# STRUCTURE AND PROPERTIES OF Fe-B-C POWDERS ALLOYED WITH Cr, V, Mo OR Nb FOR PLASMA-SPRAYED COATINGS

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In the present study, the microstructure development and properties of the starting Fe-B-C powders for plasma spraying fabricated by dispersing a consumable rotating rod were investigated as functions of alloying elements additions. These powders were prepared in the following compositional ranges: B (10...14 wt.%), C (0.01...0.5 wt.%), Me (0...5.0 wt.%), where Me – Cr, V, Mo or Nb, balance Fe. Structural properties were characterized by metallography, X-ray diffraction, scanning electron microscopy, and energy dispersive spectroscopy. Mechanical properties of the powders were measured by a Vickers indenter. Compression strength, oxidation resistance, and melting temperature were also determined. Chromium or vanadium were found to dissolve completely in Fe<sub>2</sub>(B, C) and Fe(B, C) constituent phases of the Fe-B-C powders replacing iron and forming substitutional solid solutions. By entering into the iron borides structure, these alloying elements improve ductility and oxidation resistance but lower melting temperature and hardness of the powders. Molybdenum or niobium were mainly found in secondary phases such as Mo<sub>2</sub>B, Mo<sub>2</sub>(B, C) or NbB<sub>2</sub> at the Fe<sub>2</sub>(B, C) boundaries. As a result, these alloying elements enhance hardness, oxidation resistance and melting temperature of the powders.

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#### INTRODUCTION

Boron-rich Fe-B-C alloys with boron content in the range of 10...14 wt.% are nowadays well-established materials due to their high hardness, relatively low coefficient of friction, and good oxidation and corrosion resistance [1-4]. As such these alloys have attracted significant attention both in basic as well as in applied research areas, resulting in several studies on phase stability, mechanical and performance properties of the ternary Fe-B-C system [5-7]. These properties are attributed to the formation of Fe<sub>2</sub>B and FeB borides in the structure which are major constituents of boron-rich Fe-B alloys. Meanwhile, carbon may be considered as natural addition to Fe-B alloys since these alloys are very reactive with respect to carbon. This element is found to dissolve in the iron borides forming Fe(B, C) and Fe<sub>2</sub>(B, C) solid solutions and add strength at elevated temperatures [8]. At that, with cooling rate of Fe-B-C alloys increasing, carbon solubility increases which enhances solid-solution strength, as well as hardness.

In view of the increasing importance of boron-rich Fe-B-C alloys in practical applications, it is surprising how little is known about the influence of alloying elements on their structure and properties. The studies mainly concern the effects of alloying on boroncontaining steels [9-14]. Very important elements for alloy steels are chromium, vanadium, molybdenum, and niobium. Probably one of the most well-known effects of chromium on steel is the tendency to resist staining and corrosion. Chromium can also increase the toughness of steel, as well as the wear resistance. The metal also improves hardenability, strength, response to heat treatment. Vanadium can produce stable carbides that increase strength at high temperatures. By inhibiting grain growth during heat treatment, it helps enhance the toughness and strength of the steel. Molybdenum tends to minimize temper brittleness and protects against pitting corrosion caused by chlorides

and sulfur chemicals. Found in small quantities in stainless steels, molybdenum increases hardenability and strength at high temperatures. Niobium like vanadium has the benefit of stabilizing carbon by forming hard carbides and, so, is often found in high temperature steels. In small amounts, niobium can significantly increase the yield strength and, to a lesser degree, the tensile strength of steels, as well as have a moderate precipitation strengthening effect.

Fe-B-C alloys are widely used as protective hard coatings produced by various methods, including plasma spray of atomized powders [15–19]. Plasma spraying process that utilizes a high energy heat source to melt and to accelerate fine powders onto a prepared surface is very effective for producing thick coatings due to their rapid cooling rates. Fe-B-C plasma-sprayed coatings have attracted interest due to their unique mechanical properties, high wear resistance, excellent corrosion resistance, and low cost [20–22]. The main advantages of using plasma spraying to form Fe-B-C coatings include good adhesion between the coating and substrate and reproducible results. In addition, plasma spraying is an industrial process, enabling mass production.

