Structure and Properties of Boride Layers Produced by Electron Beam in Vacuum

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Abstract – Conditions of formation, structure and properties of boride iron layers on carbonaceous steel 45 at electron beam borating are investigated. New process to make layers of iron borides (Fe_2B, FeB) using electron beam are reported. The microstructure and microhardness of boride layers are inves-tigated and also are compared to layer properties obtained at solid phase borating. Formed layers were heterogeneous structure combining solid and weak components and resulting in to fragility reduction of boride layer.

1. Introduction

Durability and reliability of machines details and mechanisms in many respects are defined by properties of surface layer, such as the staining and wear, and formation of endurance cracks begins with a surface. Recently in a surface engineering the technologies of surfacing by the concentrated streams of energy created by laser radiation, high-temperature plasma, electron and ion beams will utilize ever more. This treatment enables capability purposeful to change a surface layer condition of machine details and tool etc., as a consequence to refine their

We presented results of boride layers formation on carbon steels under a powerful electron beam. The microstructure and microhardness and also phase composition of boride layers on steel 45 are investigated.

2. Experimental Methods

The electron beam boriding [1] samples were prepared by daub drawing on a previously prepared surface of steel. The daub composition was consisted of 1:1 the boriding compounds (boron carbide B_4C or amorphous boron) and the organic binding. The solution 1:10 glue BF-6 in acetone has been used in qual-ity the binding. After drawing daubs by thickness 1 mm samples dried before complete removal acetone.

Layers Fe_2B and FeB were synthesized from daub containing mixtures of boron carbide B_4C, oxide Fe_2O_3, carbon C (birch charcoal) and the organic bind-ing. The electron beam treatment has been carried out in an electro-vacuum installation with a powerful industrial axial electron gun. The device and the technical parameters of the installation for electron heating are given in the work [2]. The pressure in chamber did not exceed $2 \cdot 10^{-3}$ Pa.

The solid phase borating was carried out at tempera-ture 950 °C and duration 4 hours in a powder mixture containing 97 % B_4C and 3 % KBF_4 in the container with fusible mechanism [3].

The boride layers were analyzed by X-ray diffraction. An X-ray powder diffract meter D8 Bruker using CuK_α-radiation was employed for phase analysis and the determination of lattice parameters. Microhardness and microstructure of prepared layers was measured by using PMT-3 hardness tester at a loading 0,5 and 1 H and with digital camera C4000zoom.

3. Results and Discussion

Fig. 1 shows the microstructure of boride layers. It is established, that at electron beam boriding on a metal surface brightly expressed layers of the thickness about 350–360 microns (the daub from amorphous boron) and depths up 100–110 micron (the daub from B_4C) will be formed. In both cases the precise border between the layer and metal basic is founded. In comparison with the metal basic the layers have lower speed of the etching that testifies to them of considerably high corrosion stability.

The structures of surface layers after solid phase borating and electron beam boriding are distinct. The layer after solid phase borating showed a needle-like structure and the transition zone settles down under it's (Fig. 1, *a*). The transition zone after electron beam boriding was not observed and the legible boundary between a layer and base metal was observable (Fig. 1, *c*). The layer consists of rounded crystals, which are settling down on a surface and a eutectic.

The X-ray diffraction analysis is established that layers contain the iron brides Fe_2B and FeB. The re-la-tive maintenance of these borides has veiled from composition of daub. In case of an amorphous boron forest it FeB, and B_4C-Fe_2B. Besides on X-ray diffraction patterns there are the lines of different in-ten-sity belonging to the cementite Fe_3C and ferrite α-Fe. The boride layer formed from daub B_4C (Fig. 1, *c*) consists from round off engagements,

