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Properties and Structure of Sintered Boron Containing Carbon Steels

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1. Introduction

The iron-carbon alloys are the most wide-spread powder structural materials due to relative simplicity of technologies and accessibility of input materials for their fabrication (Dorofeev, 1986; Metals Handbook , 1978).

However, the need for increasing of sintered steels mechanical and operational characteristics level causes the need of their supplementary alloying. The basic alloying elements, which have found a wide application in a practice of powder metallurgy, are copper, nickel, manganese, chromium, vanadium and molybdenum (Zhang, 2004; Dorofeev, 1986).

Herewith, the significant capacities for manufacturing of low-alloy steels, which operational characteristic often are not inferior of the level of the same for steels, produced with application of traditional alloying modes, makes it possible the use of boron as basic alloying element (Dudrova, 1993; Gülsoy, 2004; Napara-Volgina, 2011; Suzuki, 2002). In our previous articles it has been demonstrated that boron addition to the base material is very efficient way for improving basic mechanical properties of sintered steels as well positive effect on activation of sintering was observed too.

It is well known that boron activates sintering of iron and steels because of formation of persistent liquid phase. In addition, boron has high affinity to oxygen. Thereby while using boron as one of the alloying elements for production of sintered steels improved mechanical properties with excellent combination of hardness and ductility could be obtained.

Furthermore it is necessary to take into account that solubility of boron in the iron is exceedingly small, so its addition in powder steels will cause the number of difficulties, that make it necessary to optimize just but one boron composition in the sintered alloy, but the method of its addition in the initial powder mixture as well. The most often used method of boron addition is based on application of boron carbide powder as boron containing element. But sintering of Fe-B$_4$C powder mixtures is attended with formation of derivative porosity in consequence of dissolution of boron carbide particles in a contact with iron matrix. To eliminate the noted effect as boron containing additive Fe-B-C system master alloy can be used (Napara-Volgina, 2008; Xiu, 1999)..

So, in order to evaluate the ability of boron to activate sintering of the base powder preforms with aim of increasing of the mechanical properties and wear resistance, study was carried

on influence of boron composition and modes of its addition in the initial powder mixture on basic mechanical and tribotechnical characteristics of sintered steel.

2. Effect of powder mixture content and modes of alloy addition on the structure and properties of sintered steels

In (Kazior, 2002) the authors had studied the influence of boron addition in different forms (elemental boron powder, boron carbide B_4C powder, and mixture of boron and carbide elemental powders) and different weight percentages on density and mechanical properties of prealloyed Astaloy CrM powders alloys.

The specimens were prepared by mixing of commercial water atomized Astaloy CrM powder with above-mentioned boron containing powders and were compacted at a pressure of 600 MPa to obtain an average density of 6.9 g/cm³. The green parts were sintered at 1250 °C for 30 min. in laboratory furnace in pure dry hydrogen.

The obtained results had shown, that the additive of boron caused densification, but the effect depends on the amount and form of boron additives. For 0,2 (wt.) % B only for boron carbide small increase in density can be observed, whereas for the other modes of boron addition density is of the same level as for boron-free sintered steel (fig. 1). A significant increase in sintered density was obtained with higher content of boron (0.4 wt. %), reaching 7.44 g/cm³ for iron–boron carbide powders mixture.
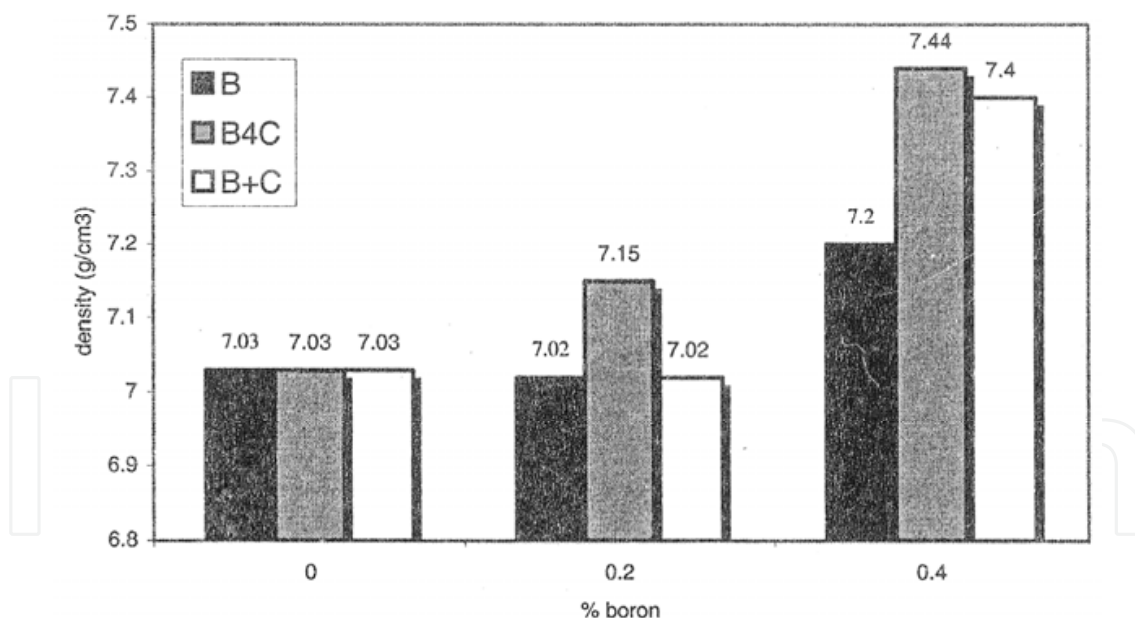

Fig. 1. Sintered density as a function of boron content and mode of its addition (Kazior, 2002).

Figure 2 shows the pore morphology of the boron-free and with 0.4 (wt.) % B_4C sintered alloys. It is evident, that boron addition considerably changed the pore morphology: while the boron-free sintered alloy has in its structure the irregular pores, typical for solid phase sintering (fig. 2, a), otherwise the boron containing sample has the large well rounded pores, which are typically formed during liquid phase sintering (fig. 2, b).
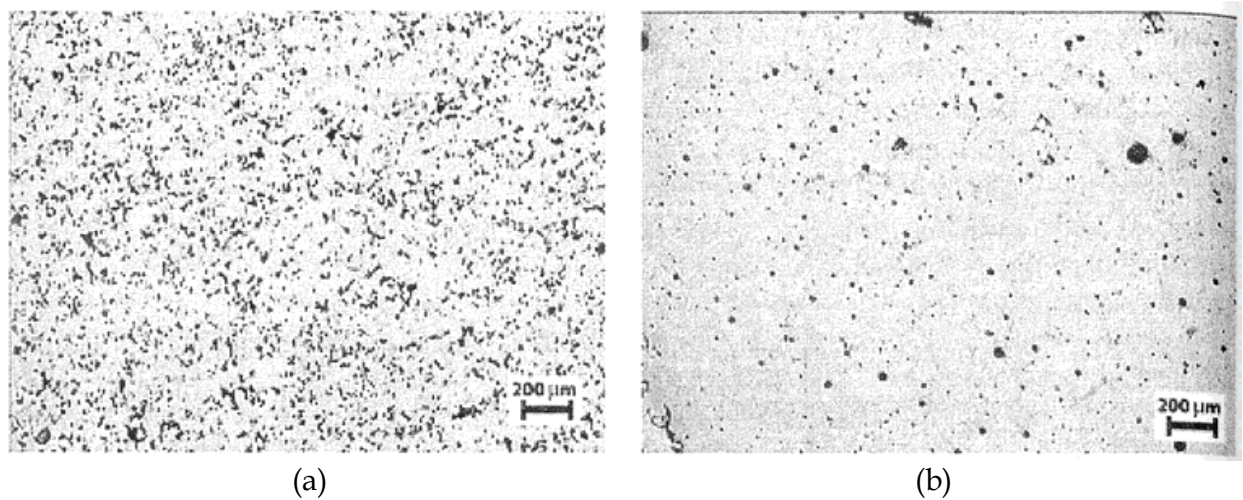
(a)   (b)

Fig. 2. Optical micrograph of unetched boron-free (a) and with 0.4 (wt.) % boron (B_4C) alloyed sintered Astaloy CrM.

