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Formation of a boron-based protective coating on the surface of D2 die steel by electron beam processing

A.S. Milonov, D.E. Dasheev, A.P. Semenov, U.L. Mishigdorzhiyn*

Institute of Physical Materials Science SB RAS, Ulan-Ude, Russia
terwer81@mail.ru

Abstract. The features of the formation, composition, structure and microhardness of boride layers on the surface of D2 die tool steel formed during electron beam processing in vacuum are considered. Formed layers have a heterogeneous structure, which combines solid and plastic components resulting in the fragility reduction of boride layer.
Keywords: electron beam, XRD, EDX, borides, metallography, microhardness.

1. Introduction

One of the promising approaches of alloys' hardening is the process of boriding, diffusion saturation with boron. Boriding is carried out in order to increase the wear resistance, corrosion resistance of iron-carbon alloys and scale resistance at temperatures up to 800 °C [1, 2]. As a result of the boriding process, chemical compounds are formed on the surface of the workpieces – borides, which have high anti-corrosion and wear-resistant characteristics, which make it possible to improve surface properties – wear resistance, corrosion resistance, heat resistance [3, 4]. The diffusion layer formed during boriding, on the one hand, has high hardness and wear resistance, on the other hand, it is extremely brittle. Therefore, one of the main directions for increasing the plasticity of boride layers is the use of concentrated energy sources, in particular, accelerated electron beams, which allow changing fundamentally the surface layer structure. [5]. Layers after electron-beam boriding (EBB) have a heterogeneous structure that combines hard (brittle) and more plastic structural components [6].

In this paper, we consider the features of the formation, composition, structure, and microhardness of boride layers formed during electron beam processing in vacuum on the surface of D2 die tool steel.

2. Research methods

The microstructure of the samples was studied on an image analyzer based on a METAM BP-22 metallographic microscope. Microhardness was determined on a PMT-3M microhardness tester at a load of 0.5 N [7, 8]. Phase composition was revealed on a Phaser D2 Bruker diffractometer ($Cu_{K\alpha}$ - radiation) [9]. Elemental composition of the layer structures was studied using a JSM-6510LV JEOL scanning electron microscope (Japan) with Energy-dispersive X-ray (EDX) spectroscope INCA Energy 350, Oxford Instruments (The United Kingdom) at the Science Center «Progress» of the East Siberia State University of Technology and Management.

EBB was carried out on the surface of D2 die steel. Cold-formed die steel with a high chromium content and inclusions of molybdenum (average 0.5%) and vanadium (average 0.2%). D2 steel has good heat resistance and strength, high hardenability, hardenability and wear resistance. Microhardness of D2 steel is 256 HV. Also, this steel is technological, well processed by cutting and pressure, satisfactorily polished. Table 1 shows the chemical composition of steel.

Table 1. Chemical composition in wt% of D2 steel in accordance with GOST 5950-2000

C	Si	Mn	P	S	Cr	Mo	Ni	V	Cu	W	Fe
1.45–1.65	0.1–0.4	0.15–0.45	0.03	0.03	11–12.5	0.4–0.6	0.35	0.15–0.3	0.3	0.197	rest

Samples were prepared by applying a past of amorphous boron to a previously prepared steel surface. Samples were heated by pulsed and stationary (scanning) electron beams. Pulse mode: accelerating voltage 20 kV; electron beam current 60 A. Processing was carried out with a single pulse duration of 20 μs; number of pulses 320; beam current pulse repetition rate 4 Hz. Pressure in the vacuum chamber $5 \cdot 10^{-2}$ Pa [10, 11]. Scanning mode: heating time ~5 minutes. Power density $5.7 \cdot 10^4$ W/cm^2, beam focal spot diameter 1 mm, sweep frequency 50 Hz. Residual pressure in the vacuum chamber 10^{-4}–10^{-3} Pa. Processing modes: accelerating voltage 17 kV and electron beam current 24 mA [12].

3. Results and discussion

Thermodynamic calculations showed that using a past coating of amorphous B, iron boride Fe_2B, manganese boride MnB_2, chromium boride CrB_2, molybdenum boride MoB and vanadium borides VB_2, V_3B_4 formation as shown in Fig.1 [6].

