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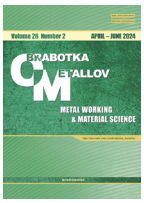

Study of Fe-matrix composites with carbide strengthening, formed by sintering of iron titanides and carbon mechanically activated mixtures

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ABSTRACT

Introduction. The addition of dispersed solid particles of refractory compounds (carbides, borides, silicides) to the structure of alloy is a widely used effective way to increase the wear resistance of steels and alloys. Composites with a matrix of iron-based alloys (steel and cast iron) strengthened by titanium carbide particles are of great practical interest. The main structural characteristics, which define hardness and wear resistance of the composites, are volume fraction, dispersion and morphology of the particles of the strengthening carbide phase. The structure of composites depends on the method of its preparation. The methods of powder metallurgy combined with preliminary mechanical activation of powder mixtures have become widespread. It is previously established that in mechanically activated powder mixtures of $FTi_{35}S_5$ ferrotitanium, consisting of 82 % of $(Fe,Al)_2Ti$ phase, and *P-803* carbon black, a reaction occurs with the formation of a composite consisting of a steel binder and titanium carbide. The synthesis reaction of carbides occurs in a solid-phase mode at combustion's temperatures of 900–950 °C. Therefore, there is no coarsening of the structure due to the growth of carbide particles, which is typical for reactions in the presence of a liquid phase. $FTi_{35}S_5$ alloy contains a plenty of impurities (silicon, aluminum and etc). **The purpose of the work** is to investigate the phase composition and structure of the products of the interaction of Fe_2Ti and *FeTi* iron titanides with carbon under the conditions of reaction sintering of mechanically activated powder mixtures and to determine the possibility of synthesizing iron-matrix composites strengthened with submicron titanium carbide particles. **Research methods.** The structure and phase composition of sintered compacts from mechanically activated powders were studied by optical metallography, X-ray diffraction (*XRD*) and scanning electron microscopy (*SEM*) using determination of the elemental composition by energy-dispersive X-ray spectroscopy (*EDX*). **Experimental technique.** The reaction mixtures were prepared using intermetallic powders obtained by vacuum sintering of compacts from iron and titanium powder mixtures of *2Fe+Ti* and *Fe+Ti* compositions. Carbon black was added to the intermetallic powders to convert all the titanium containing in the intermetallic compounds into carbide. The titanides – carbon black mixtures were processed by an *Activator 2S* planetary ball mill for 10 min milling time at a rotation speed of 755 rpm (40*g*). The mechanically activated mixtures were cold compacted into cylindrical samples with a diameter of 20 mm, which were sintered in vacuum at a temperature of 1,200 °C and an isothermal holding time of 60 minutes. **Results and discussion.** According to the results of X-ray diffraction analysis, almost all titanium contained in iron titanides reacts with carbon to form carbide and reduced iron. The sintering products of compacts of both compositions contain target phases: titanium carbide with a slight shift from the equiatomic ratio and α-iron, which has the lattice parameters close to the reference data, and also a few of other phases. The titanium carbide particles in the iron binder were identified on the back-scattered electron (*BSE*) images due to the tonal contrast: the heavy iron appears darker against the carbide, which is composed of lighter elements. According to *EDX* analysis, the relative content of titanium and carbon in the carbide particles indeed corresponds to the composition of non-stoichiometric titanium carbide. **Conclusion.** The composites including titanium carbide and α-iron binder were obtained by sintering of iron titanides and carbon (carbon black) mechanically activated powder mixtures. The granules of composite powders obtained by crushing of sintered compacts are of interest as feedstocks for wear-resistant coatings and additive technologies, as well as for manufacturing of dense materials by other compaction methods: spark plasma sintering (*SPS*) or hot pressing (*HP*).