Performance properties of plasma-sprayed coatings are strongly influenced by a structure of starting powders [23, 24]. Alloying is a powerful method to improve the characteristics of powders for the manufacture of coatings. Understanding both the positive and negative effects of alloying elements on the structure and properties of boron-rich Fe-B-C powders can help to identify what applications certain coatings can be used in. The very high cooling rates of atomization processes may also contribute to the solubility of alloying elements in phase constituents of the powders thus affecting their properties.

Therefore, the purpose of this work is to investigate the influence of alloying elements (Cr, V, Mo or Nb) on the formation of structure and properties of boron-rich Fe-B-C powders for plasma straying.

## **1. MATERIALS AND METHODS**

Fe-B-C-Me powders for plasma spraying containing 10.0...14.0% B, 0.01...0.5% C, 0...5.0% Me (where Me – Cr, V, Mo or Nb), balance Fe (in wt.%) (purity better than 98.5%) were produced by dispersing a consumable rotating rod [25, 26]. A rotating rod (anode), which was 28...30 mm in diameter and 200...300 mm in length, made of the alloy to be atomized, was melted by an electric arc between the anode and a water-cooled tungsten cathode. Liquid powders were then projected from the anode by the centrifugal force and solidified in the chamber filled with argon at a pressure of 0.1...0.15 MPa. The atomization process parameters were as follows: 380...400 A. 40 V. The lower threshold was determined by the requirement of the highest output of the process. But, if the strength of current was higher than 400 A, there was not enough time for metal to spheroidize and it solidified as shapeless powders. But the powders to be injected into the plasma must possess good flow properties, which is associated with their spherical shape. The powders diameters ranged from 100 to  $200\,\mu\text{m}$ , and average diameter defined by the cumulative weight fraction of 50% was about 125 µm. Powders with this narrow size distribution are preferred in order to achieve uniform heating and acceleration of a multicomponent powder. The cooling rate of the powders was  $10^3 \dots 10^4$  K/s.

The microstructural characterization of the powders was performed by light metallographic microscope Neophot-32 and quantitative analyzer Epiquant. The Xray diffraction (XRD) measurements of lattice parameters of the phases were carried out using diffractometer HZG-4A with  $CuK_{\alpha}$  radiation. The XRD analyses were performed with powder samples. The microstructure was also studied with a scanning electron microscope Jeol-2010 F (SEM) equipped with an energy dispersive spectrometer (EDS). The melting temperature ( $T_m$ ) of the powders was measured by differential thermal analysis at 5 K/min heating rate.

Vickers microhardness tests were carried out using device PMT-3 at 0.05 kg load in order to observe the crack propagation in the samples. A mean Vickers microhardness ( $H_{\mu}$ ) was obtained by averaging over 20 indentations. Total Vickers hardness was calculated considering additivity of this characteristic. The fracture toughness ( $K_{IC}$ ) was evaluated from the crack length initiated at the corners of the Vickers microindentation using an empirical equation proposed in [27]. Compression strength ( $\sigma$ ) was measured at room temperature with load applied up to fracture of the powders. Oxidation resistance factor ( $K_o$ ) was measured with powders oxidized in air at 1273 K for 2 h.

## 2. RESULTS AND DISCUSSION

The effects of alloying elements in boron-rich Fe-B-C powders are as follows. The addition to powders of elements such as Cr or V does not basically alter the microstructures formed. The Fe<sub>2</sub>B- and FeB-based solid solutions are observed in the structure (Figs. 1, 2). So, the chromium or vanadium do not form chemical compounds, and consequently the only form in

which they can be present in the powders is in solid solutions with iron borides. Chromium and vanadium can dissolve in either the Fe<sub>2</sub>B and FeB borides but predominantly they dissolve into iron monoboride (see Figs. 1, 2). These elements tend to cause distortions of crystal lattices of Fe<sub>2</sub>(B, C) and Fe(B, C) typical for substitutional solid solutions. Their atoms are of similar size and behaviour [28] and so the lattice structure of the Fe<sub>2</sub>B and FeB borides does not alter greatly as a result of substituting Fe atoms for Cr or V (Table 1).