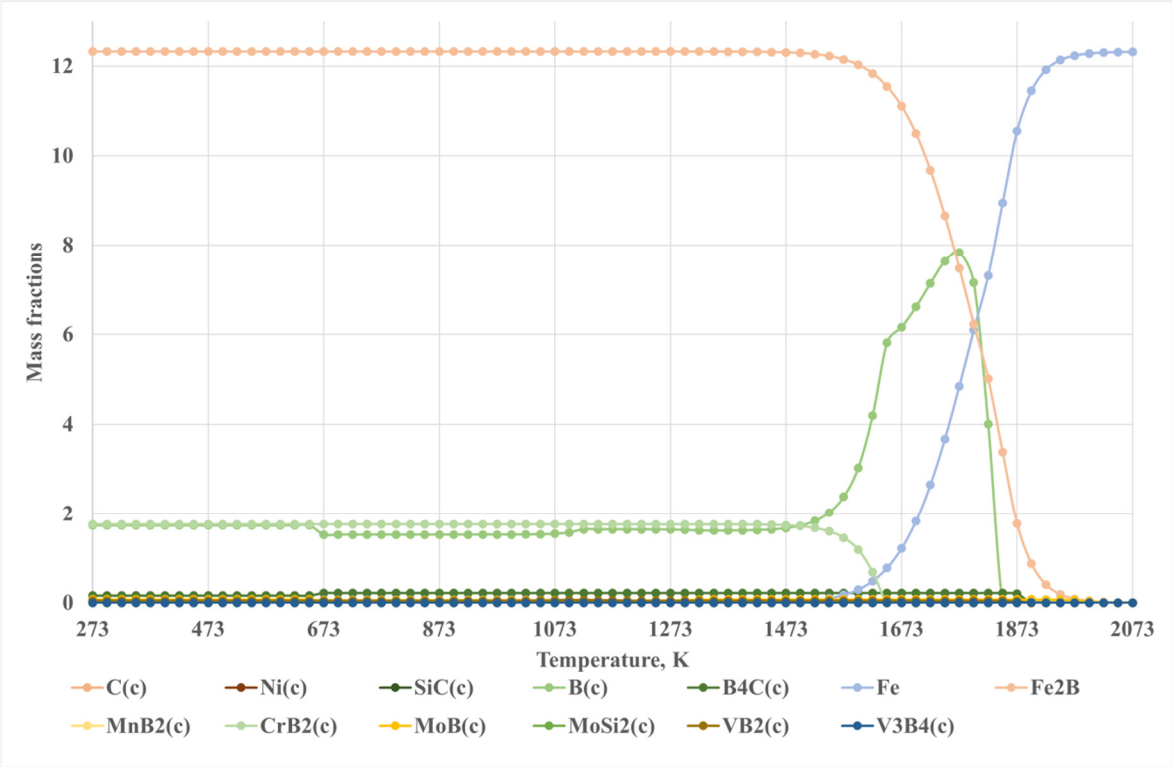


Fig.1. The output of the condensed phases during EBB of D2 steel.