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Introduction

Steels and alloys based on nickel, copper, aluminum and other metals used in industry have low wear resistance, especially under dry friction and abrasive conditions. One of the most widely used techniques to increase wear resistance is to add dispersed solid particles of refractory compounds, i.e. carbides, borides, silicides, into the alloy structure. The material obtained in this way has the structure of a metal matrix composite with dispersion strengthening. Composites with a matrix of iron-based alloys (steel and cast iron) strengthened by titanium carbide particles are of the greatest practical interest. Numerous investigations have been devoted to the study of such composites [1]. Due to the low plasticity of dispersion-strengthened metal-matrix composites, its use as structural materials is limited. Therefore, the metal-matrix composites, including composites with iron-based binders, are used primarily for the parts subjected to severe abrasive wear.

The main structural characteristics, which determine the hardness and wear resistance of the composites, are the volume fraction, dispersion and morphology of the strengthening carbide phase particles. The structure of composites depends on the method of its preparation. In casting methods, titanium and carbon are added into the melt, which, during casting and crystallization, form carbide inclusions in the volume of a steel or cast-iron matrix. Lumpy material, i.e. coal coke, pure titanium or titanium-based alloys, is sometimes used to alloy the melt with titanium and carbon [2, 3]. Titanium and carbon powder compacts are more often used, which are placed in a casting mold and poured with steel or cast-iron melt [4, 5]. The carbide phase in the structure of cast composites is represented by round particles ranging in size from 1–3 to 10–15 μm, depending on the concentration of titanium and carbon in the melt and casting conditions, i.e. melt temperature and casting mold, cooling rate, mixing conditions, etc. Attempts to obtain cast details with a surface layer strengthened with carbide particles are described. For this purpose, the surface of the casting mold was covered with a suspension of the titanium and carbon powder mixture. During casting, the covering was impregnated with a melt with the simultaneous synthesis of titanium carbide [6, 7].

Powder technologies for the production of composites with a steel matrix strengthened with titanium carbide particles are used much more often than foundry ones. The most efficient method is sintering of compacts from titanium carbide and steels powder mixtures, which are often replaced with a mechanical mixture of iron powders and alloying elements [8–10]. This method makes it possible to obtain two-layer or multilayer products by sintering of compacts, which consist from layers of various compositions [11]. When titanium carbide powder is replaced by a titanium and carbon mixture, carbide synthesis occurs during sintering, i.e. reactive sintering takes place [12]. Ferroalloy powders are sometimes added to the mixture for pressing and sintering to obtain steel binders [13, 14]. To reduce the porosity of sintered compacts and prevent the growth of carbide grains during isothermal holding, more complex sintering methods are used, requiring specialized equipment: hot pressing [15, 16] or spark plasma sintering [17, 18].

The most effective method for producing "titanium carbide – iron binder" composites is self-propagating high-temperature synthesis (*SHS*) in reaction mixtures of titanium, carbon and iron (or its alloys). Numerous studies of synthesis products in these reaction mixtures are devoted to the thermokinetic characteristics of the synthesis [19] and its influence on the formation of the composite structure [20, 21]. The dispersion of carbide particles growing from a melt-solution in a combustion wave, its morphology and crystallographic features of growth has been studied [22, 23]. Synthesis has been studied both in the wave combustion and in the thermal explosion modes [24–26]. It is known that the interaction of powder components in reaction mixtures during synthesis intensifies greatly after mechanical activation in high-energy mills [27]. However, our studies have shown that the effect of mechanical activation on the concentration limits of combustion and the initiation of the synthesis reaction in *Ti + C + Fe* alloy mixtures (high-chromium cast iron or high-speed steel) is much less than expected [28]. The main reason, in our opinion, is the binder metal, which partially blocks the titanium-carbon reaction surface and prevents the carbide synthesis reaction. This can be avoided by replacing two powders, i.e. titanium and binder metal, in reaction mixtures with a powder of an intermediate compound – metal titanide.

