# STRUCTURE AND CHARACTERISTICS COATINGS OBTAINED AFTER COMPLEX SATURATION BORON AND SILICON ON CARBON STEEL

Prof. Dr. Chernega S., Poliakov I., Krasovsky M., Medova I.
National Technical University of Ukraine "Kiev Polytechnic Institute", Department of metal science and heat treatment,
Ukraine, Kiev, st. Polytechnic, Bldg. 9, tel.: +38 (066) 990 - 90 - 35
e-mail: smchernega@mail.ru; polykov_igor@mail.ru

*The results of research coatings coatings obtained after complex saturation boron and silicon in the powder environment on steel 45. Determine the thickness and microhardness of the resulting coatings, their phase and chemical composition. It has been established that the coatings obtained after complex saturation boron and silicon more plastic and have a 1.8 times higher fracture toughness ($K_{1c}$) and 1.5 times higher than the wear resistance as compared with coatings obtained by borating.*

***Keywords:*** *BORON CARBIDE, BORIDING, BORIDE LAYER, SILICON, MICROSTRUCTURE, MICROHARDNESS, WEAR RESISTANCE, CRACK RESISTANCE.*

## 1. Introduction

Operational properties and durability of each mechanism is largely determined by the surface state of the material layers in which high stresses are concentrated and which are responsible for such properties as corrosion resistance, heat resistance and wear resistance of the article or a mechanism in general.

One of the main methods of increasing the life of machine parts and tools is chemical - heat treatment (CHT), which allows to give the surface of metals and alloys, high hardness and wear resistance, corrosion and erosion, increased heat resistance and resistance to radiation exposure. In many cases, CHT is the only possible means to solve a specific technical problem.

Improving the reliability and durability of machines and mechanisms depends largely on the durability of the surface components operating friction. The most common types of such CHT as carburizing, cyanidation, nitriding, which often can not meet the growing demands for durability work surfaces. In this regard, recently paid much attention to the development of new types of wear-resistant coatings.

Among the CHT relatively new process takes place special diffusion borating metals and alloys with the addition of other elements that can change the structure and phase composition of the boride layers and, consequently, to obtain a higher surface hardness and wear resistance as compared with other types of treatment [1 – 6].

In order to improve the wear resistance, heat resistance and corrosion resistance of metals and alloys used boriding with additions of silicon. As a result of diffusion saturation changes the structure of the surface of boride layers on steel and formed sublayer enriched saturating elements having a high performance properties.

Saturation boron and silicon allows to improve the surface characteristics, reduce the brittleness of the coating and ensures high durability and low production cost. This process can be used in metallurgical, chemical, aerospace, engineering and shipbuilding industries.

## 2. Materials and methods research

Saturation boron and silicon was performed powder method in a special container under reduced pressure at a temperature of 970 ºC for 4 hours. The study was conducted on samples of steel 20, 45, U8A.

Selection of the optimal temperature saturation process is an important factor at saturation boron and silicon. For example, at a temperature below 900 ºC, the saturation process, and slows considerably decreases the content of elements in the diffusion layer, and at temperatures above 1000 ºC, a layer with increased fragility, porosity and deep radial cracks which lead to a deterioration in performance properties. Therefore, the selected temperature is optimal for this type of treatment.

The samples, previously ground, polished and degreased in ethanol, and then charged into a stainless steel container and covered with a mixture of saturating. The distance between the sample and the walls of the container is not less than 5 mm. Saturation of boron and silicon steel conducted in mixtures containing boron carbide technical $B_4C$ and Fe-Si powder and Si. As the dopant used ftoroplast.

To separate the reaction space of the container from the furnace atmosphere and prevent the penetration of air, closed container and covered with a sheet of asbestos and filled with sodium silicate glass thickness of 10 mm. When heated to a temperature of boriding, at melted sodium silicate glass (750 – 800 ºC) and seal the container. Heating crucibles and following isothermal exposure was carried out in a laboratory furnace type SNOL – 1,6,2,5.1/11M.

At the end of the isothermal holding items fetched from the container from the oven and cooled to room temperature in air. After cooling container, silicate smashed and container unpacking and recovered items with a clean surface, which do not require further purification.

This method has the following advantages: simplicity of the process, allows the processing of products of various configurations, it is possible to obtain the diffusion layers of different thicknesses.

Visual inspection and microstructural studies were performed on boride coatings metallographic microscope Carl Zeiss increase in the range of 100 ... 1000.

Polished thin sections were performed on a polishing wheel grit diamond paste from 28 to 1 mm, which allows a number of high surface quality research.

As the reagent used for the chemical etching of 3 ... 5% solution of nitric acid in ethanol; Exposure – 30 sec.