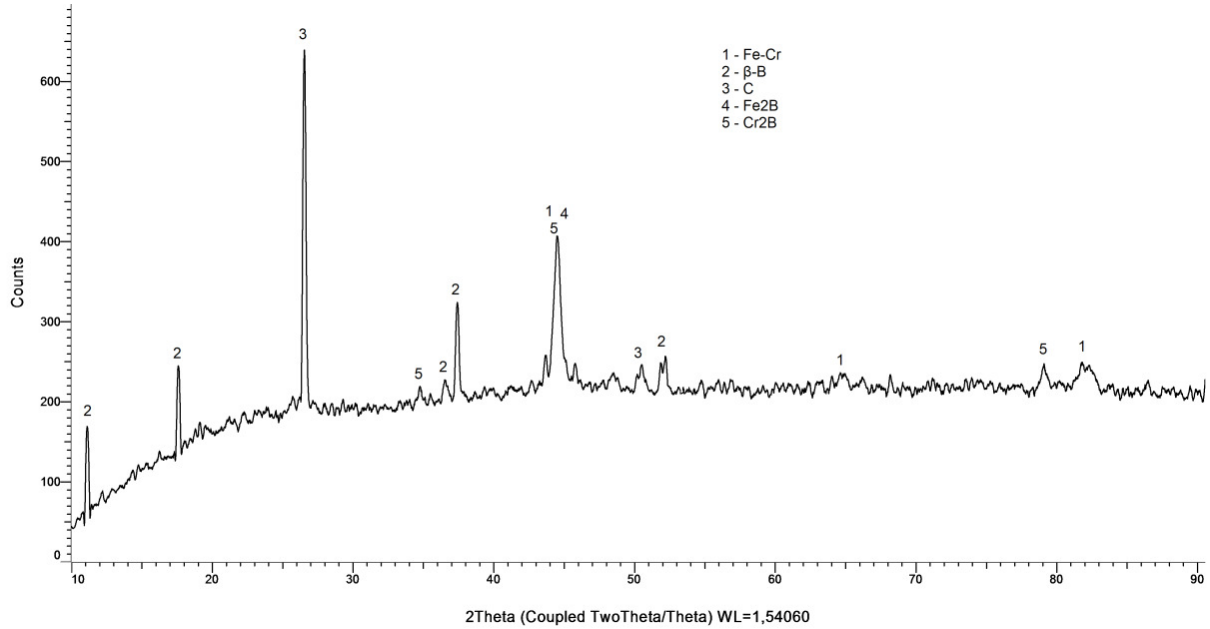
On the basis of thermodynamic calculations experiments on electron-beam boriding were carried out.

Table 2 shows the mass of samples without coating and with coating, before and after EBB. It is obvious that the weight of the samples after EBB increased and indicates that D2 steel was alloyed with boron. [13–14].

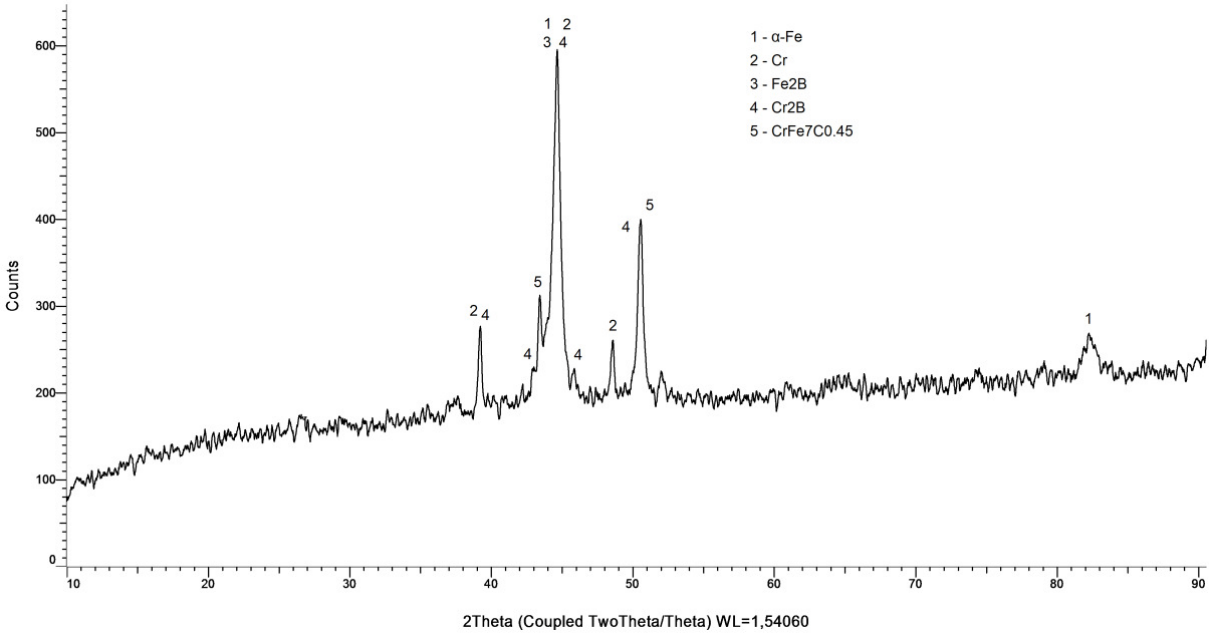
Table 2. Samples' mass change before and after the EBB on D2 steel