We have proven the effectiveness of the described approach by investigations of *FTi35S5* ferrotitanium, which consists of 82 % of $(Fe,Al)_2Ti$ intermetallic compound, and *P-803* carbon black powder mixtures. It is shown that in mechanically activated ferrotitanium and carbon black powder mixtures, a reaction occurs both in wave and thermal explosion modes with the formation of a composite based on 50 vol. % of titanium carbide. The synthesis reaction occurs in solid-phase mode at the combustion temperatures of 900–950 °C. Due to the low combustion temperatures, coarsening of the structure does not occur and the carbide particles have a submicron size. Since *FTi35S5* industrial ferrotitanium contains many impurities, i.e. silicon, aluminum, the purpose of the study is to investigate the reaction products in mechanically activated mixtures of iron titanides – Fe_2Ti and *FeTi* – with carbon (carbon black) and to examine the possibility of synthesizing iron-matrix composites strengthened with submicron titanium carbide particles.

Materials and research methodology

The intermetallic powders were obtained by vacuum sintering of compacts at a temperature of 1.250 °C with isothermal holding for 2 hours from mechanically activated mixtures of elemental powders of two compositions: *2Fe + Ti* (77.7 wt. % iron + 22.3 wt. % titanium) and *Fe + Ti* (63.6 wt. % iron + 36.4 wt. % titanium). Subsequently, the intermetallic powders were used as initial powder materials for the synthesis *TiC + Fe* binder composites. A detailed procedure for the preparation of these intermetallic powders and the specifications of the initial powders are described in [31]. By sintering the *2Fe + Ti* mixture, it was possible to obtain a Fe_2Ti single-phase intermetallic compound. According to the results of X-ray diffraction analysis, the sintering product of a compact from the *Fe + Ti* mixture contained 82 vol. % of Fe_2Ti compound and 18 vol. % of target *FeTi* phase. The reason for the predominant formation of the Fe_2Ti compound is twice the negative value of the enthalpy of the Fe_2Ti compound formation compared to that for *FeTi*: –87.45 and –40.58 kcal/mol, respectively [32].

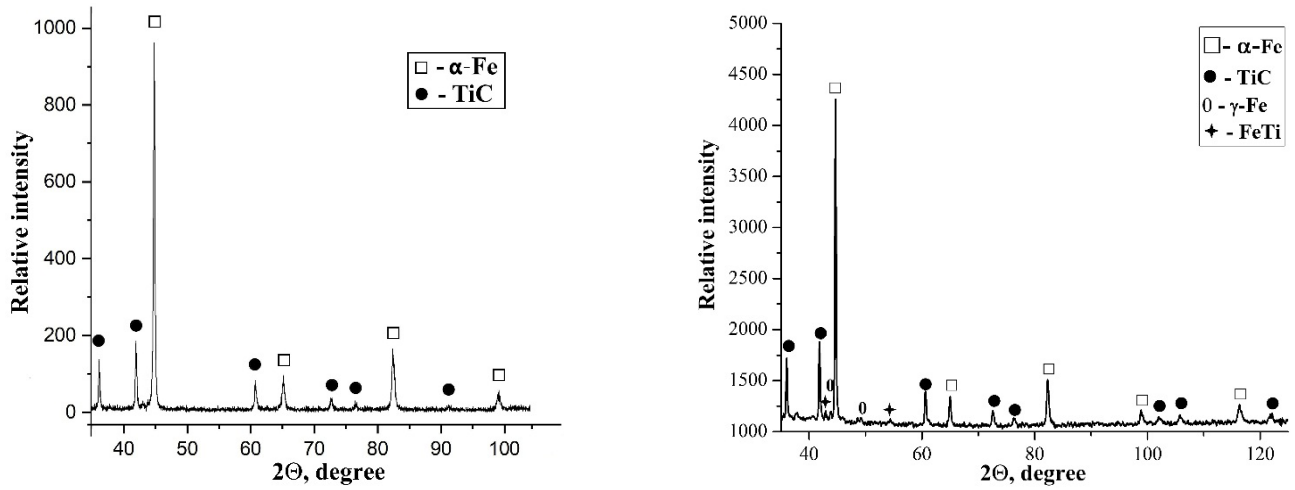
Carbon black was added to intermetallic powders with the above phase composition in the amount necessary to convert all the titanium contained in the intermetallic compounds into titanium carbide. The obtained mixtures were processed in an *Activator-2S* planetary mill at a rotation speed of 755 rpm (40 g) for 10 minutes with the ethanol additive to exclude the powder sticking to the grinding balls and drum walls. Cylindrical specimens with a diameter of 20 mm were compacted from the mechanically activated mixtures and sintered in a vacuum at a temperature of 1,200 °C with isothermal holding of 60 minutes. The structure and phase composition of sintering compacts were studied using the equipment of the *Shared Use Center "Nanotech"* of the *ISPMS SB RAS* by optical metallography (*AXIOVERT-200MAT* optical microscope, Zeiss, Germany), scanning electron microscopy (*EVO 50* scanning electron microscope, Zeiss, Germany) and X-ray diffraction analysis (*DRON-8N* X-ray diffractometer, Bourevestnik, Russia). Diffraction patterns were obtained in an angle range of $2\Theta = 35°–125°$ with a scanning step of 0.1° for exposure of 1 second using CuK_α irradiation. Identification of phases was carried out using the *ASTM* X-ray database. Processing of the primary results was carried out using the *MAUD* software and "Qualitative and quantitative phase analysis" (Bourevestnik, JSC, St. Petersburg) by *Rietveld* method using the *COD* database.

