the powder medium was found to create the favourable conditions to the boride single phase layer development in the case of steels investigated.

It was established that the composition of the boride phase layer is determined essentially by the nature of the compound containing boron and not by the mass fraction of boron in the saturated medium.

Hardness and micro brittleness of the single-phase layer of boron decrease with the increase in the carbon content of the steel. The correlation between the conditions of saturation and the brittleness of the boride layer could not be established.

The corrosion resistance in the hydrochloric acid solution of single-phase layer of boride exceeds (2-8) times that of the boride two-phase layer. The ennoblement of potential solutions used begins after 130 h of test for the boride single-phase layer while in the case of the boride two-phase layer it appears only in the solution of 30 %.

The wear resistance depends on the hardness and the brittleness of the boride layers. The study, carried out according to various diagrams of contact, showed that the coatings with the single-phase layers have advantages if the resistance is determined initially by the brittleness of the layer.

The quantitative characteristics of texture [001] Fe<sub>2</sub>B were studied. With the increase in the temperature and time of saturation the perfection of texture decreases. The angle of dispersion compared to the axis of texture varies slightly and is in the range of  $22^{\circ}$  to  $28^{\circ}$ . The tendency to the variation of the oriented crystallite rate according to the conditions of saturation is a function of the polar density. The oriented crystallites rate was found to vary between 5 % and 40 %. The linear correlation between the principal properties of the single-phase layers and the quantitative characteristics of texture was not established.

The study of the coating is recommended for tools for hot and cold stamping, for the shells and for parts, which work under the conditions of alternative load, and in the case of abrasion resistance.

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