Fig. 1. SEM of polished cross-sections of Fe-12.1B-0.1C powder alloyed with 5% of Cr: a – second electron image; b – elemental EDS X-ray mapping; c – elemental profile along scanning line

Table 1

Alloying	Fe(B, C) (rhombic lattice)			Fe <sub>2</sub> (B, C) (tetragonal lattice)		
element	a, Å	$b, \mathrm{\AA}$	<i>c</i> , Å	a, Å	$c, \mathrm{\AA}$	c/a
w/o	5.5041±0.0052	4.0596±0.0106	2.9501±0.0037	5.1120±0.0001	4.2418±0.0001	0.8298
Cr	5.4911±0.0032	$4.0569 \pm 0.0052$	2.9602±0.0021	5.1173±0.0004	4.2313±0.0046	0.8269
V	$5.4998 \pm 0.0020$	4.0576±0.0066	2.9697±0.0011	5.1174±0.0014	4.2427±0.0053	0.8291
Mo	$5.5083 \pm 0.0018$	4.0671±0.0038	$2.9663 \pm 0.0007$	5.1184±0.0010	4.2467±0.0075	0.8297
Nb	5.5055±0.0042	4.0688±0.0067	2.9491±0.0042	5.1131±0.005	4.2414±0.0028	0.8295

The lattice parameters of Fe(B, C) and Fe<sub>2</sub>(B, C) crystals in the Fe-12.1B-0.1C powders alloyed with 5% of V, Cr, Mo or Nb



*Fig. 2. SEM of polished cross-sections of Fe-12.1B-0.1C powder alloyed with 5% of V: a – second electron image; b – elemental EDS X-ray mapping; c – elemental profile along scanning line* 

Table 2

Structural and mechanical characteristics of Fe(B, C) and Fe<sub>2</sub>(B, C) crystals in the Fe-12.1B-0.1C powders alloyed with 5% of V, Cr, Mo or Nb

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Alloying	A 11 a	Fe(B, C)					$Fe_2(B, C)$	
	Dendrite parameters*, µm				$K = MD_{2} m^{1/2}$		$\boldsymbol{K}$ MDa m <sup>1/2</sup>	
	element	$d_0$	$l_0$	$d_0/l_0$	$H_{\mu}$ , GPa	$\Lambda_{\rm IC}$ , wiPa·m	$\Pi_{\mu}$ , GPa	$\Lambda_{\rm IC}$ , MPa·III
	w/o	4.9±0.1	5.3±0.1	0.92	20.1±0.1	5.0±0.2	17.0±0.1	4.5±0.1
	Cr	4.1±0.1	4.3±0.1	0.95	18.6±0.2	-	15.7±0.3	5.2±0.1
	V	4.4±0.1	4.8±0.1	0.92	19.1±0.1	-	15.8±0.2	-
	Mo	3.1±0.1	3.6±0.1	0.86	21.0±0.2	4.4±0.1	17.8±0.2	4.1±0.1
	Nb	2.4±0.05	2.8±0.1	0.86	20.2±0.1	4.9±0.1	17.1±0.1	4.4±0.2
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\*Where  $d_0$  – diameter of secondary dendritic arms,  $l_0$  – interdendritic distance.

In the Fe-B-C powders, the presence of chromium or vanadium increases fracture toughness  $K_{\rm IC}$  while not appreciably reducing Vickers hardness  $H_{\mu}$  of the constituent phases (Table 2). Chromium or vanadium improve significantly compression strength  $\sigma$  and oxidation resistance factor  $K_o$  due to formation of inert passive films on the surface which resist attack by oxidizing reagents (Table 3). In Fe-B-C powders, chromium appears to have a greater beneficial effect on compression strength but vanadium – on resistance to oxidation. These elements also provide slightly lower melting temperature  $T_{\rm m}$ .