Measuring the thickness of the diffusion layers and microhardness were carried out on a PMT-3 is not less than 10 – 15 fields of view at a load of 0.49 – 0.98 N. Microhardness measurement accuracy is ± 500 MPa.

The phase composition of the coatings was analyzed by X-ray diffractometer DRON 2.0 in copper $K\alpha_1$, $K\alpha_2$ monochromatic radiation, and chemical composition were determined on the analyzer «Camebax Sx50».

## 3. Results and discussion of research

The resulting diffusion layer at saturation technical boron carbide ($B_4C$) with addition of silicon powder unlike boride coatings obtained at saturation in $B_4C$ consisted of boride phases FeB, $Fe_2B$ and additionally detected graphite phase, which was confirmed by X-ray diffraction analysis (Fig. 1). Graphite phase first discovered when added to a mixture of saturating boriding silicon. When introducing the medium to saturate Te, Cr, V, Nb, Mo, W, or other elements of the graphite phase is not allocated [7].

Phase analysis was carried out with a sample surface boride coating, in resulting saturation boron and silicon, X-rays penetrate to a depth of 15 microns, set quantitative distribution of phases in the near-surface zone. It is found that the phase of FeB is 68.13 wt%, 28.36 wt% Fe2B, 3.51 wt% graphite. In conducting research on the same samples that obtained after boronization in the surface layer was found only FeB phase. X-ray diffraction studies

confirm the volume redistribution phases FeB and $Fe_2B$ in boride coating. At saturation in powder medium supplemented with silicon fixed volume reduction phase FeB.

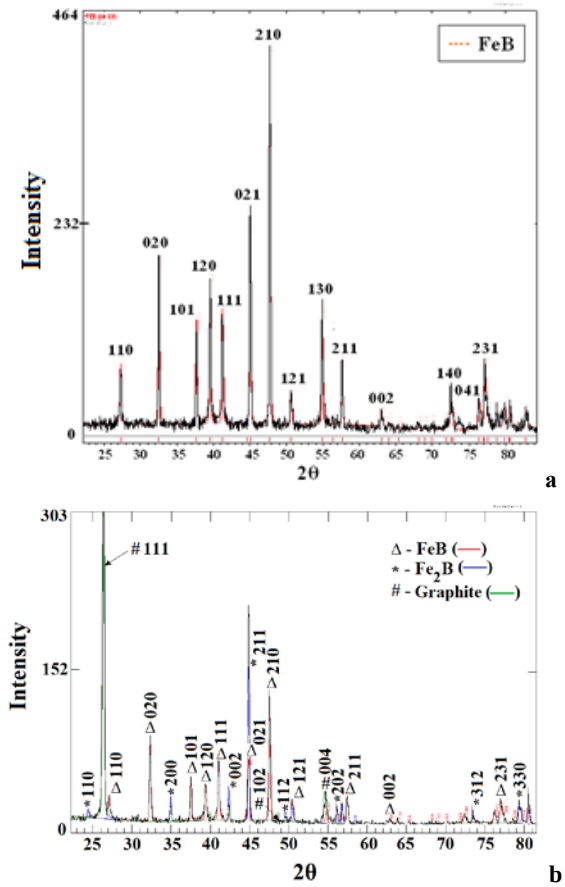


***Fig. 1:*** *Diffractograms taken from the surface of the steel 45 with boride coatings in Cu $K\alpha_1$, $K\alpha_2$ monochromatic radiation: a – coatings after boriding; b – coatings obtained after complex saturation boron and silicon, diffraction peaks of C (graphite) line (111) (102) (004)*

In table 1 shows the parameters of a crystallographic lattice boride FeB phase searches and studies the lattice constant changes FeB phase after introduction in to saturate the environment of silicon powder.

***Table 1:*** *The parameters of the crystal lattice phase after boriding and after complex saturation boron and silicon*

| Name of phase | The crystal lattice parameter, Å | | |
|---|---|---|---|
| | a | b | c |
| FeB (after complex saturation boron and silicon) | 4,0549 | 5,4899 | 2,9457 |
| $Fe_2B$ (after complex saturation boron and silicon) | 5,1021 | - | 4,2450 |
| Graphite (after complex saturation boron and silicon) | 2,6040 | - | 6,6963 |
| FeB (after boriding) | 4,0603 | 5,5023 | 2,9504 |
| Changing the crystal lattice parameters of the phase FeB after complex saturation boron and silicon | Δa= - 0,0054 | Δв= - 0,0124 | Δc= - 0,0047 |

From the data in table shows that there is a change of the crystal lattice periods FeB phase (fig. 2) that due to the formation of substitutional solid solutions (Fe, Si)B and $(Fe, Si)_2B$. Si has an atomic radius of 0.111 nm, the atomic radius Fe – 0,156 nm, B – 0,087 nm.