Sample №	Processing method	Mass of sample, (g)	Weight before processing with coating applied, (g)	Weight after processing, (g)
1	Scanning beam	13.4287	13.4641	13.4578
2	Pulsed beam	13.4185	13.4464	13.4163

X-ray diffraction (XRD) analysis of the obtained layers showed that during EBB in both modes, in the spectra there are lines of iron boride and chromium boride. The absence of lines of borides of other metals is explained by the fact that their number is extremely small Fig.2.



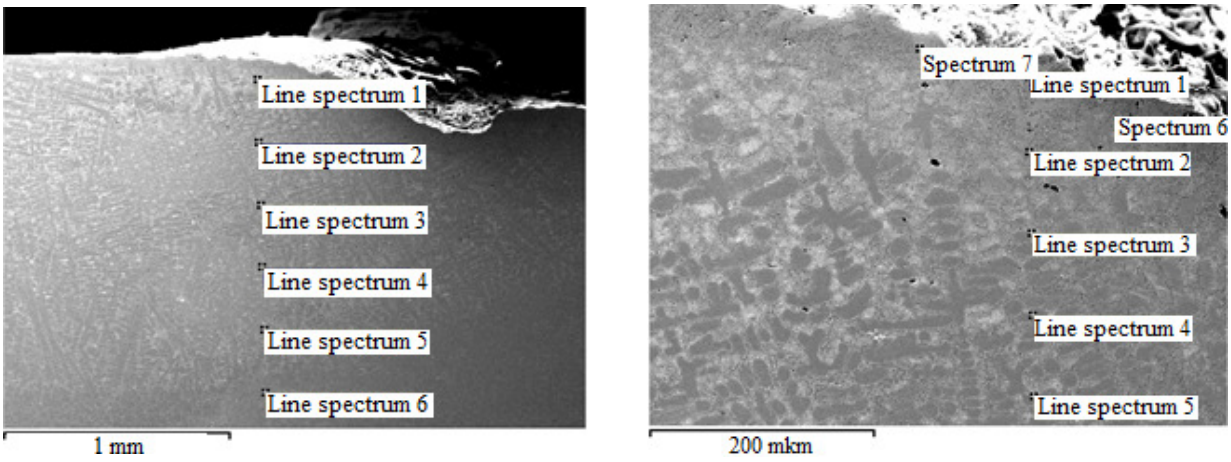
(a)



(b)

Fig.2. X-ray pattern of the layer on D2 steel (a – scanning mode. b – pulsed mode).

EDX analysis after processing by the scanning electron beam showed that boron was not detected on the surface of the layer, probably due to its very small amount. A high chromium content of 20.73 % was noted, which indicates the presence of borides of this metal (Fig.3a, Table 3). Carbon is present along the entire profile of the layer and reaches a maximum amount of 33.1 % at the surface, Fig.2b, Table 4. This indicates that iron carbides are present in the layer.



(a) (b)

Fig.3. SEM images with EDX analysis points of the layer after EBB by a scanning beam: a) – section 1 (×50); b) – section 2 (×x250).