Table 3 Performance properties of Fe-10.3B-0.7C-5.0 Me powders (Me - Cr, V, Mo or Nb)

Alloying element	Total $H_{\mu}$ , GPa	σ, MPa	T <sub>m</sub> , K	<i>K</i> <sub>o</sub> , relative units
w/o	17.9±0.1	2610±10	1723±4	1.0
Cr	16.6±0.2	2850±20	1713±4	1.32±0.12
V	16.8±0.1	2800±20	1713±4	$1.53 \pm 0.08$
Mo	19.1±0.2	2750±10	1758±4	1.12±0.10
Nb	18.8±0.1	2675±20	1763±4	1.24±0.06

Small amounts of Mo added to Fe-B-C powders completely dissolve. At some point, additional amounts of this alloying element will not dissolve. When that solid solubility limit is exceeded, Mo slightly dissolves in Fe<sub>2</sub>B boride but preferentially in FeB dendrites refining their size (Fig. 3, Table 2). The addition of this element that dissolves in the Fe<sub>2</sub>B and FeB lattices replacing iron atoms causes noticeable distortions because of the difference in atom size (see Table 1) [28]. Besides, with boron molybdenum forms Mo<sub>2</sub>B, but in the presence of carbon it forms Mo<sub>2</sub>(B, C) as well. Thus, at the amount of 5% this alloying element forms mainly special borides and borocarbides.



Fig. 3. SEM of polished cross-sections of Fe-12.1B-0.1C powders alloyed with 5% of Mo: a – second electron image; b – elemental EDS X-ray mapping; c – elemental profile along scanning line Since the molybdenum atoms are larger than the surrounding atoms, they introduce compressive lattice strains. They disrupt the regular arrangement of ions and make it more difficult for the layers to slide over each other. This makes the FeB and Fe<sub>2</sub>B phases harder and less ductile than those of the master Fe-B-C powders (see Table 2). For example, powders of 5% Mo raise the total hardness from 17.9 to 19.1 GPa (see Table 3). Besides, the addition of Mo to Fe-B-C powders rises melting temperature and slightly improves oxidation resistance.

When niobium is added to Fe-B-C powders, it does not form solutions in iron borides and enters  $NbB_2$  secondary phase formed at  $Fe_2(B, C)$  boundaries (Fig. 4).





Insolubility of niobium in iron borides is in good agreement with XRD measurements of lattice parameters of Fe(B, C) and  $Fe_2(B, C)$  phases (see Table 1). This implies that Nb is continually pushed out in the melt ahead of the moving solid-liquid interface into the interdendritic regions of growing Fe(B, C) dendrites slowing their growth and causing noticeable refinement (see Table 2). As a result, the secondary crystals of NbB<sub>2</sub> appear at the Fe<sub>2</sub>(B, C) boundaries.

Fe-B-C powders with 5% niobium are harder (see Table 3), on account of the finer Fe(B, C) dendrites formed and the presence of  $NbB_2$  secondary phase. Niobium, as such, performs in the same way as that described for molybdenum. The addition of niobium to Fe-B-C powders has a substantial effect on the melting temperature (see Table 3). Its presence also improves oxidation at high temperatures.

It should be noted that the solubility of investigated alloying elements in the constituent phases of Fe-B-C powders increases in the order Nb, Mo, V, Cr. Taking into account the metallic radii of individual metal atoms [28] allows to explain why Cr and V completely dissolve in Fe-B-C powders, but Mo and Nb have partial or no solubility, as in this sequence the atomic radii decrease. It is also understandable why alloying elements are predominantly dissolved in the rhombic lattice of Fe(B, C) than in more close-packed tetragonal lattice of Fe<sub>2</sub>(B, C).

