

# Creation of the nanostructured superhard and heatproof layers on die steels under influence of intensive electron beams in vacuum

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**Abstract:** Studying and comparing microstructure and microhardness of boride layers formed on die D2 steel by using different methods – electron beam borating with continuous and pulsed beams under vacuum. The created layers have a heterogeneous structure combining hard and plastic components resulting in decrease of boride layer embrittlement.

## 1. Introduction

Today in the conditions of ever-evolving technology there are growing requirements for the material strength of machine, device and tool components, especially with regard to their high temperature strength and heat resistance. Transition metal borides have high fusing temperatures (above 2,000°C) and hardness values and are quite oxidation-proof which makes them of particular interest in terms of coating formation basis. Boride layers have high physical-and-mechanical parameters. Layer microhardness can reach 20,000 MPa provided that these microhardness values can prevail until temperatures reach ~ 600-700°C which allows to apply boriding for improving wear resistance of products made of die D2 steel and operating under high temperatures [1-3].

**Table 1.** Chemical composition of D2 steel material.

Chemical element	%
Carbon (C)	1.45–1.65
Vanadium (V)	0.15–0.30
Silicon (Si)	0.1–0.4
Copper (Cu), maximum	0.3
Manganese (Mn)	0.15–0.45
Molybdenum (Mo)	0.4–0.6
Nickel (Ni), maximum	0.35
Phosphorus (P), maximum	0.03
Chromium (Cr)	11.0–12.5
Sulphur(S), maximum	0.03

D2 steel is cold deformation die steel with a high content of chrome and inclusions of molybdenum (0.5% on average) and vanadium (0.2% on average). D2 steel has high heat resistance, strength, through-hardening capability, hardening capacity and wear resistance. D2 steel microhardness is 2.560



MPa. This steel is also processable, can easily be machined and mechanically shaped and acceptably grinded.

As is well known, steels with 1.25-1.45% C content refer to ledeburitic class steels, i.e. contain carbide eutectics as-cast and have quench hardness of 8.41-9.07 MPa. These steels contain a high amount of carbide-forming elements and an increased amount (20%) of chrome carbides ( $\text{Cr}_7\text{C}_3$ ,  $\text{Cr}_{23}\text{C}_6$ ).

Since chrome (Cr) represents an alloying element of cold deformation die steel it improves machining (cutting) properties and wear resistance and enhances steel hardness and through-hardening capability which is particularly important for fabricating large male dies and die blocks.

Wolfram (W) is introduced into steel to improve hardness, wear resistance and through-hardening capability which also enhances the cutting capability of the tool.

Vanadium (V) contained in die steels is present in VC carbide and solid solutions. Vanadium significantly reduces the die steel responsivity to overheating, enhances steel heat resistance and improves the proeutectoid constituent particle distribution. With 0.3–0.5 % content of vanadium steel strength and plasticity will be significantly higher compared to those of high vanadium steels.

Molybdenum (Mo) is introduced into high-chromium steel to enhance its ductility and improve through-hardening capability. At the same time molybdenum has a negative effect on scaling resistance and due to this reason its content is limited to 1.4–1.8 %.

Therefore, high-chromium D2 steel is applicable for fabricating dies, male dies and rollers with 4.56...5.80 MPa hardness and under the operating temperature of up to 700 °C.

In the context of this study layers were obtained as a result of electron-beam borating (EBB) [4]. Saturating coating with 0.5-1 mm thickness was applied to the prepared surface of samples. The coating composition included boron carbide  $\text{B}_4\text{C}$  and an organic binder.

## 2. Experimental details

Vanadium boride synthesis was conducted on the surface of D2 die steel. The samples were prepared by applying the coating to the prepared steel surface. The coating composition included mixtures of oxide  $\text{V}_2\text{O}_3$ , amorphous boron and carbon using 1:1 proportions and an organic binder – 1:10 BPH-6 solvent acetone adhesive.