Table 3. The elemental composition of D2 steel (sample 1) in section 1. wt%

Line spectrum	C	Si	S	V	Cr	Fe	Ni
1	5.94	0.19	-	0.38	12.88	80.38	0.24
2	7.33	0.10	-	0.35	17.14	74.76	0.32
3	8.05	0.57	-	0.14	7.46	83.43	0.35
4	7.97	0.48	-	0.12	7.99	83.15	0.29
5	9.24	0.10	0.27	0.56	20.73	68.98	0.12
6	5.45	0.31	-	0.10	9.09	84.79	0.26
Max.	9.24	0.57	0.27	0.56	20.73	84.79	0.35
Min.	5.45	0.10	0.27	0.10	7.46	68.98	0.12

Table 4. The elemental composition of D2 steel (sample 1) in section 2. wt %

Spectrum	C	Si	V	Cr	Fe	Ni
1	8.59	0.35	0.36	11.73	78.76	0.21
2	5.92	0.55	.09	7.21	85.91	0.31
3	7.83	0.37	0.21	11.42	79.90	0.27
4	8.93	0.40	0.15	7.84	82.32	0.37
5	8.92	0.48	0.08	7.90	82.45	0.16
6	33.10	0.10	0.18	9.62	56.79	0.21
7	6.19	0.57	0.08	7.31	85.58	0.27
Max.	33.10	0.57	0.36	11.73	85.91	0.37
Min.	5.92	0.10	0.08	7.21	56.79	0.16

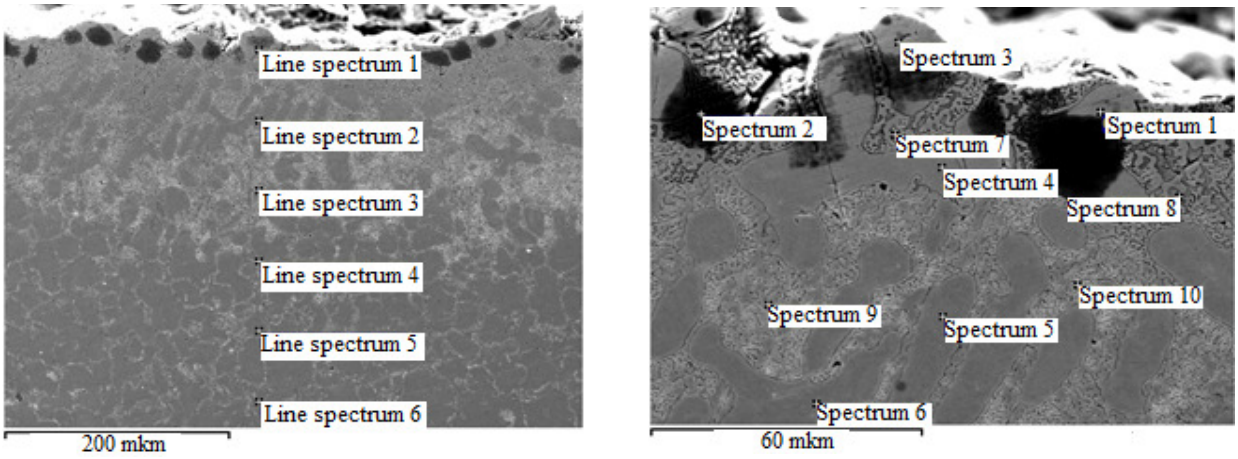
During pulsed boriding chromium content reaches up to 21.38%. Fig.4a, Table 5. Carbon is also present throughout the coating profile and reaches a maximum amount of 39.48% at the surface. Fig.4b, Table 6.

Table 5. The elemental composition of D2 steel (sample 2) in section 1. wt%.

Line spectrum	C	Si	S	V	Cr	Fe	Ni
1	10.01	0.49	-	0.10	8.16	80.94	0.29
2	9.30	0.54	-	0.13	7.83	81.83	0.37
3	7.00	0.13	0.31	0.46	19.94	71.93	0.23
4	11.63	0.51	-	0.14	8.73	78.73	0.26
5	12.17	0.40	-	0.12	9.30	77.56	0.46
6	15.47	0.18	-	0.48	21.38	62.34	0.14
Max.	15.47	0.54	0.31	0.48	21.38	81.83	0.46
Min.	7.00	0.13	0.31	0.10	7.83	62.34	0.14

Table 6. The elemental composition of D2 steel (sample 2) in section 2. wt%.