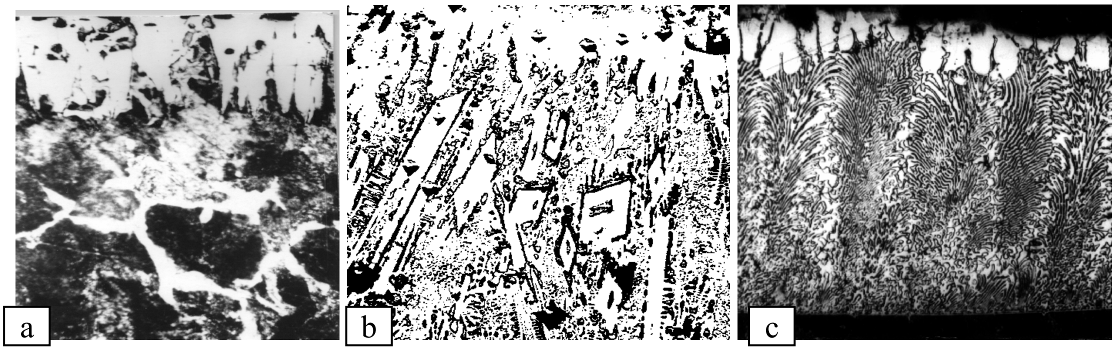

Fig. 1. Layers boride microstructure formed on steel 45 surface: solid phase borating (a) and electron beam boriding – daub from amorphous boron (b) and B_4C (c); a – 250, b, c – 500

which locating on the layer surfaces and eutectic. The microhardness values 820–840 and 510–530 HV for the layer surfaces and eutectic were obtained. The rounds off engagements are primary crystals of borides that answers entropic criterion of stability of the crystals limited form at the crystallization in conditions, approached to equilibrium. In turn, the boride round form determine the form of eutectic crystals.

The boride layer formed from daub with amorphous boron has other structure (Fig. 1, *b*). It consists of particles of the various forms: rhombic, prismatical, dendritical. On layer surface the continuous light film with needles, directed deep into of a sample is placed. Microhardness of film makes up 1200–1250 HV. Inside this film the rare (1–2) large inclusions with microhardness 1750–1820 HV is meet. Under the film there are the primary crystals and eutectic with microhardness 840–880 HV and 500–540 HV, accordingly.

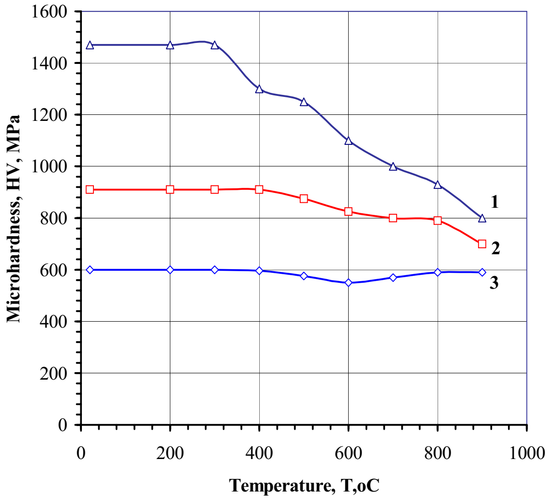

Fig. 2. The effect of heating temperature on boride layer microhardness HV_{50}: 1 – solid phase borating; 2 – rounded crystals (elec-tron beam boriding); 3 – eutectic's