Results and discussion

Sintered materials

According to the results of X-ray diffraction analysis (fig. 1, Table 1), the sintering products of compacts of both compositions contain the target phases (titanium carbide, α-iron) and trace amounts of other phases. Thus, the titanium contained in iron titanides reacts with carbon to form carbide and reduced iron.

The titanium and iron carbide relative content in the sintering products depend on the elemental ratio in the reaction mixtures, which kept to be unchanged during the synthesis process. The lattice parameter of titanium carbide is slightly lower than the reference values [33] for equiatomic titanium carbide, i.e. 0.4327 nm, which is the result of a composition shift from equiatomic towards titanium. According to the titanium-carbon equilibrium diagram [34], titanium carbide has a wide homogeneity region extending from $TiC_{0.5}$ to



a *b*

Fig. 1. X-ray patterns of compacts sintered (1.200 °C, 60 min) from mechanically activated $Fe_2Ti + C$ (*a*) and *FeTi* + *C* (*b*) mixtures

Table 1

Phase composition (vol. %) of products of sintered compacts from mechanically activated $Fe_2Ti + C$ and *FeTi* + *C* mixtures

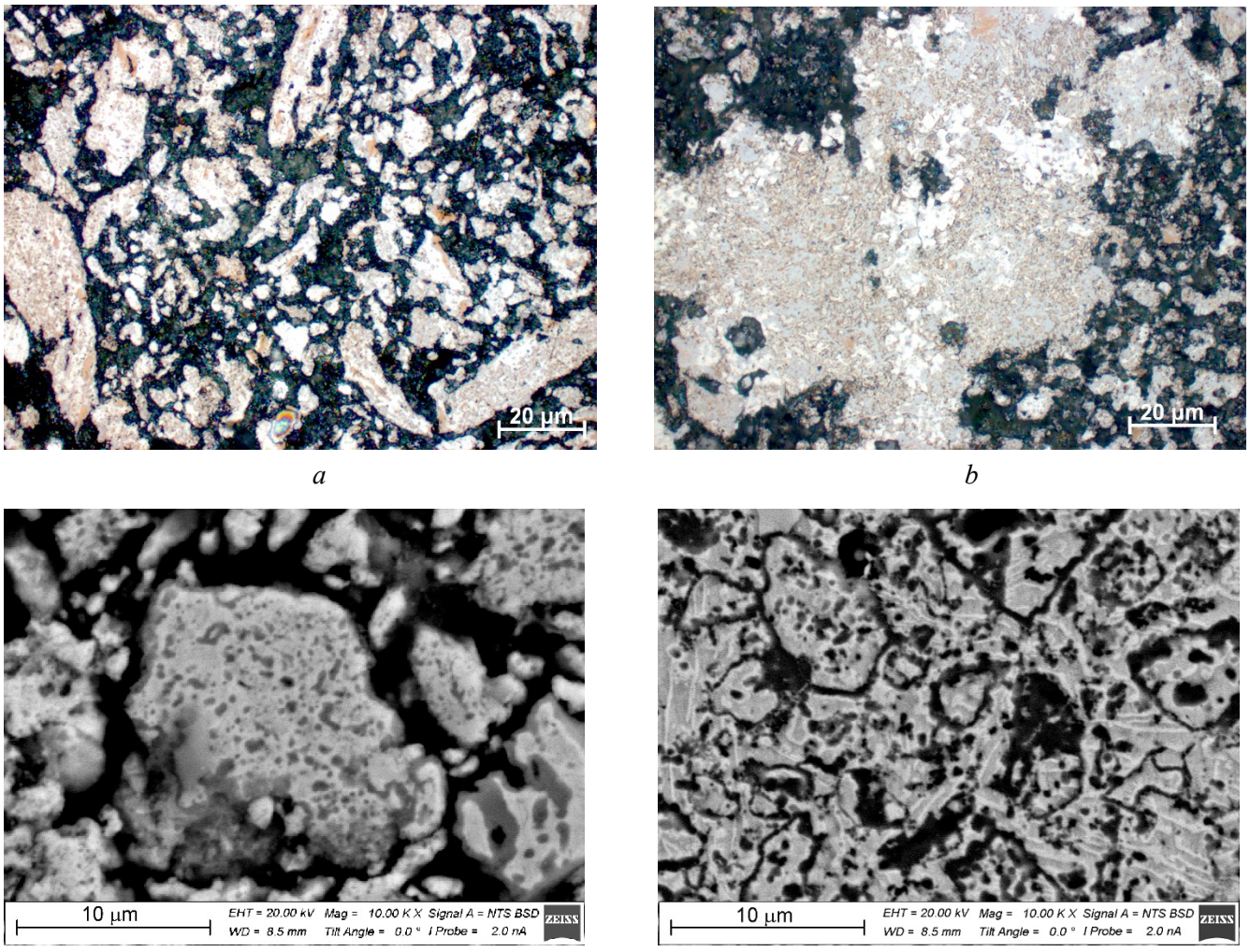
№	Mixture composition	Volume content of phases, %			Lattice parameters, nm	
		TiC	*α-Fe*	Others	*TiC*	*α-Fe*
1	$Fe_2Ti + C$	45	54	1.0 (*Ti*)	0.43173	0.28676
2	*FeTi* + *C*	57.3	40.3	0.8 (*γ-Fe*), 1.6 (*FeTi*)	0.43204	0.28696

TiC. The cubic lattice parameter decreases as the elemental composition of the carbide moves away from the equiatomic one [35, 36]. The lattice parameter of α-iron is close to the reference value, i.e. 0.2866 nm.

The microstructure of sintering materials is shown in figure 2. With pressureless sintering, it is not possible to obtain a dense material. In the optical metallography images (fig. 2 *a*, *b*), areas ranging in size from several up to tens of microns, separated by epoxy-filled pores, are visible. In the backscattered electron microscopic images in figure 2, *c*, *d*, the iron binder and titanium carbide are clearly distinguishable due to the tonal contrast: heavy iron appears lighter than carbide consisting of lighter elements.