For a thorough understanding of mechanical properties of alloyed Fe-B-C powders, it is important to consider the electronic structure of the constituent phases, including the electron distribution [29]. In Fe<sub>2</sub>(B, C), boron forms strong covalent Fe-B bonds [30]. According to the crystal structure, these bonds connect B to eight neighboring Fe atoms. In the crystal structure of Fe(B, C), B atoms forming zigzag chains are in the interstices surrounded by Fe atoms. The strong interactions between the atoms lead to strong covalent B-B bonds. The combination of the covalent Fe-B bonds and metallic Fe-Fe bonds in Fe<sub>2</sub>(B, C) or the combination of the covalent B-B bonds and metallic Fe-Fe bonds and metallic Fe-Fe bonds in Fe(B, C) contribute to the properties of these compounds [30].

In assessing the alloying effects, the relative change in concentration of the collectivized valence electrons forming the Fe-B and Fe-Fe bonds, when Fe atoms are replaced by Me atoms (where Me – Cr, V or Mo), is responsible for the observed changes in mechanical properties. Here, a decrease in hardness is predicted by alloying with Cr and V, related to a decreased concentration of collectivized electrons taking part in the electronic exchange [29]. As a result, Me-B and Fe-Me bonds become weaker; the hardness of the Fe-B-C powders is found to reduce, but compression strength improves.

According to electronic structure of Mo and Nb, these elements rather act as electron acceptors. Their valence electrons are localized in the stable  $d^5$ -configurations [29]. Any re-distribution of bonding electrons caused by replacing Fe with Mo or Nb in the crystal lattices of Fe(B, C) and Fe<sub>2</sub>(B, C) phases leads to destruction of stable configurations. It results in limited or extremely low solubility of correspondingly Mo or

Nb and formation of secondary phases. In the Fe-B-C powders, some of the added molybdenum ions, larger than iron ions making up the lattice, disrupt the regular arrangement of ions and make it more difficult for the layers to slide over each other. This makes the powders harder and less ductile than the master powder (in which the layers slip over each other more easily). Niobium does not contribute to any hardness increase of Fe(B, C) and Fe<sub>2</sub>(B, C) phases and enhances the hardness of powders with preserving compression strength by strengthening via the precipitation of secondary phases.

#### CONCLUSIONS

This study shows that the major constituents of boron-rich Fe-B-C powders sized from 100 to 200  $\mu$ m in diameter are found to be Fe(B, C) and Fe<sub>2</sub>(B, C) solid solutions. When adding up to 5% of Cr or V to Fe-B-C powders, these elements have complete solubility in the structural constituents, preferentially dissolving in Fe(B, C) phase and forming substitutional solutions. Cr or V introduce the smallest lattice distortions which relates to the relatively small differences in the atomic sizes between the iron and substituting atoms.

Molybdenum or niobium have correspondingly a limited or extremely low solubility in Fe-B-C powders. At that, the refinement of structure is observed by Mo or Nb additions. When the Mo or Nb content reaches 5%, these elements are found in powders in the form of chemical compounds with boron and/or carbon (Mo<sub>2</sub>B, Mo<sub>2</sub>(B, C), NbB<sub>2</sub>) precipitated at Fe<sub>2</sub>(B, C) boundaries.

All alloying elements that form solid solutions in Fe(B, C) and  $Fe_2(B, C)$  phases affect their hardness. Chromium, similar to vanadium, decreases hardness, while molybdenum gives the hardness increase caused by substitutional strengthening. This is because the different atom size of Mo interrupts the orderly arrangement of atoms in the lattices. Besides, molybdenum like niobium increases hardness due to formation of secondary phases.

Chromium has the largest effect on compression strength, followed by decreasing effects from vanadium, molybdenum and then niobium. Although different mechanisms are involved in compression strength, the addition of all alloying elements produces a rise in this characteristic.

Chromium or vanadium are most effective for improving oxidation resistance whereas molybdenum or niobium increases this property to a lesser degree. This improvement of the oxidation resistance is credited to the affinity of alloying elements for oxygen and, as a consequence, protective films are formed on the surface of the powders that prevent the further diffusion of oxygen.