The unalloyed sintered Astaloy CrM has a homogeneous ferritic microstructure (fig. 3,a). The microhardness of the ferritic grains is 150-170 $HV_{0,02}$. The representative microstructure of the boron alloyed sintered specimens is shown in figure 3,b. Boron causes hardening of the matrix, and its microhardness is very sensitive of the form of boron. For elemental boron with increasing boron content the microhardness of matrix ranges between 270-300 and 330-370 $HV_{0,02}$ respectively. For B_4C with increasing boron content the microhardness of matrix ranges 330-370 and 400-470 $HV_{0.02}$ respectively. Beside, as a consequence of the eutectic reaction between austenite matrix and borides, the microstructure contains second constituent, which forms for higher boron content a continuous network on the grain boundaries. Figure 3,b shows the typical representative morphology of the second constituent, observed in 0.4 wt % boron (B_4C) alloyed sintered specimen. Two different borides phases were found in the second constituent. The gray component is the Fe-Cr rich boride, while the white part of the component is the Fe-Mo rich boride.
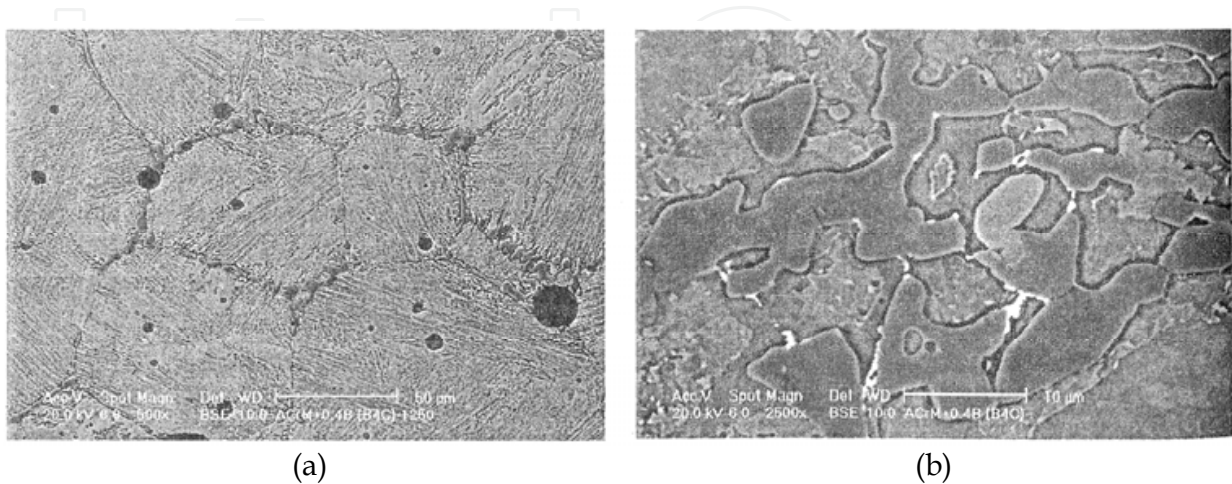

(a)   (b)

Fig. 3. SEM micrograph of 0.4 (wt.) % boron (B4C) alloyed sintered Astaloy CrM. ×500 (a); ×2500 (b).

Table 1 summarizes the effect of boron on mechanical properties of the studied materials. The general trend is that, the higher boron content, the higher are the yield, strength, hardness and lower elongation of course. However, the results indicate that form of boron has significant effect on the tensile properties. For alloyed sintered specimens, both by boron carbide or elemental boron and graphite mixture, the microhardness of the matrix and in consequences tensile properties are higher as compared with elemental boron additive. Furthermore, it is clear that the tensile properties are not simply related to density. Boron causes both hardening of the matrix and densification, but the prevailing effect on properties is hardening. For example for 0.2 (wt.) % boron contents, despite of the increase in tensile strength, density does not increase significantly.

The above considerations are confirmed by fracture surface analyses. Figure 4 shows the representative fracture surface of tensile specimens of unalloyed and 0.4 (wt.) % boron (B4C) alloyed specimens, respectively. The fracture surface of the unalloyed material (fig. 3,a) presents the typical fracture morphology of a sintered material with interconnected porosity, while 0.4 (wt.) % boron (B_4C) alloyed material fracture surface has a brittle character and crack propagation occurs along the eutectic constituent (fig. 3,b).

Material	0.2 % offset yield stress (MPa)	MTS (MPa)	Elongation (%)	Hardness HV 10
Astaloy CrM	157	287	4.36	182
Astaloy CrM + 0.2 % B	463	613	1.99	187
Astaloy CrM + 0.4 % B	515	688	1.43	247
Astaloy CrM + 0.2 % B (B_4C)	509	666	1.74	272
Astaloy CrM + 0.4 % B (B_4C)	592	741	1.27	302
Astaloy CrM + 0.2 % B (B_4+C)	515	725	1.76	268
Astaloy CrM + 0.4 % B (B_4+C)	662	806	1.57	320

Table 1. Mechanical properties of boron-free and boron alloyed sintered Astaloy CrM (Kazior, 2002).

The research of structure formation at sintering of Fe - B_4C powder mixtures had shown, that active interaction of the components takes place already at temperatures of 850÷900 ^{0}C with generation of Fe_2B and FeB (at ~1100 ^{0}C) phases (Turov, 1991). During heating of powder mixture yet before the beginning of diffusion of the boron to iron matrix contact surface of B_4C particle with iron diminishes owing to considerable difference of their coefficients of thermal expansion (fig. 5,a). Despite of that, at 900÷950 ^{0}C it is beginning the formation of the new phases in neighborhood of boron carbide particles (fig. 5,b). This phase has a typical for boride layers needle-shaped structure. The X-ray analysis of insoluble residue, which was isolated by means of electrolytic etching, has shown the presence in a structure of the material of FeB and Fe_2B phases with microhardness of 14÷18 GPa.
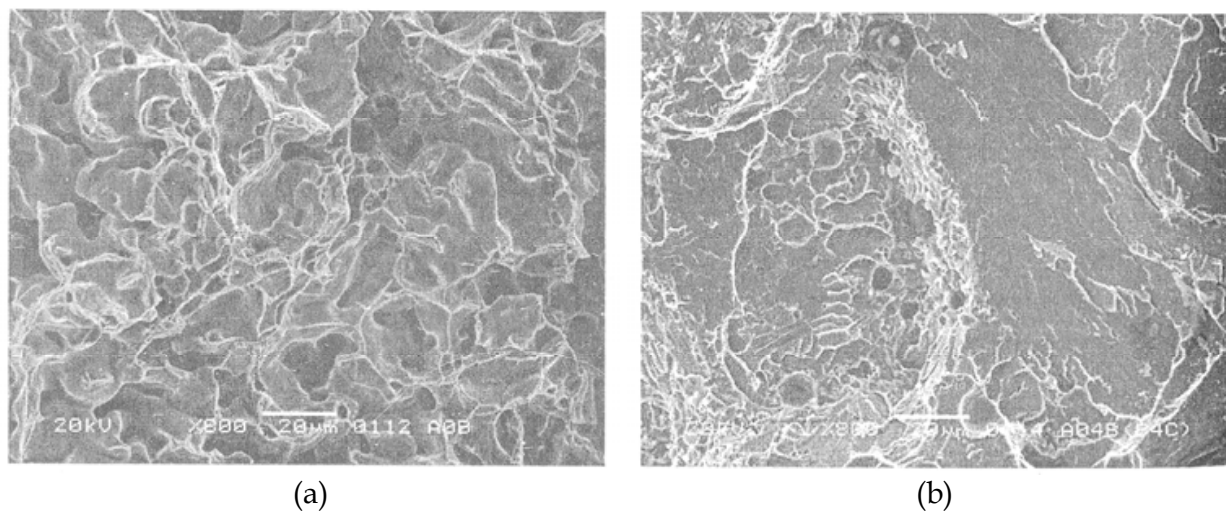
(a)   (b)