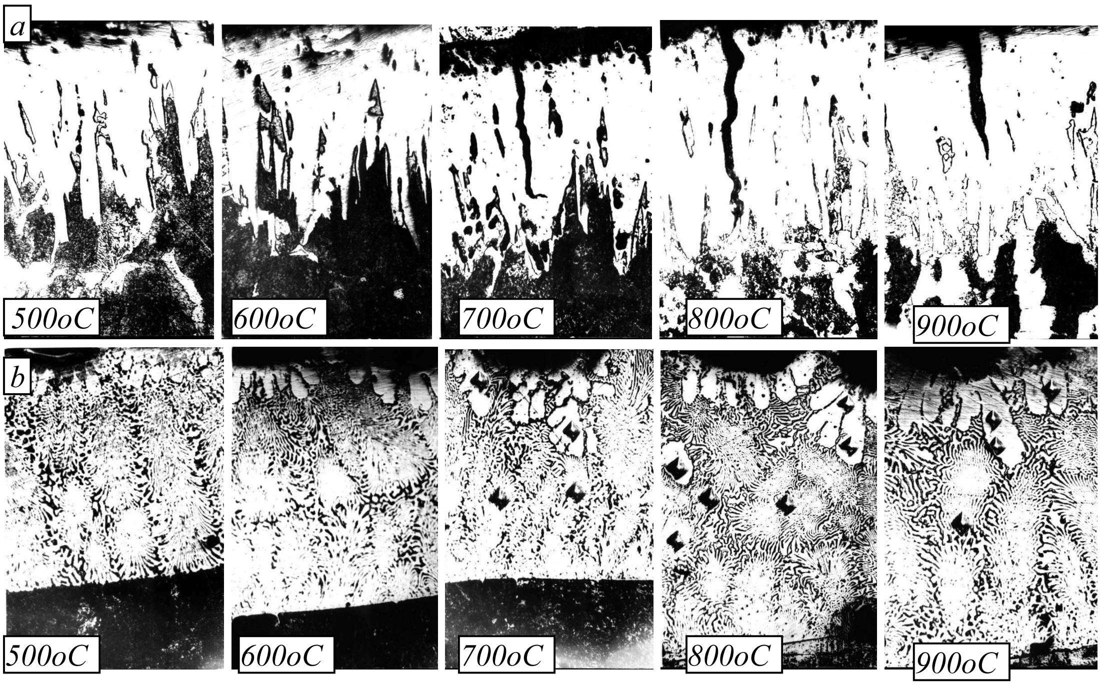

Fig. 3. Layers boride microstructure formed on steel 45 surface: solid phase borating (a) and electron beam boriding (b)

According to [4], the boride iron Fe_2B has tetragonal crystal lattice (Space. group I4/mcm with parameters of an elementary cell *a*=0.51087, *c*=0.42497 nm). At layer formation from daub with amorphous boron, the iron boride crystals have been inherited the form of an elementary cell. Therefore, the primary crystals of borides have the form of rhombuses, parallelograms and etc., caused by different corner inclination of the crystal lattice (prism) to plane of polished specimen. It is necessary to note, that the similar forms of borides crystals are observed and at laser boriding [5].

According to data X-ray diffraction analysis, the layer after solid phase borating consists from iron boride FeB and boron cementite $Fe_3(C,B)$. The microstructure of boride layers is showed on fig. 1, *a*. In mild steels the boride layer has a needle structure, at which the needles of borides growing together in the basis, will form a continuous layer. The plume allocations of carboborides phase are joined directly to the boride needles. Microhardness of borides needles makes up 1300–1350 HV, plume allocations – 300–330 HV. Thickness of borides layers are 70–90 microns.

The effect of heating temperature on microhardness of boride layer is shown (Fig. 2). When the boride layers received as a result of solid phase borating ini-tial condition showed higher hardness by comparison with in layers, received at electron beam boriding and at a heating up to temperature 800–900 °C microhardness becomes practically comparable.

From metallographic analysis it is observed that, from temperature of 700 °C cracks begin to be formed in boride layers received as a result of solid phase borating, (Fig. 3, *a*). The crack was originated on a surface. The increase of heating temperature resulted in propagation of crack deep into of layer. In layers obtained at electron beam boriding, the cracks were not found (Fig. 3, *b*).

It is known [3], that alongside with high hardness and wear resistance, borides layers have also essential lack – the increased fragility. It is established, that layers received by electron beam boriding are more plastics than after solid phase borating. Besides this the layers have heterogeneous structure combining solid and more plastic structural components. Such combination partly explains the lack of thermal cracks at boride layers heating up to high temperatures.

Electron beam boriding from sating daub, possibly, occurs on diffusion mechanism. Application of an electronic beam promotes increase in diffusion of boron in volume of metal, interaction and formation iron borides (Fig. 4 and 5).

We have made attempt of Fe_2B and FeB layers formation during their synthesis from mixtures with participation Fe_2O_3, B_4C and C on a surface of steel 45. For this purpose a mixture of $4Fe_2O_3$:B_4C:11C (Fe_2B) and $2Fe_2O_3$:B_4C:5C (FeB) took and carefully frayed in an agate mortar, mixed with organic binding. Electron beam treatment was carried out in vacuum not above $2{\cdot}10^{-3}$ Pa at capacity of electron beam 250–450 W during 1–3 minutes.

