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

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Abstract – The microstructure and microhardness of boride layers are investigated and also are compared to layer properties obtained at traditional thermal treatment. Formed surface layers were heterogeneous structure combining solid and weak components and resulting in to fragility reduction of boride layer.

1. Introduction

Durability and reliability of machine 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 operational properties.

In this study we presented results of boride layers formation on carbon steels under a powerful electron beam. The microstructure and microhardness of boride layers are investigated and also are compared to layer properties obtained at traditional borating.

2. Experimental Methods

The materials studied in this work, were steel 45.

The daub was piled up a previously preformed surface of samples with thickness of 0.5–1 mm. Boron carbide B_4C and the organic binding were entered into the daub composition. 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 [1]. The pressure in chamber did not exceed $2 \cdot 10^{-3}$ Pa. An electronic heating was carried out within 2–5 minutes at specific power of 2–2.5 W/m^2.

For comparison the traditional borating was carried out at temperature 950 °C and duration 4 hours in a powder mixture containing 97% B_4C and 3% KBF_4 in the container with fusible mechanism [2].

The boride layers were analyzed by X-ray diffraction. An X-ray powder diffractometer DRON-2M using Co Kα- radiation was employed for phase analysis. Microhardness of prepared layers was measured by using PMT-3 hardness tester at a loading 0,5 and 1 H. The microstructure of the samples was observed using a metallographic microscopy "Neophot-21".

3. Results and Discussion

3.1. Layers structure of iron borides

As it shown in Fig. 1, the structures of surface layers after traditional borating and electron beam boriding are distinct. The layer after traditional borating showed a needle-like structure and the transition zone settles down under its (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,b). The layer consists of rounded crystals, which are settling down on a surface and a eutectic.

The layer thickness made after traditional borating was 70–90 microns and after electron beam boriding – 220 microns.

The effect of heating temperature on microhardness of boride layer is shown (Fig. 2). When the boride layers received as a result of traditional borating initial 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 973 K cracks begin to be formed in boride layers received as a result of traditional 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 [2], that alongside with high hardness and wear resistance, borides layers have also essential lack – the increased fragility. For an estimation of borides layers fragility a number of fragility have been determined. The number of fragility is determined on character of brittle failures incipient in an impression, which is formed at dip microindenter in metal under a various loading 20–150 g (Table). From this table it was recognized follows that layers received by electron beam boriding are more plastics than after traditional borating.
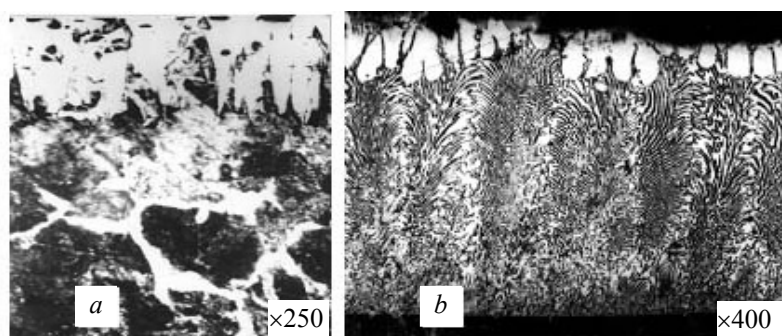
Fig. 1. Layers boride microstructure formed on steel 45 surface: traditional borating (*a*) and electron beam boriding (*b*)
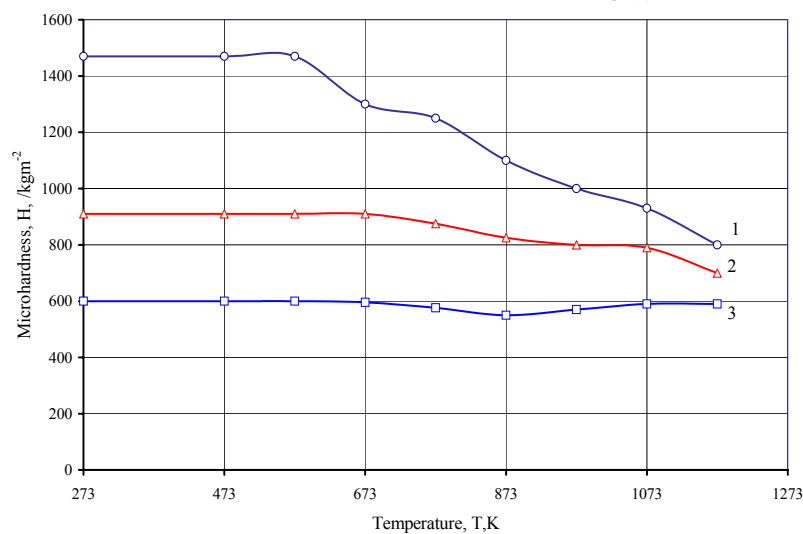