In Table 1 we marked the $Fe_2Ti + C$ composition as composite No. 1 and the *FeTi* + *C* composition as composite No. 2. In the structure of composite No. 1, which has a larger volume fraction of the binder, carbide inclusions of micron and submicron size are located predominantly in the volume of the binder (fig. 2, *a*, *c*). Due to the small size of carbide inclusions, it is difficult to accurately determine its elemental composition. The dispersed structure of composite No. 2, containing a larger volume fraction of the carbide phase, can be discerned only at high magnification (fig. 2, *d*). Submicron carbide inclusions in the composite No. 2, as well as in the composite No. 1, are located in the volume of the binder (fig. 2, *d*), but because of the great volume fraction of the carbide phase in composite No. 2, some of it are located outside of the composite granules. In the volume of the binder in figure 2, *d*, an unidentified phase is visible in the form of lighter lamellas. It is possible, that is austenite lamellas, weak lines of which are present in the X-ray diffraction pattern (fig. 1, *b*).

The elemental composition of the binder in composite No. 1 was evaluated by *EDX* point analysis in a few areas free of carbide particles (fig. 3). According to local elemental analysis data given in Table 2, the binder has a significant carbon content. The probable reason for the increased carbon content in *α-Fe* is the influence of titanium, which, according to the phase diagram of the *Fe-Ti* system [34], is a strong



a *b*

c *d*

Fig. 2. Optical (*a*, *b*) and *SEM* images (*c*, *d*) of the microstructure of sintered compacts from mechanically activated mixtures: $Fe_2Ti + C$ (*a*, *c*) and $FeTi + C$ (*b*, *d*)

Table 2

Elemental composition of the steel binder in composites sintered from a mechanically activated $Fe_2Ti + C$ mixture (Fig. 3)

Number of spectrum	Content of elements, at. %		
	Titanium	Carbon	Iron
1	2.48	12.02	85.51
3	1.86	16.81	81.33

ferrite promoter. Titanium solubility in α-*Fe* exceeds 10 at. %. Another reason for the increased carbon content in the binder can be insufficient locality of the electron probe method for determining the elemental composition in small-sized areas.

The elemental composition of the binder in the structure of composite No. 2 was also assessed by *EDX* point analysis (fig. 4, Table 3).

According to the results of local elemental analysis (Table 3), the binder in composite No. 2 contains an anomalously large amount of carbon. One possible explanation may be the effect of titanium on the solubility of carbon in ferrite. However, in our opinion, the main reason is the insufficient locality of the electron probe method for local elemental analysis in the finely dispersed composition structure.

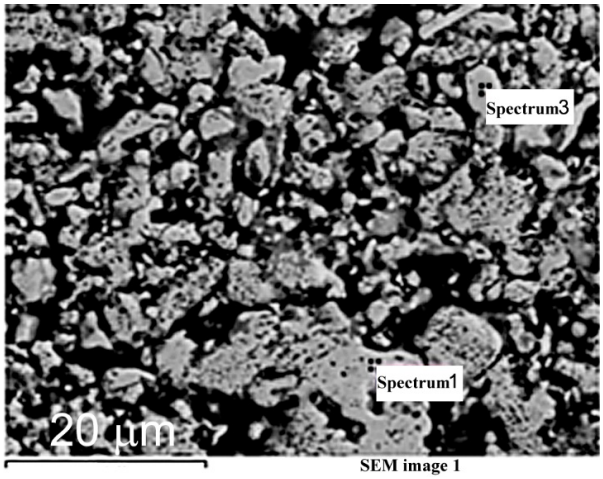


Fig. 3. Microstructure of a sintered composite from the mechanically activated $Fe_2Ti + C$ mixture (*BSE*-mode)