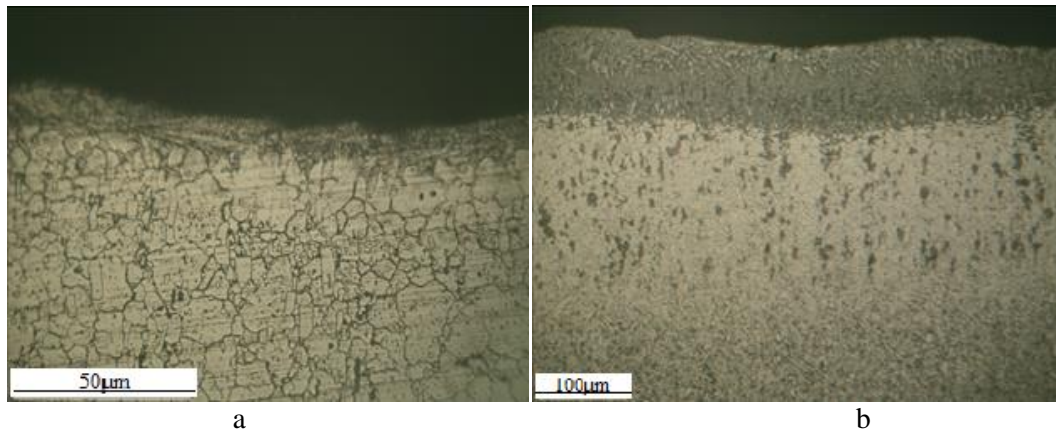
The samples were heated by means of electron beams using pulse conditions with the following parameters: accelerating voltage -  $U=24$  kV; beam current -  $I_b=63$  A. Processing was carried out with pulse length  $t=20$   $\mu\text{s}$ ; number of pulses  $N=1800$ ; beam current pulse repetition frequency  $f=6$  Hz. Vacuum vessel pressure:  $5 \times 10^{-2}$  Pa. [5,6]

Continuous electron beam heating for 2-5 minutes with specific capacity of 2-2.5 W/ $\text{cm}^2$ . Residual pressure in the vacuum vessel did not exceed  $2 \times 10^{-3}$  Pa.

The boride layers were analyzed by x-ray diffraction (Advance D8 Bruker, Cu  $K\alpha$ - radiation). The samples microstructure was observed using a metallographic microscopy METAM RB-21 (completed by digital chamber VEC-335 and program complex ImageExpert Pro 3.0) and scan electron microscopy LEO 1430 VP. Microhardness was measured by using PMT-3 microhardness tester at a loading 0.5 H [7].