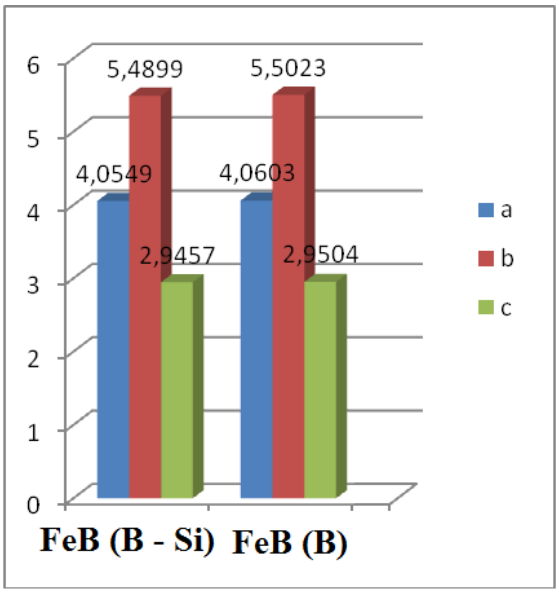


***Fig. 2:*** *Change the lattice parameters phases after boriding and after complex saturation boron and silicon*

Reduction unit periods of the crystal lattice due to saturation of boride phase elements with smaller atomic radius, contributes to the strength of the crystal lattice during the formation of substitutional solid solutions.

Reducing the volume of the crystal lattice leads to a change of the interatomic bonds in the crystal lattice, which promotes the growth of microhardness of boride phases doped Si. By doping Si lattice periods rhombic phase FeB decrease, the largest change is observed for parameter b.

Microstructure of boride layer is a needle borides, which are oriented perpendicular to the sample surface and wedged into the matrix and graphite inclusions, which we revealed (Fig. 3).

Acicular structure boride layers indicates that for the formation of boride phase growth occurs in a direction perpendicular to the sample surface, while along the surface development of these phases occurs slightly.

Graphite phase is concentrated in the surface and near-surface coating zone, accumulates in the pores to form dark zones alternating with light portions FeB phase (clearly seen in the structure of the cross-sections of boride coatings on Fig. 3). The size and number of pores which can be identified as dark component structure of the surface layer increases with increasing carbon content in the steel. So, for steel U8A (Fig.3, c), up to 40% of the surface occupied by pores, which may be graphite.

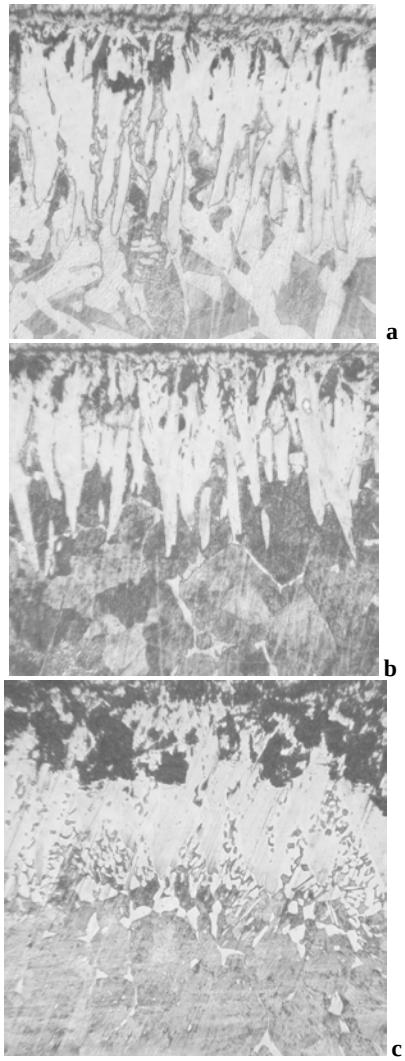


***Fig. 3:*** *The microstructure of the coatings on steels after complex saturation boron and silicon: a – 20, b – 45, c – U8A, x200*

To measure the fracture toughness of single crystals of solid crystalline materials used method Charles – Evans [8 – 11]. In this $K_{1C}$ determined by the length of radial cracks formed around the indentation of the Vickers indenter with a semi – empirical relation:

$$\mathbf{K_{1c}=0{,}015\cdot(E/H)^{1/2}\cdot P/C^{3/2}} \qquad (1)$$

or a graphic dependence between ($K_{1C}$-F/H)-(H/E-F) and c/a, where F – Marsha constant; H – Vickers hardness; a – half of the diagonal of the impression; c – length of radial cracks; E – Young's modulus.