Fig. 2. The effect of heating temperature on boride layer microhardness HV_{50}: 1 – traditional borating; 2 – rounded crystals (electron beam boriding); 3 – eutectic's
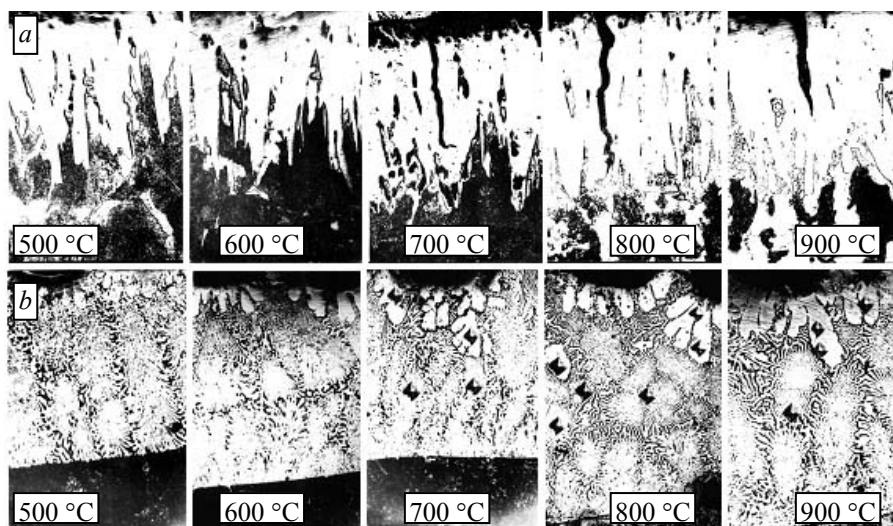

Fig. 3. Layers boride microstructure formed on steel 45 surface: traditional borating (*a*) and electron beam boriding (*b*)

Table. Fragility of borides layers on steel 45

Traditional borating		Rounder crystals (electron beam boriding)		Eutectic's (electron beam boriding)	
Loading (g)	Fragility number	Loading (g)	Fragility number	Loading (g)	Fragility number
20	0	50	0	70	0
50	1	70	1	100	0
70	2	100	2	120	0
100	4	120	4	150	1
120	5	150	4		

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.

The boride layers fragility depends on phase composition. Is fixed, that the number of brittle failure of boride iron Fe_2B is less, than FeB, approximately twice [3]. The aggregate fragility number is determined by phase composition of boride layer. On the X-ray data the surface layer after traditional borating consists of borides FeB, Fe_2B and boron cement carbide. Layer is two phases. The first zone is zone of borides. On a surface the needles of boride FeB, under them Fe_2B, then transition zone boron cement carbide settle down. Alongside with high fragility the boride two-phase layer has the brightly expressed propensity to shear. The shear occurs on a demarcation of phases. In a single-phase layer the shear is observed on boundary of continuous layer. Hence, single-phase boride layers are less inclined to shear.

Boride layer formed at electron beam boriding, has a microstructure of an eutectic type. The structure it depends on composition of borating component. So, the layer received at boriding from amorphous boron, consists mainly from Fe_2B. The boride FeB is a dominating phase in layer formed from sating daub containing a boron carbide B_4C. Notwithstanding what the layer consist mainly of boride FeB, nevertheless, microstructure is formed under influence of boride Fe_2B. Circle plugging are primary crystals. It responds an entropic stability criterion of the restricted molding box of crystals at crystallization in requirements, approximate to equilibrium. According to this test, if the value of entropy of fusion (ΔS) does not exceed 2 кал/mol the crystals have circle form [4]. Received in work [5] values of entropy of fusion for boride iron Fe_2B make $\Delta S = 1.5$ кал/mol. In turn, circle form of borides determines the molding box eutectic crystals.

3.2. Layers Structure of Iron Borides Synthesized from an Iron Oxide, Boron and Carbon

We have tried to receive single-phase layers consisting of borides of iron Fe_2B and FeB. For this purpose stoichiometrical mixes Fe_2O_3:3B:3C (Fe_2B) and Fe_2O_3:2B:3C (FeB) carefully frayed in an agate mortar, immixed with organic binding and superimposed glutinous suspension on a surface of a sample of steel Ст3. We used amorphous boron and carbon (birch charcoal). Electron treatment carried out in vacuum not above $2 \cdot 10^{-3}$ Pa at power of an electron beam 250–450 W during 1–3 min.

The layer thickness made 200–280 microns (Fe_2B) and 30–45 microns (FeB).

On the X-ray data, the first layer mainly consists of boride Fe_2B. The layer of the second sample has in the composition of boride iron FeB.

The microstructure of a layer on a base Fe_2B is submitted in Fig. 4a. The structure composite includes primary crystals of boride, dendrite plugging of a eutectic.
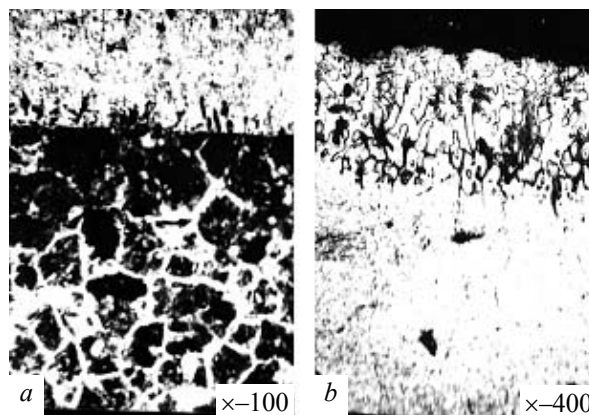

Fig. 4. Layer boride microstructures: Fe_2B (*a*), FeB (*b*)

The layers FeB by thickness 30–45 microns, irregularly settled down on a surface, had no a legible demarcation with a metal base. The particles in a course had no the particular molding box, were laminar.

All studied layers have structure of an eutectic type. The similar microstructures are formed in no isothermal condition of a high-speed heating up to temperatures (1100–1150 °C), exceeding a melting temperature threefold (Fe–B–C) or double (Fe–B) of a eutectic. The formation of a eutectic begins on boundary «boride layer – transition zone», then it promptly propagates both in the party of a base metal, and to a surface. The process interrupts at the moment of a run out of a solution phase on a surface or hardly earlier. The layers having a similar microstructure are characterized by smaller fragility and propensity to shear, and also higher operational properties.

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