Alloying elements, such as Cr and V, which enter into Fe(B, C) and  $Fe_2(B, C)$  solid solutions lower the melting temperature, with the exception of Mo and Nb that give rise to melting temperature.

The effects of investigated alloying elements replacing iron atoms in the Fe-B-C powders may be explained considering a local change of the electronic structure of alloyed Fe(B, C) and  $Fe_2(B, C)$  phases which is an indicator for stronger/weaker bonded atoms in the crystals.

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## СТРУКТУРА И СВОЙСТВА ПОРОШКОВ Fe-B-C, ЛЕГИРОВАННЫХ Cr, V, Mo ИЛИ Nb ДЛЯ ПЛАЗМЕННОГО НАПЫЛЕНИЯ

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Исследовали влияние легирующих элементов на процессы формирования структуры и свойств порошков для плазменного напыления, изготовленных методом распыления вращающегося стержня. Состав порошков находился в следующем концентрационном диапазоне: В (10...14 вес.%), С (0,01...0,5 вес.%), Ме (0...5,0 вес.%), где Ме - Сг, V, Мо или Nb, Fe - остаток. Структуру порошков изучали методами рентгеноструктурного анализа, сканирующей электронной микроскопии, металлографии, рентгеноспектрального микроанализа. Механические свойства порошков измеряли на микротвердомере Виккерса. Также определяли прочность на сжатие, окалиностойкость, температуру плавления. Хром и ванадий полностью растворяются в структурных составляющих Fe<sub>2</sub>(B, C) и Fe(B, C) порошков Fe-B-C, замещая железо в кристаллических решетках фаз и формируя твердые растворы замещения. Присутствуя в структуре боридов железа, эти элементы повышают их пластичность и сопротивление окислению, но снижают температуру плавления и твердость порошков. Молибден и ниобий в основном присутствуют в структуре в виде вторичных фаз, таких как Mo<sub>2</sub>B, Mo<sub>2</sub>(B, C) или NbB<sub>2</sub>, выделяющихся по границам кристаллов Fe<sub>2</sub>(B, C). Как следствие, эти легирующие элементы повышают твердость, сопротивление окислению и температуру плавления порошков.

## СТРУКТУРА І ВЛАСТИВОСТІ ПОРОШКІВ Fe-B-C, ЛЕГОВАНИХ Cr, V, Mo Або Nb Для плазмового напилення

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Досліджували вплив легуючих елементів на процеси формування структури та властивостей порошків для плазмового напилення, виготовлених методом розпилення обертового стрижня. Склад порошків діапазоні: В (10...14 ваг.%), С (0,01...0,5 ваг.%), знаходився в наступному концентраційному Ме (0...5,0 ваг.%), де Ме - Сг, V, Мо або Nb, Fe - залишок. Структуру порошків вивчали методами металографії, рентгеноструктурного аналізу, сканувальної електронної мікроскопії, рентгеноспектрального мікроаналізу. Механічні властивості порошків вимірювали на мікротвердомірі Віккерса. Також визначали міцність на стиск, окалиностійкість, температуру плавлення. Хром та ванадій повністю розчиняються в структурних складових Fe<sub>2</sub>(B, C) і Fe(B, C) порошків Fe-B-C, заміщуючи залізо в кристалічних гратках фаз і формуючи тверді розчини заміщення. Проникаючи в структуру боридів заліза, ці елементи підвищують їх пластичність і опір окисленню, але знижують температуру плавлення і твердість порошків. Молібден та ніобій в основному присутні в структурі у вигляді вторинних фаз, таких як Мо<sub>2</sub>B, Мо<sub>2</sub>(B, C) або NbB<sub>2</sub>, що виділяються по межах кристалів Fe<sub>2</sub>(B, C). Як наслідок, ці легуючі елементи підвищують твердість, опір окисленню і температуру плавлення порошків.