Synthesis of transition metal borides layers under pulsed electron-beams treatment in a vacuum for surface hardening of instrumental steels

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Abstract: The saturation of the surface layers of metals and alloys with boron is conducted for increasing their surface hardness, wear resistance, etc. Multicomponent layers containing in its composition borides of refractory metals, as a rule, are formed by the methods of chemical-thermal processing in the interaction of boriding component with refractory one or by the method of saturation of refractory metal impurities or alloy with boron. In this work, we studied the features of vanadium and iron borides formation on the surface of instrumental steels U8A and R18 under the influence of intense electron beams in continuous and pulse modes.

Keywords: Electron beam, borides, alloys, microhardness, X-ray diffraction, structure, self-propagating high-temperature synthesis (SHS)

1. Introduction
The saturation of the surface layers of metals and alloys with boron is carried out to increase their surface hardness, wear resistance, etc. Application of electronic heating with high (> 10^9 W/m^2) power density in a vacuum owing to fast inertialess allows obtaining extremely high temperatures and facilitating the regulation of heating in a wide temperature range, as well as opens ample opportunities for creating protective layers on the basis of borides of refractory metals.

In [1] reported about formation of strengthening coatings based on TiB_2, CrB_2, VB_2, W_2B_5 on carbon steels under influence of an electron beam on boron-containing reactionary daubs in vacuum. The assumption of an active role of a surface metal has been made at electron-beam welding of self-heating synthesis products which was initiated by an electron beam and proceeding in reactionary daub of stoichiometric mixtures.

In this work, we studied the features of vanadium and iron borides formation on the surface of instrumental steels U8A and R18 under the influence of intense electron beams in continuous and pulse modes.

2. Layers of based on the boride VB_2 on steel U8A
The layers of vanadium boride VB_2 (later also borides of iron Fe_2B and FeB) are synthesized and formed on the surface of cutting plates in size 12×12×5 mm with roughness Rz = 4.41 ± 4.02 μm of instrumental steels U8A and R18 at the same time. Samples were prepared by applying the reactionary
daubs on the pre-prepared (well-fat) surface of the steel. The composition of the daubs consisted of 1:1 by volume the stoichiometric mixture of oxide, boron-containing component and carbon, as well as organic binder - solution 1:10 of potassium BF-6 in acetone. Amorphous boron, charcoal (birch) and oxides \( V_2O_3, Fe_2O_3 \) were used as initial substances. Processing of samples was being conducted for 2-5 minutes with the power of electron beam of 150-300 watts. The pressure in the vacuum chamber did not exceed \( 2 \times 10^{-3} \) Pa [2, 3].

X-ray diffraction revealed the prevalence of carbide phases (cementite) in the boride layer on steel U8A. This can be explained only by the deviation from stoichiometry at evaporation of the intermediate boron oxide. Application of the protective layer of the amorphous oxide \( B_2O_3 \) (1:1 by volume of reactionary daub: daub based on \( B_2O_3 \)) allowed to form more uniform boride layers. It was found that the weight of the crystalline phases was 92.3%, and the amorphous phases' weight was 7.7%, while the crystallite size ranged from 15 to 70 nm.

The X-ray patterns of all the investigated boride layers of vanadium show the reflections of vanadium ferrite \( \alpha-Fe-V \), corresponding to phase \( \alpha-Fe_{9}V \) of space group \( I\overline{m}3m \), with a cubic lattice parameter \( a = 0.2878 \) nm and cementite \( Fe_3C \) (PDF 00-003-0989) of space group \( Pbnm \) with elemental rhombic cell parameters \( a = 0.4518, b = 0.5069 \) and \( c = 0.6736 \) nm, \( z = 4 \) (Fig. 1).

![Figure 1. X-ray pattern of layer based on VB2 formed on steel U8A](image)

Boride \( V_2B_4 \) (PDF 03-065-2551, space group \( I\overline{m}mm \), with elemental rhombic cell parameters \( a = 0.303, b = 1.318 \) and \( c = 0.2986 \) nm, \( z = 2 \)) can be found in X-ray patterns of diboride vanadium layers. Formation of diboride vanadium \( VB_2 \) was not radiographically observed, although there were reflexes of light intensity related to the interplane distances of this phase. So, it should be investigated further.

Fig. 2 shows a general view (Figure 2.a) and the structure of the boride layer VB2 on the surface of cutter plate steel U8A (Fig. 2.b). Layer thickness reaches up to 500 µm.