Fig. 4. Fracture surface of unalloyed (a) and 0.4 % boron alloyed (b) sintered Astaloy CrM.
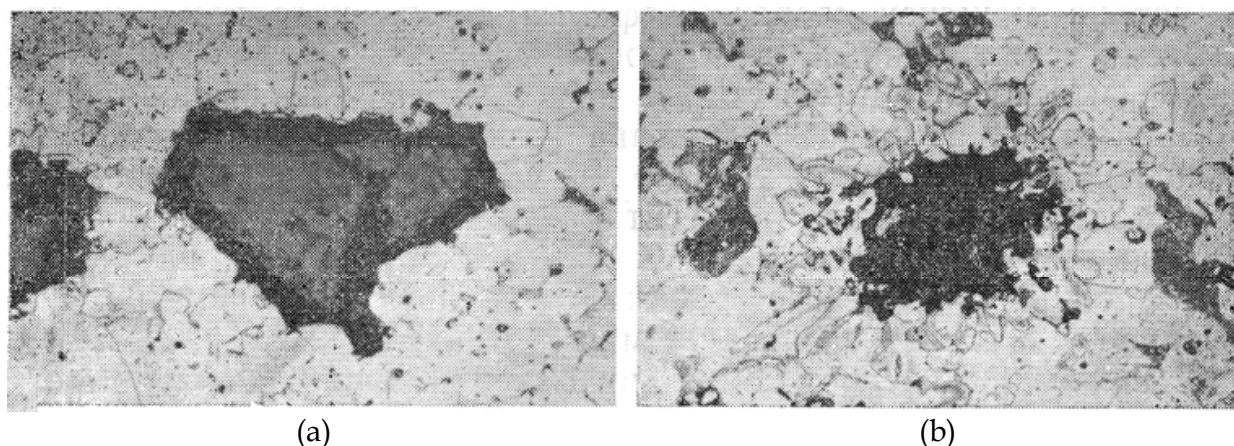

(a)   (b)

Fig. 5. Optical micrograph of sintered at 900 ^{0}C for 10 (a) and 120 (b) min.
Fe+3 % (wt.) B$_4$C powder mixture.

With increasing of sintering temperature to 1050 ^{0}C around B$_4$C particle the new phase appears, which composition was identified by microX-ray spectrum analysis (fig. 6) as boron cementite with ~75 % (atomic weight) Fe, ~15 % C and ~10 % B.

The above presented results testify, that boron carbide is the compound, that is easily dissociates in a contact with iron at relatively low temperatures. Herewith B$_4$C is a source of atomic boron and carbon, which are reacting with iron with establishing of new strengthening phases. Boron carbide is a sintering activator as well. However, thereupon that B$_4$C is the compound relatively poor with carbon, the opportunity of supplementary strengthening of Fe-B$_4$C system by additive of carbon powder probably exists.

To evaluate both boron and carbon content in the initial powder mixture on structure and properties of sintered steel mixtures were prepared by mixing of water atomized pure iron powder with particles size smaller than 160 μm, 1÷3 % (weight) commercial boron carbide and 0,5÷2,0 % graphite powders.

Powders were compacted at a pressure of 800 MPa to obtain cylindrical specimens with ⌀10 and h≈10÷12 mm. The green parts were sintered at 1100÷1200 ⁰C for 60 min. in the container with fusible glass bath gate (Fedorchenko, 1972). To estimate the influence of carbon on properties of the material the same specimens were prepared with boron carbide but without graphite additive. After sintering some specimens were quenched from 1000 ⁰C in a water with subsequent tempering at 250 ⁰C.

Density, hardness and compression strength of all specimens were examined. For the purpose of determining changes of structure and identifying phases, structural investigations by optical microscopy and phases microhardness estimations were carried out.

Figure 7 shows the influence of graphite content in the powder Fe-B_4C mixtures on porosity and hardness of sintered at 1150 ⁰C materials. As it can be seen from fig. 7,a porosity of compositions rises with increase of graphite content, and very considerably for Fe-3 % B_4C in consequence of worse powder compressibility at compaction of B_4C rich mixtures.
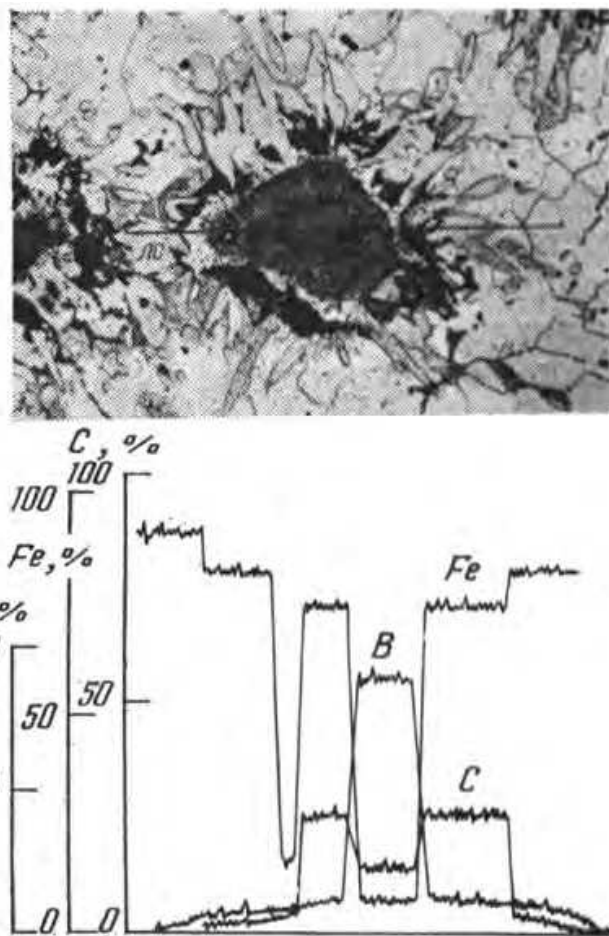

Fig. 6. Microstructure of sintered at 1050 ⁰C Fe+3 % (wt.) B_4C steel and elements distribution in the area of B_4C particle.

Hardness of the sintered materials with 1 and 3 % of B_4C with addition of graphite depends on two factors: values of their porosity and microstructure nature. So, with rise of B_4C and graphite content hardness of sintered steels increase for all mixture compositions except of

Fe-3 % B$_4$C, which hardness for mixtures with 1.5 and 2.0 % additive graphite considerably decreases (fig. 7,b, plot 3) owing to respective increase of specimens porosity (fig. 7,a).