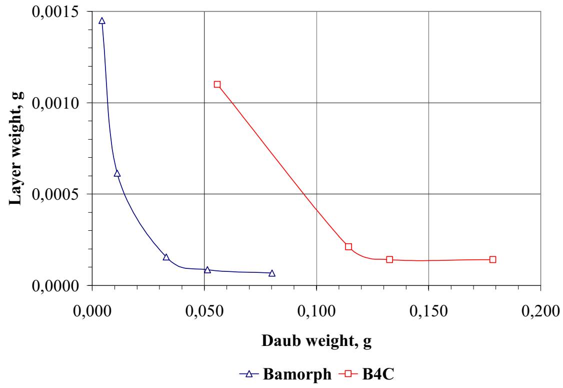

Fig. 4. Influence of daub weight on boride layer weight (steel 45, 270 W, treatment 5 min)

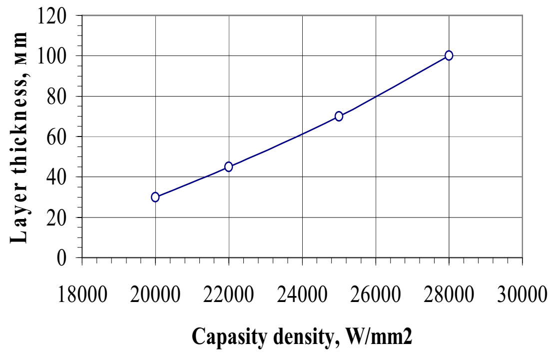

Fig. 5. Influence of electron beam capacity on boride layer thickness (steel 45, daub from amorphous boron, treatment 1 min)

The layers thickness made 200–280 microns (Fe_2B) and 50–80 microns (FeB). The microstructure of a Fe_2B layer is presented on Fig. 6, *a*. The structure is complex, includes primary crystals of boride, dendrite inclusions, and eutectic. On Fig. 5, *b* the microstructure of a FeB layer is resulted. According x-ray analysis, the dendrite inclusions are the B-doped ferrite (boron solid solution into α-Fe).

During formation of a layer by electron beam boriding the evaporation of boron oxides is observed. Therefore to prevention of a deviation of a reactionary mixture from the stoichiometrical composition we applied a blanket of amorphous powder of boron oxide.

Application of a blanket by amorphous powder of the boron oxide promoted reception of the equilibrium borides coating. In all layers we observed eutectic having microhardness 650–700 HV. The round and extended inclusions had the ordered arrangement in a layer, their microhardness was 1080 and 1150 HV (FeB layers), and 1250 1150 HV (FeB+B_2O_3 layers), accordingly. The round inclusions were only in Fe_2B layers (1200 HV) and Fe_2B+B_2O_3 (1150 HV).

According to x-ray analysis, the layers are mainly consisted from borides FeB or FeB.

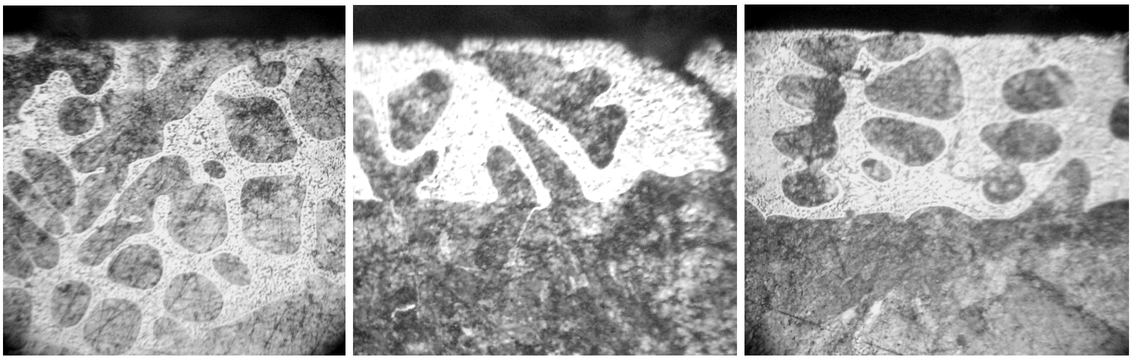

Fig. 6. Microstructure of layers Fe_2B (a), FeB (b) и $FeB+B_2O_3$ (c): ×400

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