Critical fracture stress was determined by the formula:

$$\mathbf{\sigma = K_{1c}/\ \alpha * L^{0,5}} \qquad (2)$$

where σ – critical stress fracture, L – transverse grain size of boride, α – geometric constant (π).

Calculation data cleaving stresses which may arise boride phases depending on the parameter $K_{1C}$ steel 45 shown in table 2.

***Table 2:*** *Calculation data stresses chipping boride coatings (phase FeB)*

| **Environment for saturation** | **$K_{1C}*m^{0,5}$** | **$\sigma_{cleaving}$ at L =** | |
|---|---|---|---|
| | | **15 microns** | **20 microns** |
| | | **MPa** | **MPa** |
| $B_4C$ + Si | 1,97 | 299,27 | 237,18 |
| $B_4C$ – Fe-Si | 1,94 | 289,07 | 229,93 |
| $B_4C$ | 1,10 | 218,69 | 189,53 |

The chemical composition of the coatings was determined by X-ray analysis, which was performed on a scanning electron microscope – SEM at 2000 times magnification, accuracy measurement is 0.01 wt.%. Chemical analysis performed by EDS, the calculation of the quantitative chemical composition – by the method of ZAF.

It has been established that silicon is uniformly distributed in a coating thickness of up to 0.8% by volume. In addition, there are areas in which the silicon contains up to 50% by volume.

## 4. Conclusions

Saturation boron and silicon provides a change of the phase composition of boride phases, namely the allocation of graphite phase, and a reduction in phase FeB 30% in the coating. Also the redistribution characteristics boride phases, namely increasing microhardness FeB phase to 20 – 22 GPa, $Fe_2B$ phase to 17 GPa and fracture toughness $K_{1C}$ increase 1.8 times and an increase in cleaving stress values of 1.3 – 1.4 times. Growth of porosity in the surface areas of boride coatings when the pores are filled with graphite enhances durability boride layers by reducing the friction coefficient. Increasing microhardness FeB phase, $Fe_2B$ after complex saturation boron and silicon compared microhardness after these phases boriding phase due to saturation of the silicon – (Fe, Si)B, $(Fe, Si)_2B$, which is confirmed by changing the periods a, b, c orthorhombic phase grating, wherein the lattice parameters decrease.

It was established that the carbon content of the steel affects the thickness of the diffusion layer. This is due to the fact that the carbon in the transition zone is displaced. Therefore, with the increase of carbon content in the base material, the coating thickness decreases.

## 5. Literature

1. Ляхович Л. С. Состояние и перспективы развития процесса борирования. – К.: «Наукова думка», 1972. – 194 с.

2. Алиев А. А., Булгаков В. П., Приходько Б. С. Диффузионное борирование стали и шероховатость поверхности // Вестник Астраханского государственного технического университета. – 2005. – № 2. – С. 91 – 95.

3. Кухарева Н.Г., Петрович С.Н., Галынская Н.А., Протасевич В.Ф., Смирнова Т.Н. Борирование углеродистых и легированных сталей в кипящем слое // Наука и Техника. – 2012. – № 5. – 11 – 17.

4. Самсонов Г. В., Серебрякова Т. И., Неронов В. А. Бориды. – М.: Автомиздат, 1975. – 376 с.

5. Похмурский В. И., Далисов В. Б., Голубец В. М. Повышение долговечности деталей машин с помощью диффузионных покрытий. – Киев.: Наукова думка, 1980. – 188 с.

6. Спиридонова І. М. Фазові перетворення в композиційних матеріалах з залізними зв'язками, що містять бор та вуглець / І. М. Спиридонова, О. В. Сухова, О. Г. Безрукава // До-повіді НАН України.–2002. – № 10. – С. 93 – 97.

7. Баландин Ю.А. Комплексное насыщение поверхности инструментальных сталей бором, медью, хромом в псевдоожиженом слое// Изв. Вуз. Черная металлургия. – 2005. – №5. – 50с.

8. Чернега С.М. Комплексное насыщение углеродистых сталей бором и хромом в активированной среде// Изв. Вуз. Черная металлургия. – 1999. – №1. – 58с.

9. Нарва В.К. Полеченко А.В. Нанесение покрытий карбидосталей на стальную основу// Изв. Вуз. Черная металургия. – 1995. – №1. – 47с.

10. Ирвин Дж., Перле П. Основы теории роста трещины и разрушения // Разрушение. – М.: Мир, 1976. – Т.3. – С.17 – 66.

11. Evans A. G., Charles E. A.. Fracture toughness determination by indentation. – J. Amer. Ceram. Soc., 1976, v. 59, N 7 – 8, p. 371 - 372.