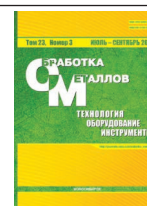




Obrabotka metallov -

Metal Working and Material Science

Journal homepage: http://journals.nstu.ru/obrabotka_metallov



Influence of the chemical composition of the matrix on the structure and properties of monolithic SHS composites

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ARTICLE INFO

Article history:

Received: 15 June 2021

Revised: 08 July 2021

Accepted: 14 July 2021

Available online: 15 September 2021

Keywords:

Self-propagating synthesis
 Composite
 The matrix
 Boron carbide
 Titanium carbide
 Titanium diboride
 Hardness
 Wear resistance
 Strength

Funding

The work was carried out on the equipment of the Plastometriya Shared Use Center of the IMASH UB RAS in accordance with the state assignment on the topic No. AAAA-A18-118020790145-0.

Acknowledgements

The authors are grateful to Ph.D. R.A. Savrai and I.A. Zabolotskikh for help with the experiments.

ABSTRACT

Introduction. The development of new wear-resistant materials obtained by the method of self-propagating high-temperature synthesis (SHS) is an urgent problem in materials science. The SHS method is most widely used in the field of creating new powder materials. Much less attention has been paid to the production of monolithic non-porous composites. For monolithic composites, it is very important to identify the role of the metal matrix and phase transformations in the process of secondary structure formation after the completion of the synthesis process when the obtained material is cooled. **The aim of this work** was to carry out a comparative analysis of the structure and properties of SHS composites of the Fe-Ti-C-B, Fe-Ni-Ti-C-B, Fe-Ni-Cr-Ti-C-B, and Cu-Ti-C-B systems. **Materials and research methods.** Composites were obtained from powder mixtures consisting of thermoreactive components Ti, C, and B, as well as matrix Fe, Fe-Ni, Fe-Ni-Cr, and Cu. The initial powders were thoroughly mixed, loaded into a steel tube container, and the powder mixture was preliminary compacted. Then, the workpieces were heated in an electric furnace to the temperature of the onset of autoignition. After completion of the SHS, the workpieces were deformed with a force of 250 MPa in a hydraulic press at a temperature not lower than 1000 °C. Samples were cut from the obtained sandwich plates for microstructural studies, density determination, hardness measurements, transverse bending tests and abrasive wear resistance tests. **Results and discussion.** All investigated composites were characterized by an uneven distribution of strengthening particles TiC and TiB₂ over the volume. The use of the Fe-Ni matrix led to the formation of regions with the γ -Fe + Fe₂B eutectic structure in the composite and an additional strengthening phase Ni₃Ti. The use of Fe-Ni-Cr metal-matrix components led to the formation of two solid solutions in the matrix - austenite and ferrite, and Cr₂₃C₆ particles were formed along the boundaries of austenite grains. The maximum transverse bending strength was shown by SHS composites of the Fe-Ti-C-B and Cu-Ti-C-B systems with a matrix of FCC solid solutions. All composites had a hardness of 66–72 HRC and showed the same abrasion resistance.

For citation: Pugacheva N.B., Nikolin Yu.V., Bykova T.M., Senaeva E.I. Influence of the chemical composition of the matrix on the structure and properties of monolithic SHS composites. *Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science*, 2021, vol. 23, no. 3, pp. 124–138. DOI: 10.17212/1994-6309-2021-23.3-124-138. (In Russian).

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Introduction

The technological development of new composite materials formed by the SHS method has been carried out for about 50 years, but there are still many unresolved issues. Throughout the development of wear-resistant *SHS*-composites, main focus is usually drawn towards the hardening phase – carbides and borides, which ensures high values of hardness and wear-resistance [1-5]. *SHS* method is mostly known in the area of development of new powder materials [6-8]. There is not enough attention paid to monolithic *SHS*-composites, which are used mostly as instructional materials. The expansion of the scope of application of monolithic *SHS* composites, associated with the expansion of the range of its strength characteristics, makes it important to identify the role of the metal matrix. Phenomenon of secondary pattern formation is not studied enough in such composites. Data around the influence of eutectic systems that inevitably happen during the synthesis of multicomponent systems, such as Fe-Ni-Ti-C-B and Ni-Cr-Ti-C-B [9–16] is contradictory. Particular difficulties exist when it comes to obtaining non-porous monolithic composites, used for the details and construction elements, experiencing abrasion on a large surface area. A danger of cracking and rapid destruction exists with the mechanical loading, particularly with shock loads. In order to reach a satisfactory combination of wear-resistance and durability, it is important to pay particular attention to metal matrix and processes happening during the secondary structure formation within the metal matrix as well as discovering additional ways for structural changes, chemical composition and matrix properties, which eventually leads to changes of composition characteristics as a whole. In connection with the above, studies of the influence of a metal matrix on the properties of *SHS* composites are relevant, since it will make a certain contribution to the creation of scientific foundations for the production of highly wear-resistant materials with high strength indicators.

The main aim of this work was to conduct a comparative analysis of the structure and properties of *SHS* composites of the Fe-Ti-C-B, Fe-Ni-Ti-C-B, Fe-Ni-Cr-Ti-C-B and Cu-Ti-C-B systems.