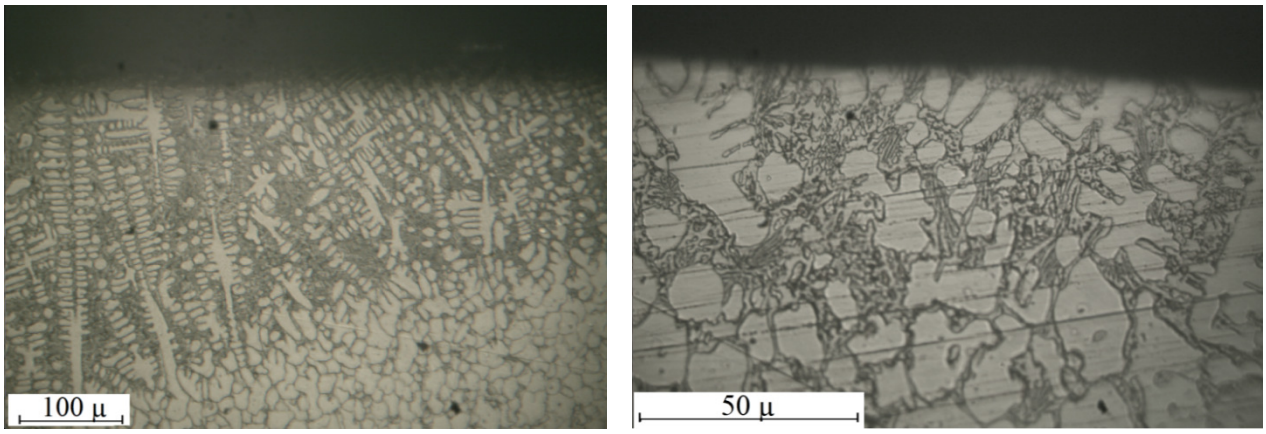
Spectrum	C	O	Si	S	V	Cr	Fe	Ni
1	14.48	-	0.46	-	0.05	7.46	77.28	0.28
2	39.48	2.47	0.16	-	0.26	10.07	47.53	0.03
3	6.95	-	0.41	-	0.12	7.68	84.52	0.31
4	6.54	-	0.48	-	0.10	8.28	84.34	0.26
5	8.59	-	0.60	-	0.10	7.92	82.49	0.30
6	8.77	-	0.47	-	0.10	8.12	82.15	0.39
7	9.49	-	0.28	0.42	0.38	17.85	71.42	0.16
8	8.86	-	0.07	-	0.43	19.36	71.10	0.18
9	7.78	-	0.15	-	0.47	18.54	72.81	0.26
10	7.89	-	0.15	-	0.40	16.44	75.08	0.05
Max.	39.48	2.47	0.60	0.42	0.47	19.36	84.52	0.39
Min.	6.54	2.47	0.07	0.42	0.05	7.46	47.53	0.03



(a) (b)

Fig.4. SEM images with EDX analysis points of the layer after EBB by a pulsed beam:
a) – section 1 (×250); b) – section 2 (×1000).

The microstructure and microhardness of the resulting layers are shown in Figs.5–6. Metallographic analysis showed that as a result of processing, inhomogeneous coatings with low roughness and no visible signs of zoning (layering) up to ~0.5 mm thick were formed.