The investigation of structure of sintered at relatively low temperatures (1100 ^{0}C) materials had shown, that for all steel compositions on a basis of ferrite-pearlite matrix structures contain multiple isolated inclusions, located in pores, that correspond to the configuration of boron carbide particles (fig. 8,a). Herewith, quantity of such inclusions increases with rise of boron carbide content in the mixture.
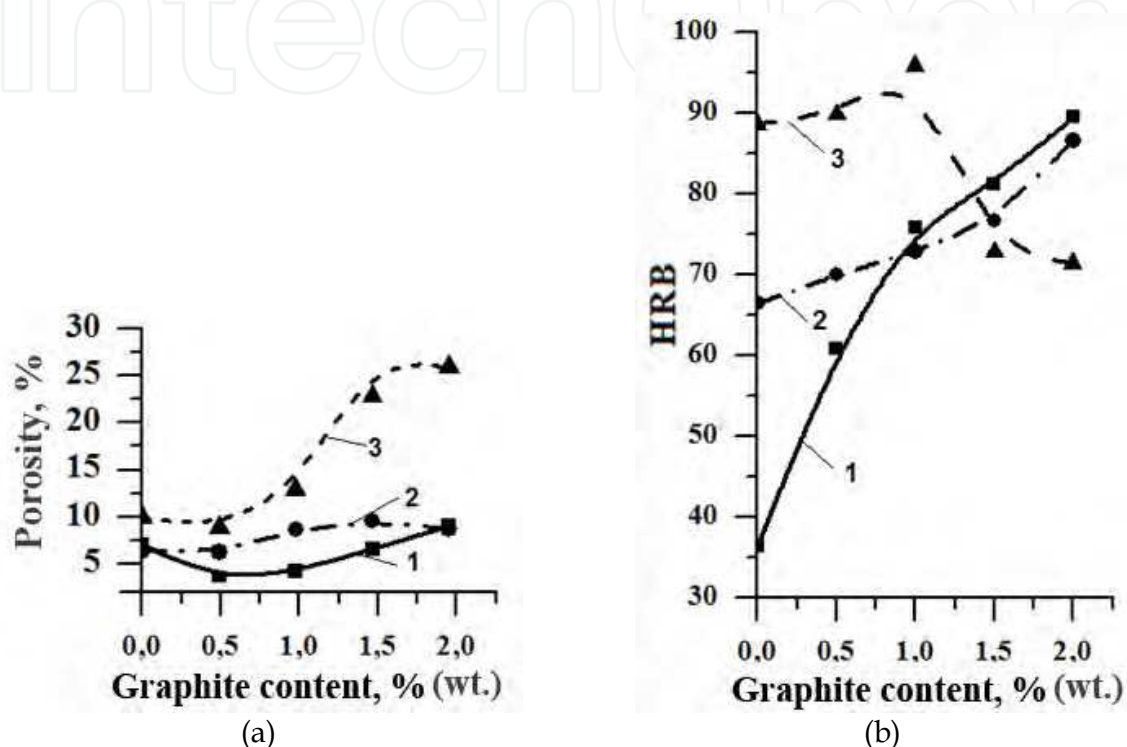
Fig. 7. Effect of graphite content on porosity (a) and hardness (b) of sintered at 1150 ^{0}C steels: 1 – boron-free; 2 – 1% B$_4$C; 3 – 3 % B$_4$C.

Rise of sintering temperature up to 1150÷1200 ^{0}C is attended by dissolution of carbon in the matrix, formation of Fe$_2$B + γ eutectic and severable healing of pores with inclusions and, as a consequence of this effect, alteration of structure (fig. 8, c-f). After sintering at 1150 ^{0}C material structure consist of substantially pearlite with trace of abnormal inclusions and isolated eutectic deposits (fig. 8, c), while after 1200 ^{0}C - pearlite inclusions on a basis of eutectic and separate pearlite areas with cementite net (fig. 8,e).

With rise of carbon content the microhardness and amount of eutectic in sintered material increase. So, at enhancing of graphite content in a mixture from 0.5 to 2.0 % the eutectic microhardness increased from 7.4 to 9.2 GPa, while its amount – from 50 to 80 %.

After sintering of all examined materials at 1150 ^{0}C some free carbon remain in their structure; its amount rises from 0.07 to 0.39 % in proportion to graphite content in the initial mixture.

Thermal treatment of the sintered materials results in essential advance of hardness, which values for the steels with 1 % B$_4$C acquire 35÷40 HRC and with 3 % B$_4$C - 50÷56 HRC, while for the boron-free specimens – from 25÷27 to 40÷43 HRC (subjected to carbon content) (fig. 9).

Investigation of sintered and heat treated materials compression strength had shown, that ultimate values of durability have the steels with 1 % B_4C and 1÷1.5 % additive carbon (fig. 10, plot 1). The least strength is for materials with 3 % B_4C and carbon content more than 1.0 % (fig. 10, plots 4 and 4a). That kind of compositions are characterized by fragile nature of destruction, that can be explained by relatively high porosity (see fig. 7,a) and presence of significant amount of fragile eutectic.
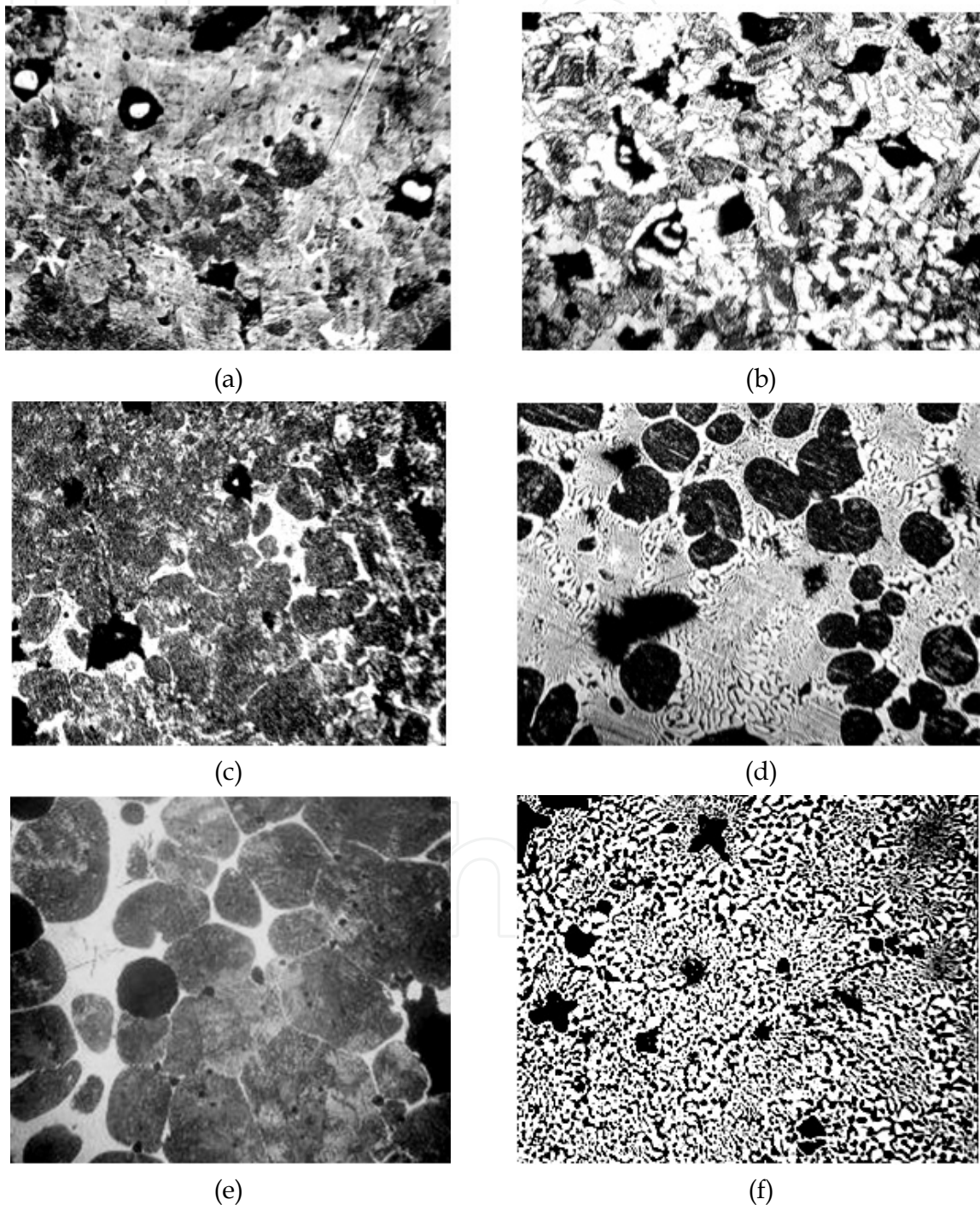