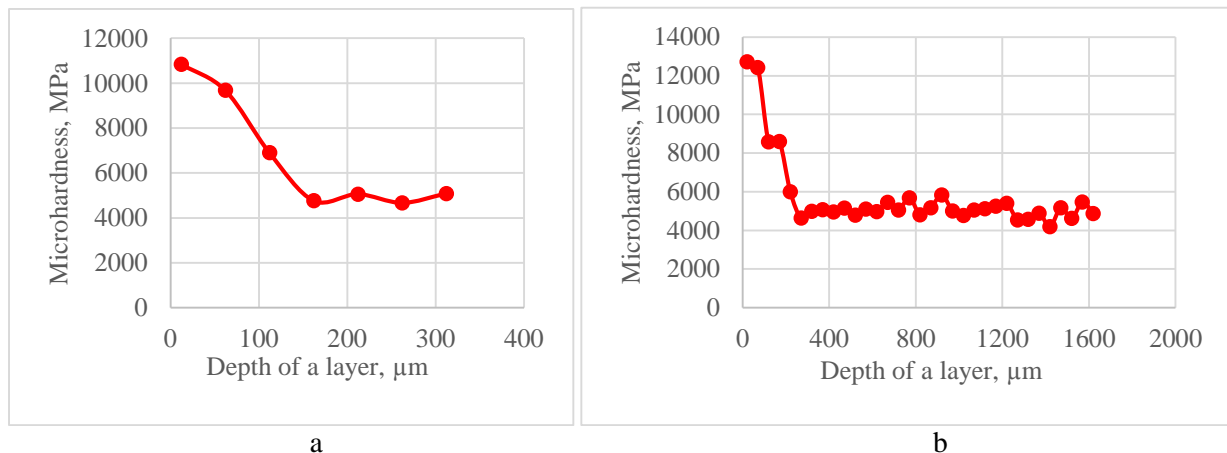
## 3. Results and their discussion

Metallographic analysis discovered that the structures of D2 steel surface layers obtained as a result of pulse and continuous electron beam borating (EBB) were different (Fig.1).



**Figure 1.** Structure of layer on the basis of  $\text{VB}_2$  on D2 steel: a – impulsive electron beam, b – continuous electron beam.

After applying EBB the transition zone is missing and there is a distinct visible border between the layer and main part of the steel. The layer is composed of rounded crystals located on the surface or throughout and eutectics (Fig.1). Layer thickness is 20-30 and up to 250-300  $\mu\text{m}$  after pulse and continuous electron beam bonding, respectively. Small layer thickness obtained after pulse electron beam bonding is attributable to the fact that the beam picks off the resulting products of self-propagating high-temperature synthesis before D2 steel surface melting.