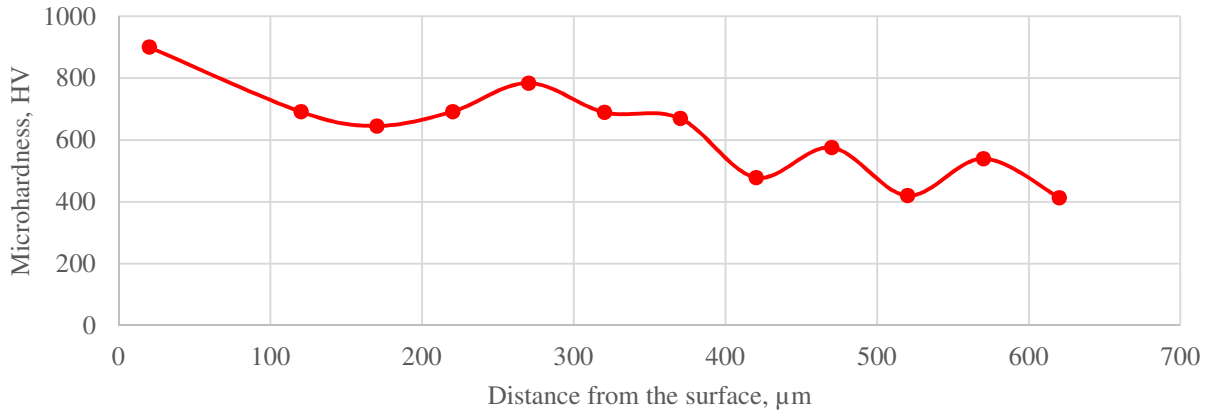


(a) (b)

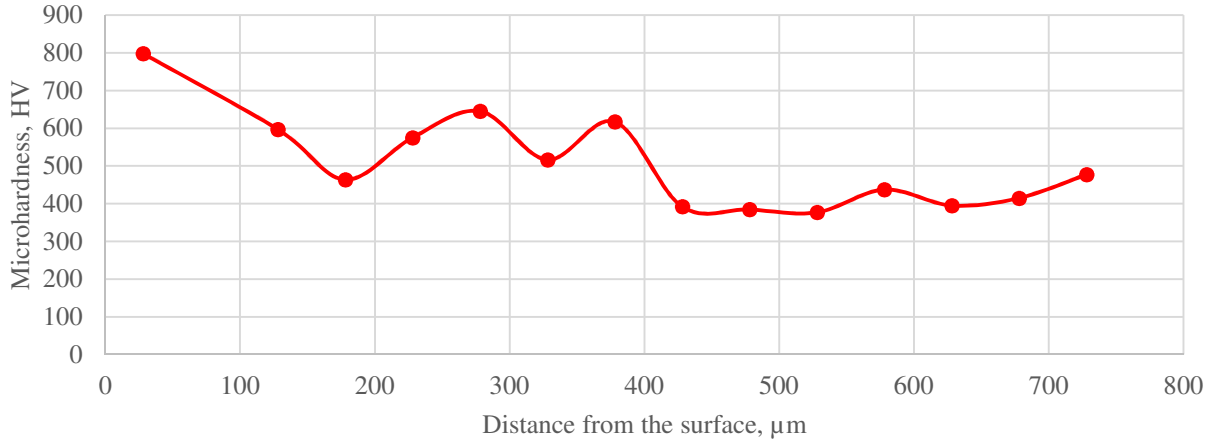
Fig.5. Microstructure of the layer after EBB. a) – scanning mode; b) – pulsed mode.

Ultrafast heating and subsequent cooling led to the formation of dendrites containing α-Fe throughout the entire thickness of the coating, oriented at different angles to the surface, Fig. 5. Layer thickness in the sample was 200–500 micrometers, Fig.5. It can be seen that various inclusions are present over the entire surface of the layer, which are mainly particles of iron and chromium borides, while there are borides of other metals that make up this steel.

The microhardness of the obtained layers reaches 900 HV in the scanning mode and 800 HV in the pulsed mode Fig.6. The fluctuations in the microhardness values in the layer in both samples are explained by the fact that the solid particles of borides are in the soft matrix of α-Fe. It can be seen that the harder particles are closer to the layer surface.



(a)



(b)

Fig.6. Microhardness distribution in the layer after EBB. a) – scanning mode; b) – pulsed mode.

4. Conclusions

Electron-beam boriding with amorphous boron of D2 steel leads to the formation of modified layers up to 0.5 mm thick. It has been shown by thermodynamic calculations that iron borides Fe_2B and borides of metals that make up this steel, in particular chromium boride Cr_2B, can be formed during EBB. XRD and EDX analyses showed that the calculation results correspond to the experimental results. The microstructure and microhardness of the obtained layers indicate their heterogeneous structure, which combines hard (brittle) and more ductile structural components, which generally determine the effect on reducing the fragility of the layers. The data obtained make it possible to recommend the use of EBB D2 die steel to increase surface wear.

Acknowledgements

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