Fig. 8. Structure of the materials sintered at 1100 (a, b); 1150 (c, d, f) and 1200 ^{0}C (e); composition of mixtures: Fe + 1 % B_4C + 2 % C (a, c, e); Fe + 3% B_4C + 2 % C (b, d, f).
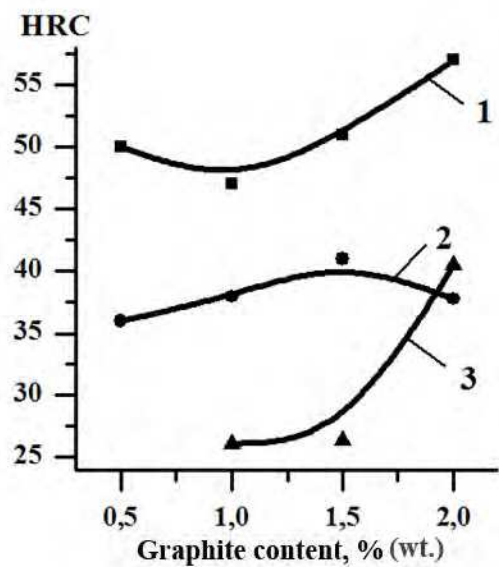
Fig. 9. Effect of graphite content on HRC hardness of sintered and thermal treated steels:
1 – Fe + 3 % B_4C; 2 – Fe + 1,5 % B_4C; 3 – boron-free Fe.
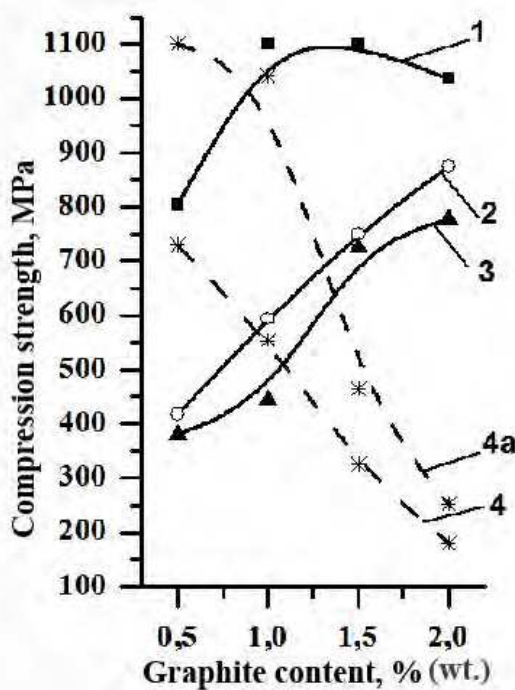

Fig. 10. Effect of graphite content on compression strength of sintered (1-4) and thermal treated (4a) steels with 1 % B_4C (1), 3 % B_4C (4, 4a) and boron-free Fe (2,3). Sintering temperature: 1, 2 – 1200 ^{0}C; 3, 4 – 1150 ^{0}C.

3. Application of boron containing master alloys for manufacture of sintered steels

As it can be seen from the above presented results, sintering of Fe-B_4C powder mixtures is attended with formation of derivative porosity in consequence of dissolution of boron

carbide particles in a contact with iron matrix. To eliminate the noted effect as boron containing additive Fe-B-C system master alloy can be used. The last one can increase the boron uniformity in the volume of sintered steels too, that probably result in improvement of their mechanical properties.

For estimation of the proposed approach several routes of boron addition to a powder mixture on the amount from 0,3 to 2,4 % and some mechanical properties of the sintered steels had been investigated. As boron-containing components boron carbide, amorphous boron of high purity and Fe-B-C system master alloy, produced by thermal synthesis from Fe and B4C powder mixtures, were used. All of the noted boron-containing components were mixed with water atomized iron powder with maximal particles size of 160 μm. Samples of ⌀10×(10÷12) mm size were compacted by uniaxial die pressing at 800 MPa and sintered at 1100, 1150 and 1200 °C for 1,5 h in the container with fusible glass bath gate.

Three types of powder master alloys chemical compositions (table 2) were used for estimation of sintered steels mechanical properties. Boron content in the powder mixtures for the samples manufacturing was 0,4; 0,8; 1,2 and 1,6 % (weight) for every mode of boron addition (table 2).

Sintered iron samples with 0,5; 1,0; 1,5 and 2,0% boron carbide additives, that provided the same total boron in powder mixtures, were used as the reference ones.

Some sintered specimens were heat treated, i.e. water quenched from 1000 °C with subsequent tempering at 250 °C over a period of 3 hours.

No.	Modes of boron addition	Boron content, % (weight)	Carbon content, % (weight)
1	Master alloy	3.6	1.7
2	Master alloy	5.9	2.3
3	Master alloy	8.7	3.5
4	B_4C	80.0	20.0

Table 2. Chemical composition of master alloys and boron carbide, used for manufacturing of sintered steels.

Values of die pressed green compacts and sintered at 1200 °C preforms porosity for sintered steels, produced from different raw materials (powder mixtures with master alloy, B4C and amorphous boron powders) with different boron contents were investigated (fig. 11). It can be seen, that with increasing of boron concentration in the powder mixture preform green density decreased for all kinds of boron-containing components.

Maximal porosity values were observed for the green compacts, produced from master alloy containing powder mixtures (fig. 11, curve 3). It can be explained by significant its amount in the source mixture in comparison with other boron containing components and, owing to that, deterioration of such mixtures compressibility.

No.	Mixture composition, %			Carbon content in a mixture, %	Boron content in a mixture, %
	Master alloy		Content of B$_4$C in a mixture, %		
	No. of master alloy (from table.1)	Content of master alloy in a mixture, %			
I	1	11,0	-	0,19	0,4
	2	6,8	-	0,17	
	3	4,6	-	0,16	
	4	-	0,5	0,10	
II	1	22,2	-	0,37	0,8
	2	13,6	-	0,35	
	3	9,2	-	0,32	
	4	-	1,0	0,20	
III	1	33.3	-	0,56	1,2
	2	20,3	-	0,52	
	3	13,7	-	0,48	
	4	-	1,5	0,30	
IV	1	44,4	-	0,76	1,6
	2	27,1	-	0,69	
	3	18,4	-	0,64	
	4	-	2,0	0,40	