Microhardness testing showed its uneven distribution by thickness in the cross-section. Very rare separate inclusions have microhardness of \( HV \approx 24000 \) MPa and are located in the subsurface layers. Next, we observe two regions: in the first region the microhardness reaches 2500 MPa, and in the second one it is 1500 MPa. The metal base has microhardness of 200 MPa.
3. **Layers of iron borides Fe₂B and FeB on high-cutting steel R18.**

X-ray diffraction (XRD) showed that after electron beam treatment in the samples of cutter plate steel R18 we observed the reflexes of the following phases: metal substrate - α-Fe and carbide W₃Fe₃C. It was found that the weight of the crystalline phase is 92.7% and the amorphous phase is 7.3%.

Boride FeB is located near the surface of the coating. This is evidenced by the results of the investigation of the end surface of the cutter plate steel R18 and X-ray analysis data (scanning electron microscope JSM-6510LV JEOL with microanalysis system INCA (Fig. 3).

Application of the protective layer of amorphous oxide B₂O₃ lead to obtaining equilibrium boride layers Fe₂B and FeB.

Figure 3. General view (a) and structure (b) of boride layer FeB on steel P18
After the electron-beam boriding of steel R18 with the power density of the electron beam \( J = 2.8 \times 10^4 \) W/cm\(^2\) layer was formed as a result of deep weld penetration, which determined its structure. Fig. 4a shows that at directed crystallization the main axis of dendrites line is oriented to the direction of heat removal. The structure of dendrites (a chain of separate globules) indicates about the intermittent nature of their formation. When the the power density of the electron beam is \( J = 2.5 \times 10^4 \) W/cm\(^2\) the layer mainly consists of stellate dendrites (Fig. 4b). When reducing the power density up to \( J = 2.2 \times 10^4 \) W/cm\(^2\), surface layer also contains stellate dendrites, but their number is not dominant (Fig. 4c, d). Microhardness of the layer is 1100-1860 kg/mm\(^2\). The layer thickness is 25-100 µm.

After electron beam treatment of cutting plate with boron-containing daubs, a layer with thickness of 8-10 µm was formed on the surface (Fig. 5). The formed layer has a specified thickness that is practically along its entire length. The image at 500× magnification shows that the layer contains particles which are located not only on the layer, but also on a boundary of base layer. This allows concluding that these particles are carbides of alloying elements (tungsten, chromium, molybdenum and vanadium). The layer firmly holds on the metal base. The microhardness of the layer is slightly higher than that of the base, and it is 550 HV and 410 HV, respectively.

To evaluate the tribological properties of the resulting layer we conducted wear resistant tests of the cutters. Tests were performed on screw-cutting lathe 1A616 in the following modes: feed was 0.1 mm/rev, rate speed was 224 rev/min, depth of cut was 1.0 mm. Processed material was steel 45 and sus321. The cutters wear was determined by the clearance face using Brinell magnifying glass.
Geometric parameters of cutters are as follows: rake angle $\gamma = 15^\circ$, main back angle $\alpha = 8^\circ$, main cutting edge angle $\varphi = 45^\circ$. Criterion of cutters resistance is the time of reaching the limit flank wear of 0.6 mm.

The Fig. 6 shows that formation boride layers with thickness of 8-10 $\mu$m on the front surface, allows to increase the resistance of cutters almost in 1.5 times (for processed steel sus321).

![Figure 5. The microstructure of cutters from steel R18 after electron beam treatment](image)

![Figure 6. Wear kinetics of cutters from steel R18; 1-R18 hard (steel sus321), 2-R18 thermochemical treatment (steel 45), 3-R18 electron-beam boriding (steel sus321), 4-R18 hard (steel 45)](image)

A more significant increase of cutters resistance is connected with increase of boride layer thickness. At electron beam boriding the thickness of boriding layer can reach 300 $\mu$m, but the processing temperature is 1100-1200$^\circ$C. The processing time is 2-3 minutes. On that basis, the electron-beam boriding cannot be recommended as a final processing, but as an intermediate processing operation of the cutting tool, for example, before hardening. It should be noted that to
achieve the same thickness of a layer when diffusion borating (for example, when boriding in hermetically sealed containers with safety valve) it take at least 3 hours.

5. Conclusion
The structure and properties of boride layers and their formation on the surface of cutting plates of carbon steel U8A formed during electron beam treatment in vacuum were investigated and discussed. It was found that at the directed crystallization the main axis of dendrites line is oriented to the direction of heat removal. The structure of dendrites (a chain of separate globuly) indicates about intermittent nature of their formation. The microhardness of layer is 1100-1860 HV; the thickness is 25-100 µm.

The formation of boride layers with the thickness of 8-10 µm on the front surface allows increasing the resistance of cutters almost in 1.5 times (for treated steel sus321).

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