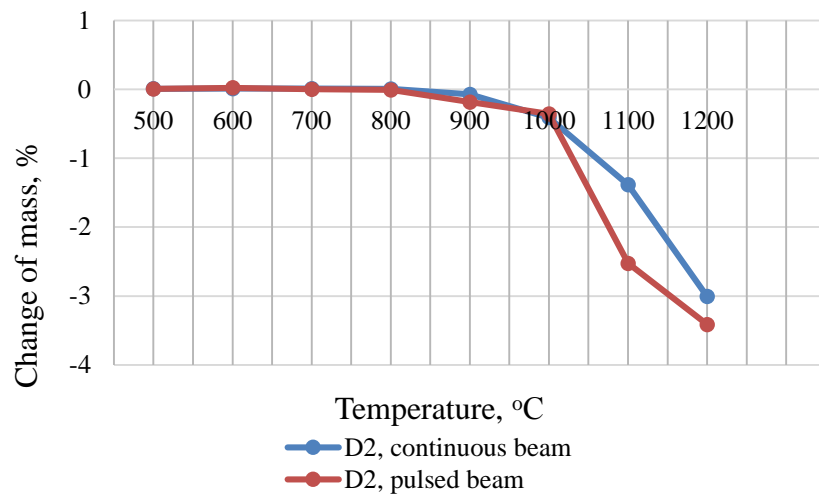


**Figure 2.**  $\text{VB}_2$  layer microhardness on D2 steel: a - pulse electron beam, b - continuous electron beam.

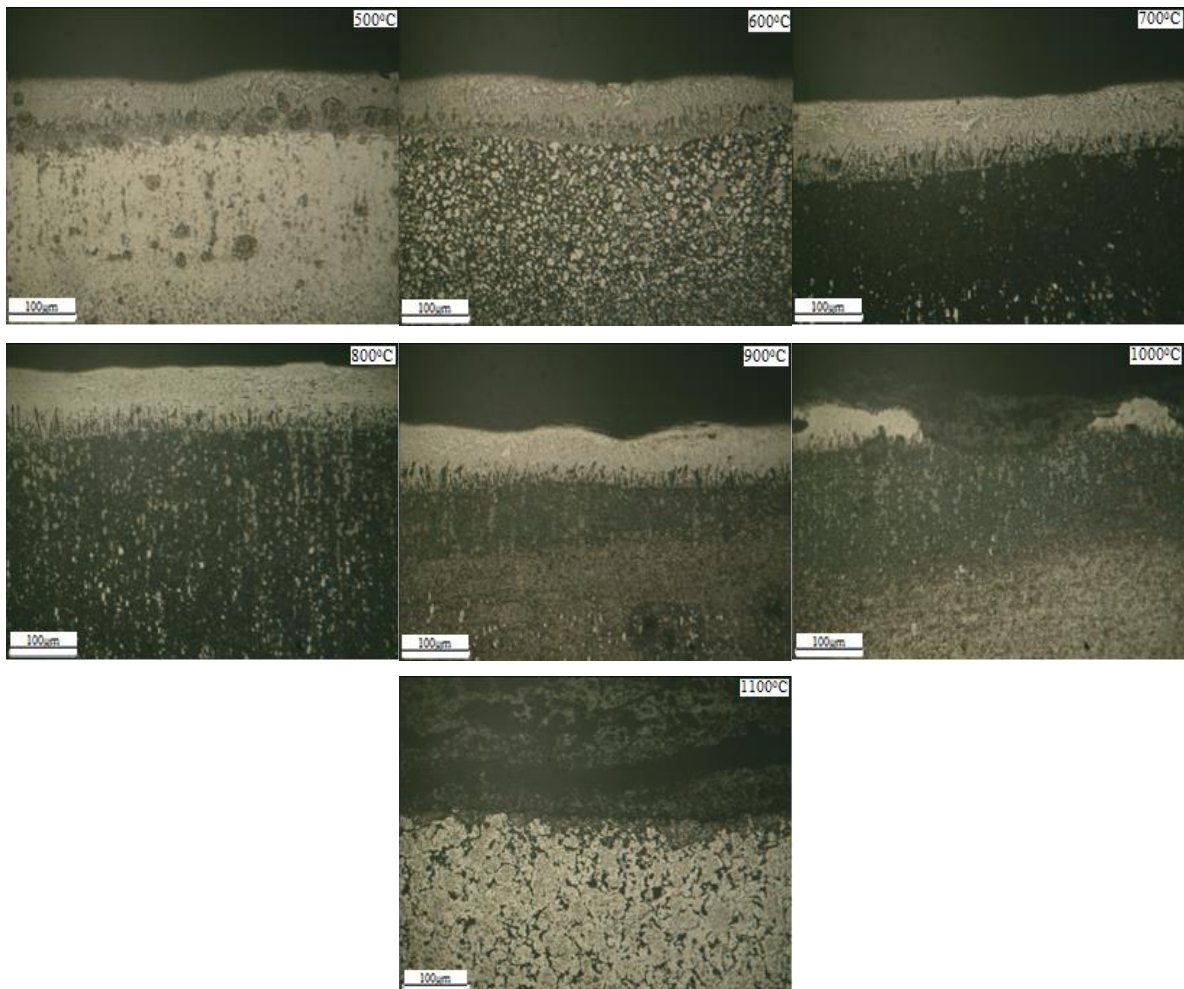
Measurement applied to the hardness of vanadium boride layers with 30-50  $\mu\text{m}$  spacing discovered its uneven distribution across the thickness (Fig. 2). However, every examined sample revealed regular microhardness distribution depending on layer thickness. Particular very rare inclusions have  $\text{HV} \approx 20000$  MPa and are located in near-surface layer zones. The layers are characterized with the most complex disordered structure. Increase in microhardness of the basis to  $\text{HV} \approx 5000$  MPa is attributable to the fact that it has been strengthened by using electron beams.

Boride layers were tested for thermal resistance and high-temperature strength. For this purpose all samples were heated inside a KO-14 resistance furnace until reaching particular temperatures and soaked for 2 hours to reach equilibrium.

Figure 3 shows a graph of weight variance with increase in temperature. It can be seen that when the temperature is increased to 900  $^{\circ}\text{C}$  and above the weight starts to decrease. This is because of layer oxidation and breakup which is supported by research of microstructure and microhardness (Fig. 4, 5).