Table 3. Composition of powder mixtures, used for manufacturing of boron containing sintered steels.
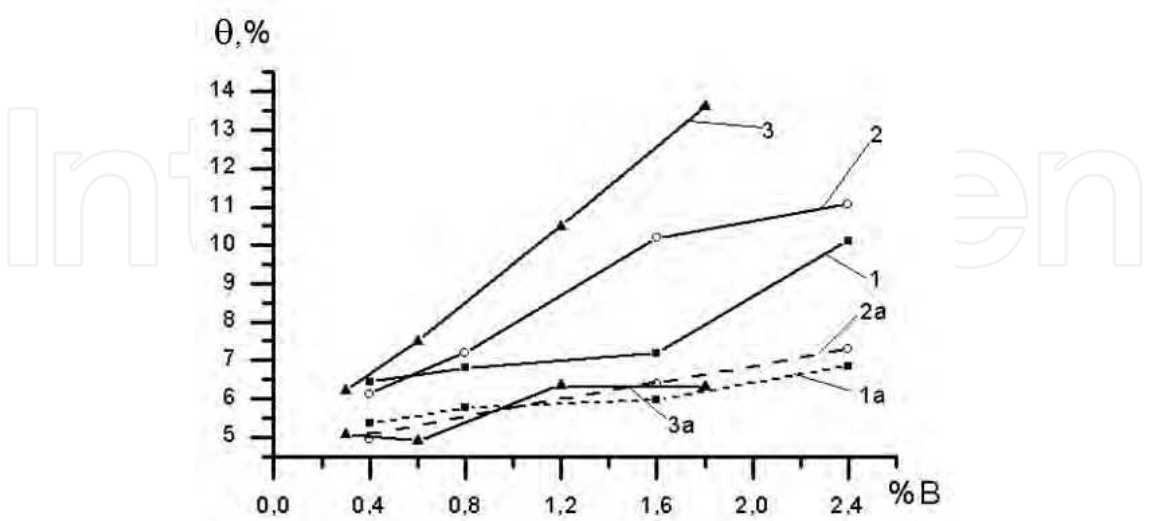
Fig. 11. Effect of boron content on porosity θ of green compacts (1, 2, 3) and sintered at 1200 ^{0}C preforms (1a, 2a, 3a) for different modes of his addition: B$_4$C (1, 1a); amorphous boron (2, 2a); master alloy with 6% of B (3, 3a).

However, after sintering at 1200 ⁰C porosity of the samples, which contains master alloy admixtures, don't differ virtually from that, produced with use of the other modes of boron addition, since sintering of master alloy containing powders is accompanied by noticeable increased value of shrinkage. The denoted data are confirmed as well by phenomenon of shrinkage of the samples with master alloy for all sintering temperatures, unlike the materials with other boron containing components in the initial powder mixture, which are characterized by dilatation after sintering (fig. 12). The latter phenomenon is explained by the effect of boron carbide high temperature dissociation in contact with iron and high reaction activity of amorphous boron at process of sintering accompanied by formation of nonhealing "derivative" porosity in the areas of their location.
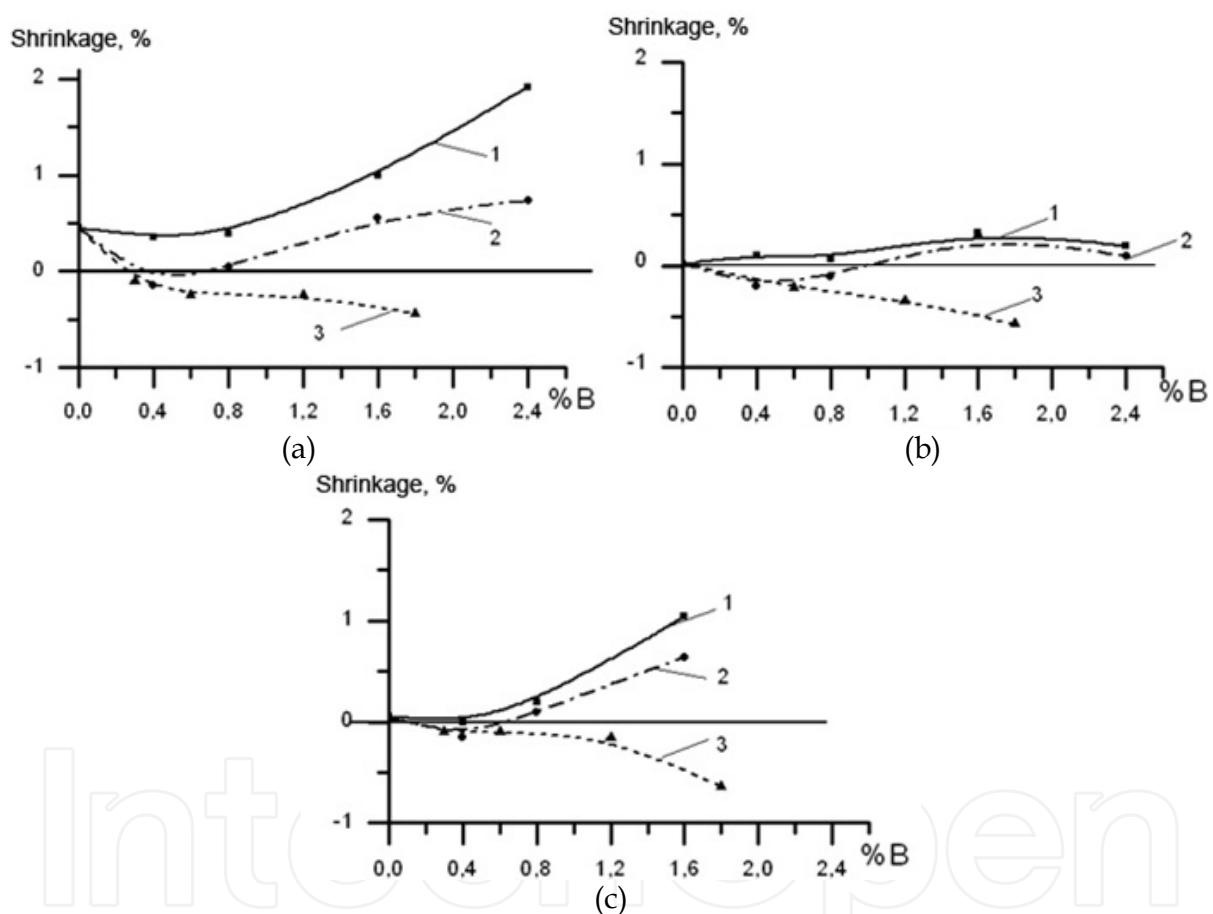
Fig. 12. Effect of boron content on linear shrinkage of billets after sintering at 1100 ⁰C (a), 1150 ⁰C (b) and 1200 ⁰C (c) for different modes of boron insertion: B4C (1); amorphous boron (2); master alloy with 6 % of B (3).

After sintering of powder preforms with boron carbide, the last easily dissociates in contact with iron at relatively low temperatures and is a source of monatomic boron and carbon, which generate at interaction with Fe hard compounds, that reinforce the sintered metal. After sintering at 1050 ⁰C it can be seen in the microstructure light-coloured and relatively hard (about 14 GPa) phase, located in the vicinity of incompletely dissociated boron carbide particles (fig. 13,a). Similar phase can be seen also after sintering at 1150-1200 ⁰C, but its

morphology is different: it is situated along the grain boundaries, shaping the developed framework (fig. 13, b), which can appear only as a result of liquid phase appearance in consequence of eutectic contact melting.

According to X-ray micrography data, the composition of interface region, which appears around the boron carbide particle, is similar to boron cementite composition (about 75 % of Fe, 15 % C and 10 % B).

The feature of structure of sintered steels, manufactured from powder mixtures with boron carbide, unlike those of steels, made with use of master alloy, is the availability of diffusion porosity, generated owing to dissociation of boron carbide particles (fig. 14).