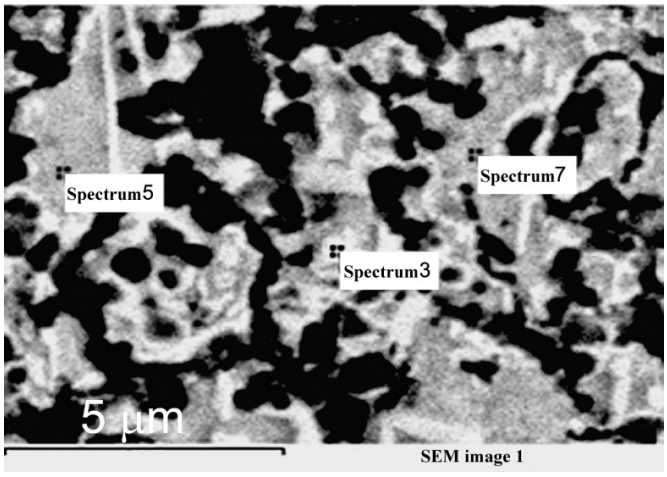


Fig. 4. EDX elemental analysis of the steel binder in sintered composites from a mechanically activated *FeTi* + *C* mixture (*BSE*-mode)

Table 3

Elemental composition of the steel binder in composites sintered from a mechanically activated *FeTi* + *C* mixture (Fig. 4)

Number of spectrum	Content of elements, at. %		
	Titanium	Carbon	Iron
5	3.25	46.67	50.09
3	4.15	48.63	47.21
7	7.87	46.80	45.32

For the same reason, when locally determining the elemental composition in the small dark inclusions (fig. 5, Table 4), a lot of iron is detected in the spectra, since the areas in which X-ray radiation is induced exceed the size of the carbide inclusions. In this case, the relative content of titanium and carbon in the spectra corresponds to the composition of non-stoichiometric titanium carbide. The result further confirm that the dark phase in the *BSE* images is titanium carbide.

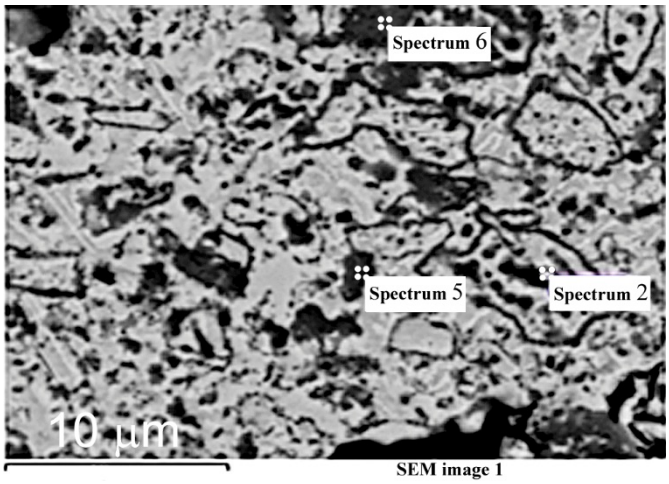


Fig. 5. EDX elemental analysis of carbide inclusions in sintered composites from a mechanically activated *FeTi* + *C* mixture (*BSE*-mode)

Table 4

Elemental composition of carbide inclusions in sintered composites from a mechanically activated *FeTi* + *C* mixture (Fig. 4)

Number of spectrum	Content of elements, at. %			
	Titanium	Carbon	Iron	Others
6	59.80	32.90	7.30	–
5	43.57	32.16	24.27	–
2	34.64	30.16	21.87	13.34 (oxygen)

Conclusions

By sintering mechanically activated powder mixtures of iron titanides with carbon (carbon black), the composites were obtained, including, according to the results of X-ray diffraction analysis, titanium carbide and alpha iron. In the structure of a composite sintered from a $Fe_2Ti + C$ mixture, the main part of the carbide in the form of dispersed inclusions is localized in the volume of the steel binder. In a composite sintering from a *FeTi* + *C* mixture, the volume fraction of carbide is one and a half times higher than that of *α-Fe*. Thus, the metal binder in the composite No. 2 is present in the form of a mechanical mixture with titanium carbide. Due to the dispersion of the composites structure, it is difficult to determine the elemental composition of the structural components of sintering composites using *EDX* analysis.

The granules of composite powders obtained by crushing of the sintering compacts are of interest as feedstocks for wear-resistant coating and additive technologies, as well as for manufactiring of dense materials by other compaction methods: spark plasma sintering or hot pressing.

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Conflicts of Interest

The authors declare no conflict of interest.