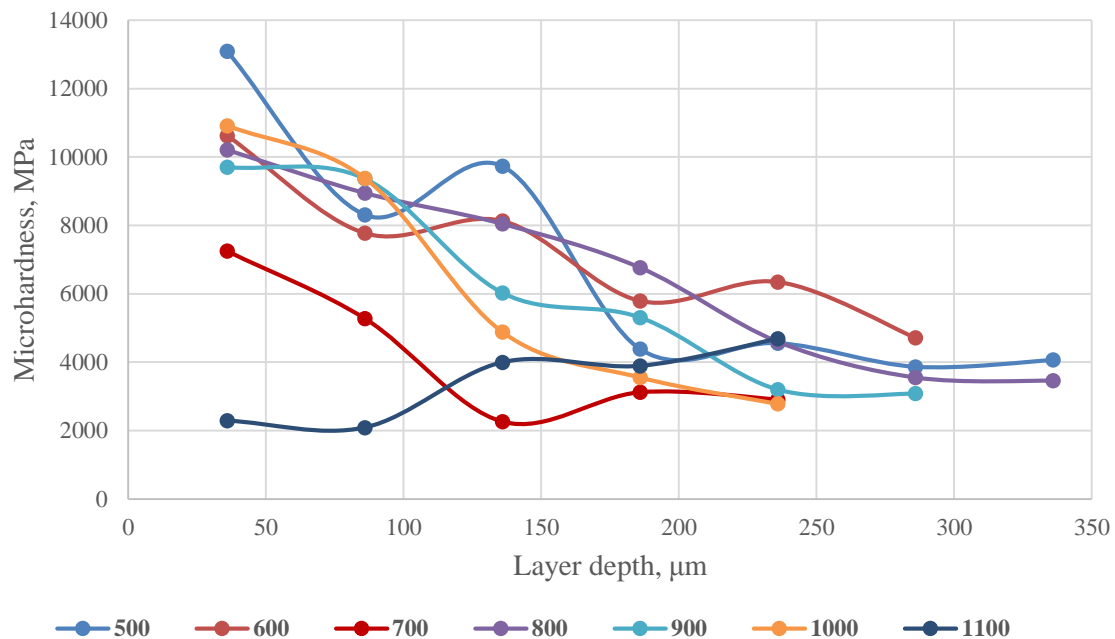
**Figure 3.** Change of mass with the height of temperature.



**Figure 4.** Thermal stability of boride layers after treatment a continuous bunch on D2 steel at heating on air.

The examination of microstructure shows that the thicknesses of boride layers obtained in both cases gradually start to decrease when the temperature is 700°C and above (Fig. 4).

Further rise of the heating temperature to 1100 °C in the open air results in a complete breakup of bonded layers (Fig. 4). This is most intensively observed in the layers formed during pulse beam borating, where the layer loses its weight and completely breaks down when temperatures exceed 900-1100 °C (Fig. 4).



**Figure 5.** Microhardness the boride of layers when heating on air.

The research allows to make a conclusion regarding EBB application for hardening cutting tools, etc. exposed to heat up to high temperatures during the operation without a significant reduction of performance properties.

It is known that alongside with high hardness and wear resistance the boride layers also have a significant disadvantage, i.e. high embrittlement. The conducted research showed that the use of electronic heating allowed to reduce embrittlement and to improve plasticity. After electron beam borating the layers become more plastic compared to after solid phase borating. Also, after electron beam borating the layers have a heterogeneous structure combining hard (brittle) and more plastic structural components. Such a combination partly explains the absence of thermal cracks during the heating of boride layers to high temperatures.

#### 4. Conclusion

1. Therefore, the electron-beam processing under vacuum resulted in the formation of vanadium boride layers which have an uneven structure across the thickness, contain different phases and as a result have an uneven distribution of physical-and-mechanical properties (for example, microhardness). Hard particles with 5-7 μm size are located in plastic eutectics. The layer surface has maximum values of microhardness.

2. The examination of microhardness of boride layers after pulse and continuous electron-beam borating allows to make a conclusion about the use of both borating methods for hardening cutting tools, etc. exposed to heat up to high temperatures during the operation without a significant reduction of performance properties.

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