Mechanical properties testing results of sintered steels, produced from powder mixtures with different types of master alloys and boron carbide (table 2), had shown, that materials with 0.8-1.2 % B, produced from powder mixtures with low-alloy additives, provided higher values of hardness, than that, made with use of boron carbide as boron containing additives after both sintering and thermal treatment (fig. 15, plots 1 and 2).
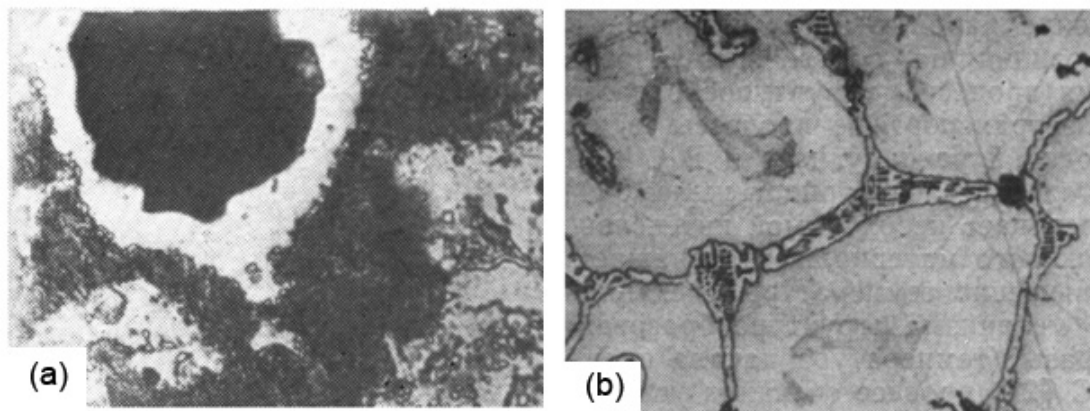

Fig. 13. Microstructure of steels with 1.0 % B_4C sintered at 1050 (a) and 1200 ^{0}C (b).

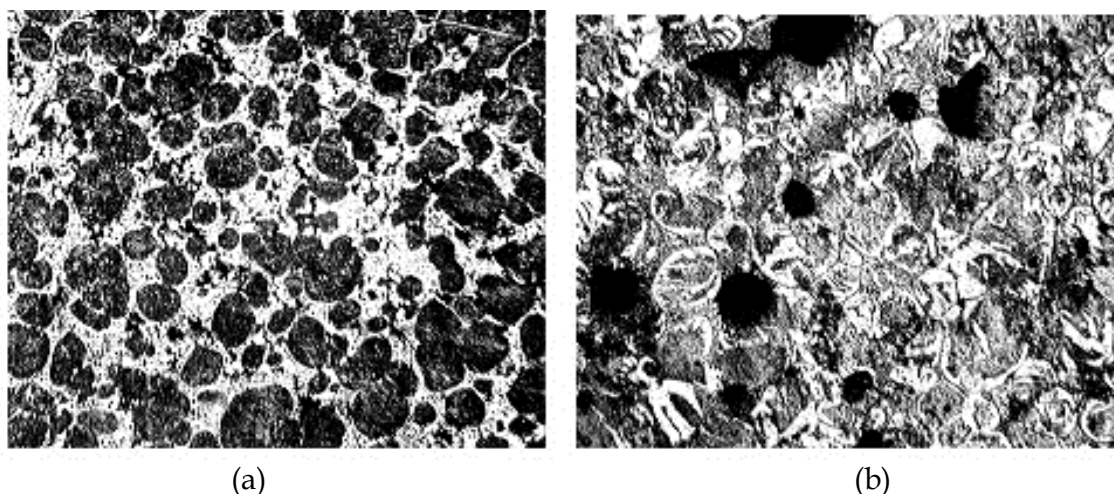(a)   (b)

Fig. 14. Microstructure of sintered steels with 1,2 % B after sintering at 1150 ^{0}C, manufactured with application of master alloy (Fe - 5,3 % B - 2,3 % C) (a) and boron carbide (b); ×150.

Similar behavior for relation of compression strength with boron content for different modes of boron addition in the powder mixtures (fig. 16) had been registered.

Indeed, hardness values of the same materials, produced with application of master alloy with 8,7 % B and boron carbide (fig. 15, plots 3 and 4) are considerably lower than that, produced with low-boron master alloy.
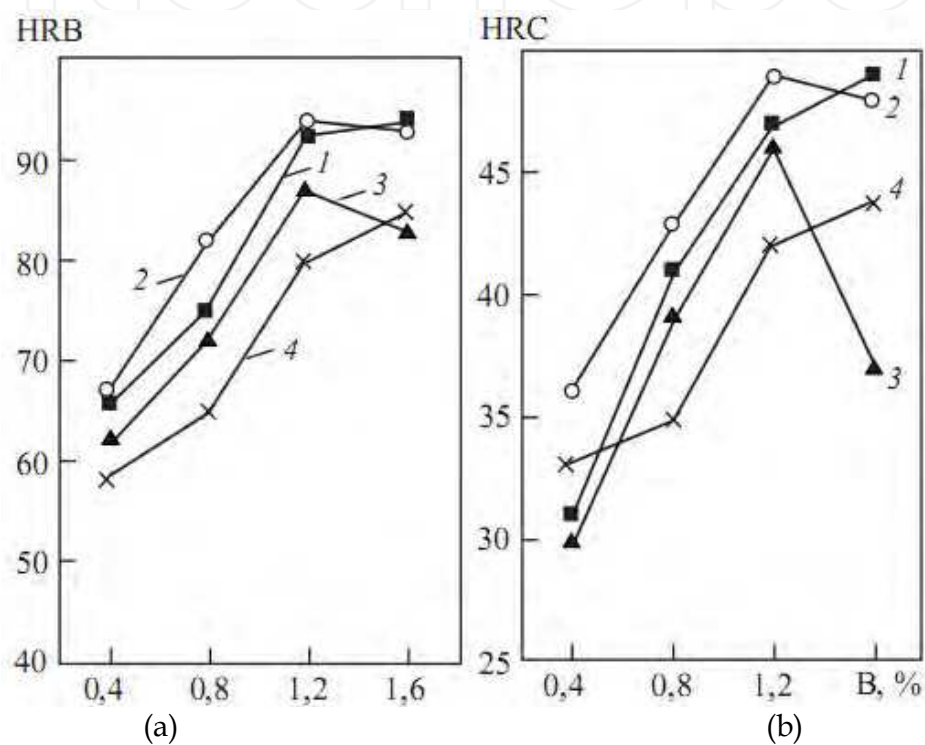

Fig. 15. Effect of boron content on hardness of sintered at 1150 ^{0}C (a) and heat-treated (b) steels, manufactured with use of master alloys of three compositions (No. 1–3, table 2) and boron carbide (4).

This difference in the mechanical properties of sintered steels with different kinds of boron source can be explained by more uniform distribution of low-boron containing elements in a balk of material, particularly – of eutectic constituent, in case of employment of master alloys in the source powder mixtures and higher content of carbon in the master alloy as compared with boron carbide.

The drawn conclusions were confirmed by the results of sintered steels metallographic study, which had shown, that microhardness of structural components in the materials, manufactured with use of master alloy with 8.7 % B4C (no. 3, table 2) is somewhat lower than that, produced with low-boron containing master alloys (No. 1 and 2) with 3.6 and 5.8 % of B.

So, for example, in a steel, sintered at 1150 ^{0}C with use of master alloy with 8.7 % of B, microhardness of eutectic is in the range of 6.3÷7.7 GPa, whereas the same for a steel, produced with master alloy with 5.3 % B – 8.58÷9.56 GPa.

Similar relationship can be seen too for microhardness of base material for steels, produced with use of master alloys.
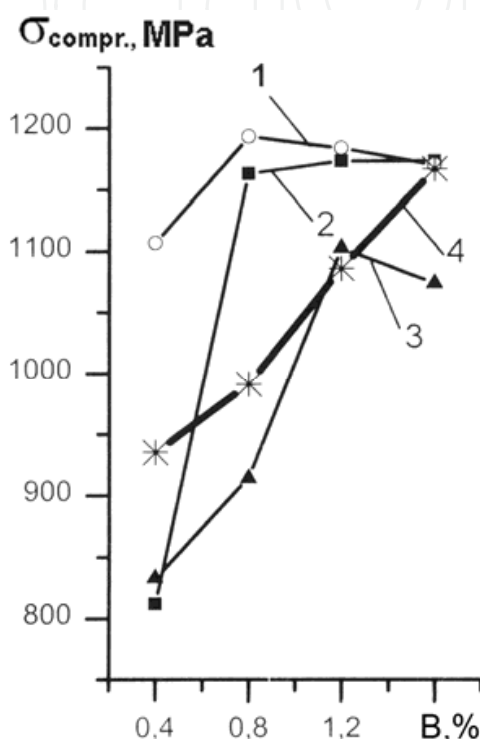

Fig. 16. Effect of boron content on compression strength for steels sintered at 1080 ^{0}C, manufactured with use of master alloys of different compositions
(No. 1–3, table 2) and boron carbide (4).

The wear tests of the sintered steels with 1.2 % B (table 4) were performed in a pin on disc test bed in conditions of unlubricated friction with utilization of step loading method at loads 10-40 N/cm^2 and sliding speed 5 m/s in air. As opposite element 65Mn steel with hardness 45 HRC had been used, while as a pattern material - 100Cr6 bearing steel (HRC 52). Evaluation for magnitude of wear was carried out by assessment of weight loss after every stage of testing and translation the data into μm/km.

Wear testing results for three compositions of sintered steels are presented on fig. 17. It can be seen, that wear resistance for all kinds of sintered boron steels considerably exceeds that for 100Cr6 cast and rolled bearing steel. However, in spite of sufficiently near compositions of boron and carbon in the sintered steels under test, wear resistance of materials, manufactured with use of master alloys with increased composition of boron carbide (No. 2

and 3) at moderate loads (50 and 75 N/cm²) considerably exceeds the same for the steels, synthesized with relatively "poor" master alloy (No. 1).

No.	Composition of master alloy	Composition of B and C in steels, %			
		B	C	B+C	B/C
1	3,6 % B + 1,7 % C	1,2	0,86	2,06	1,4
2	5,9 % B + 2,3 % C	1,2	0,82	2,02	1,5
3	8,7 % B + 3,5 % C	1,2	0,78	1,98	1,5

Table 4. Compositions of sintered and heat-treated boron steels for wear testing.

With increasing of loads to 100 N/cm² wear characteristics for sintered steels of all compositions are flattening.

High wear resistance of boron sintered steels as compared with 100Cr6 bearing steel can be explained by their heterophase structure and, particularly, by presence of hard eutectic component, which amount growth with increase of boron and carbon composition in the source powder mixture.
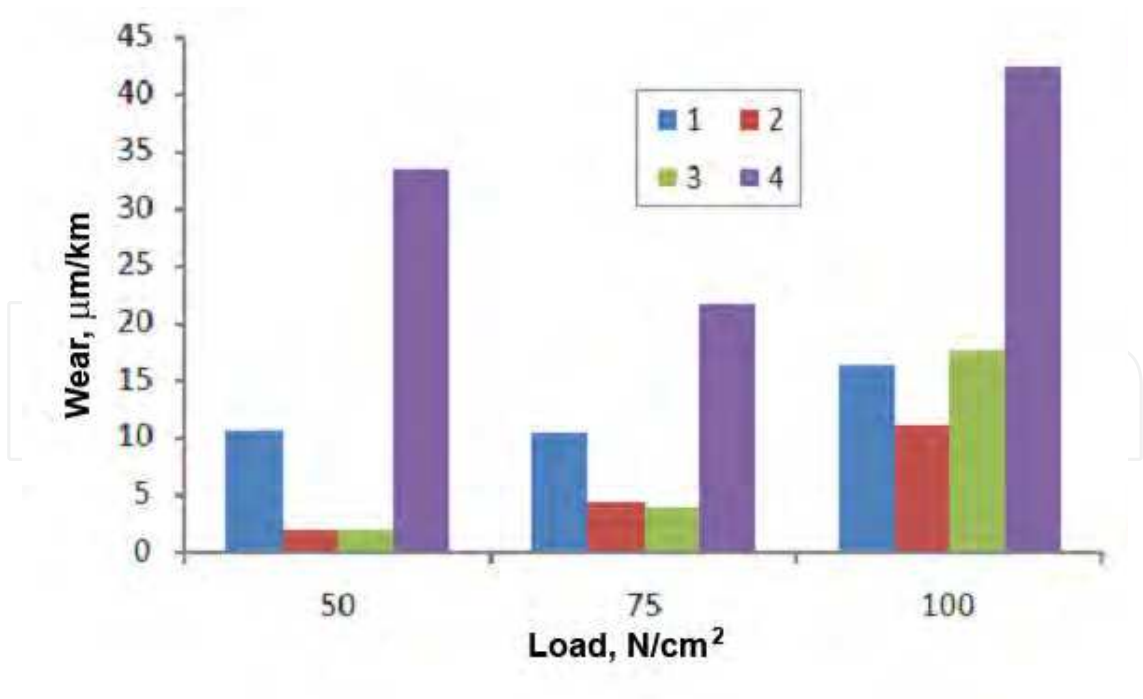
Fig. 17. Effect of load on wear at unlubricated friction of sintered boron steels (No. 1-3, table 3) and 100Cr6 bearing rolled steel (4).

4. Conclusions

The presented results had shown that application of boron containing master alloys provides a superior level of sintered and heat-treated boron steels mechanical properties as compared with boron carbide.

Boron content in the sintered steels that provides an optimum combination of their properties with application of master alloys of different composition, is 1.2 % (weight). This content of B corresponds to ultimate values of hardness and strength for both sintered and heat-treated materials and high level of wear resistance as well.

Comparison of the mechanical and tribotechnical characteristics of the tested materials testify that there is no direct relation among values of their strength, hardness and wear resistance properties. However, every of this steels, in spite of presence of residual porosity, while possessing by commensurable mechanical characteristics with 100Cr6 bearing rolled steel, is distinguished by considerably raised wear resistance.

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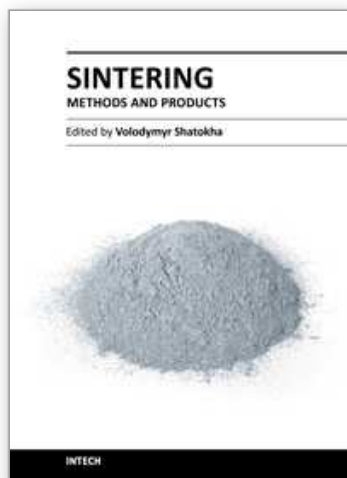
Sintering - Methods and Products

Edited by Dr. Volodymyr Shatokha

This book is addressed to a large and multidisciplinary audience of researchers and students dealing with or interested in sintering. Though commonly known as a method for production of objects from fines or powders, sintering is a very complex physicochemical phenomenon. It is complex because it involves a number of phenomena exhibiting themselves in various heterogeneous material systems, in a wide temperature range, and in different physical states. It is multidisciplinary research area because understanding of sintering requires a broad knowledge - from solid state physics and fluid dynamics to thermodynamics and kinetics of chemical reactions. Finally, sintering is not only a phenomenon. As a material processing method, sintering embraces the wide group of technologies used to obtain such different products as for example iron ore agglomerate and luminescent powders. As a matter of fact, this publication is a rare opportunity to connect the researchers involved in different domains of sintering in a single book.

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