Materials and research methods

Monolithic *SHS* composites of different chemical composition obtained by the technology described in [23, 24] are studied. Initial powder mixture consists of thermotactic and matrix components. Thermally reactive components (TRC) are titanium powders, boron carbide B_4C . Percentage calculation of those components in TRC was made on the condition of a reaction (1) in stoichiometric proportion:



Matrix components are powders Fe, Cr, Ni, Cu. The following powders were utilized: titanium PTM-1 (particle size 15-45 μm), boron carbide M20 (12-20 μm), technical carbon P-804T (1–4 μm), iron PGRV-3 (40–100 μm), nickel PNK-UTZ (1–20 μm), copper PMR-1 (40-100 μm), chrome PH1M (20–100 μm). Initial powders were put into a ball mill with a 5-liter capacity together with grinding balls made out of ball-bearing steel SHH15. Powder mass ratio in relation to the mass of balls was 1:3. Mixing type: dry. Mixing time: 12 hours.

It was previously shown that the minimum porosity of the Fe-Ni-Ti-C-B composites is obtained when the content of TRC in the powder mixture is no more than 30 wt. % [25–29]. The chemical composition of powder mixtures for the production of *SHS* composites is shown in Table 1.

Steel pipe container made of a low-carbon structural steel (St3 grade) was filled with the obtained powder mix. Initial compacting of the powder mix was achieved with a special snap. Then the initial sample was immersed in an electric furnace and heated up to the temperature of exothermic reaction (1,030 °C). After *SHS* completion hot sample was transferred to a hydraulic press and compacted with a weight of not less than 250 MPa, in order to eliminate porosity. As a result, sandwich plates were obtained, the appearance of which is shown in Fig. 1.

Structure of those composites was investigated on a scanning electron microscope TESCAN VEGAII XMU. Rockwell hardness was measured on a hardness tester. Local chemical composition of phases was

Table 1

The chemical composition of powder mixtures for obtaining SHS composites, mass. %

No.	TRC (Ti + C + B)	Fe	Ni	Cr	Cu
1	30	70	0	0	0
2	30	50	20	0	0
3	30	35	20	15	0
4	30	0	0	0	70

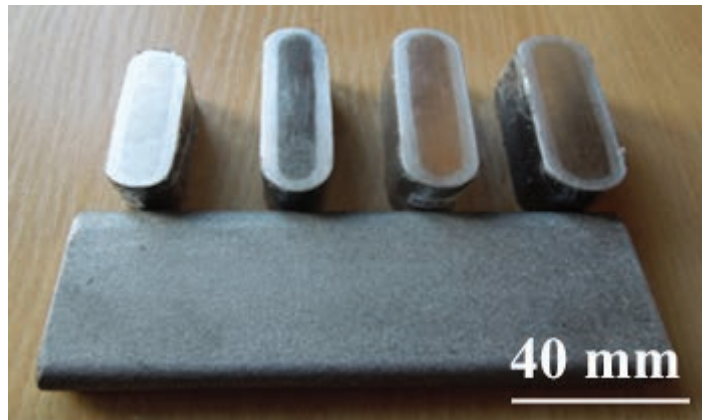


Fig. 1. Appearance of sandwich panels made of SHS composites

determined by energy and wave dispersive spectroscopy by OXFORD. Phase X-ray diffraction analysis was performed on a SHIMADZU X-ray diffractometer in $K\alpha$ chromium radiation.

Density was determined using hydrostatic weighing of the samples in the air and in the distilled water. Weighing was carried out using analytical weights Ohas Pioner PA 214. Density of the composite was determined using the following formula:

$$\rho = \frac{m_1}{m_1 - m_2} \rho_B, \quad (2)$$

where m_1 – mass of the sample in the air, m_2 – mass of the sample in the water, ρ_B – density of the distilled water ($\rho_B = 998 \text{ kg/m}^3$). Sample error was no more than 0.2 % from the measured value.

Lateral bend trials were conducted using GOST 20019-74 “Sintered hard alloys. Transverse bending strength trial method”. Trials were conducted on samples Type A (dimensions 35x5x5 mm), trial speed was 0.2 mm/min, distance between support axis was 30 mm. Transverse bending strength R_{bm} 30 was calculated using the following formula:

$$R_{bm} = \frac{3Fl}{2bh^2}, \quad (3)$$

where F – the greatest force, corresponding to the moment of destruction of the supports, N; l – distance between support axes, mm; h – height of the sample (size, corresponding to the direction of force during the trial), mm; b – width (size, corresponding to the perpendicular height), mm.

Abrasive wear resistance tests were carried out according to the procedure described in detail in [26, 27]. The wear resistance of the samples was evaluated by the relative change in mass (Δm_{rel}), which was calculated by the formula:

$$\Delta m_{rel} = \Delta m / m_0, \quad (4)$$

where m_0 – initial mass of the sample; m – mass of the sample after the trials.

Besides that, the values of the friction coefficient (K) were calculated using the following formula (5) and relative wear (ε) using the following formula (6):

$$K = F_{fr}/N; \quad (5)$$

$$\varepsilon = \Delta m / \Delta m_{ben}, \quad (6)$$

where F_{fr} – friction force, N; N – normal pressure force, N; Δm – sample weight loss, Δm_{ben} – benchmark weight loss (a sample made out of steel 40Cr was selected as a benchmark).

Wear-surface after the trials was explored using optical interferometer-profilometer Wyko, which was also used to determine the surface roughness Ra .

Results and its further discussion

Heating conditions and geometric size of the samples with different powder mixture compositions were identical. Hot compaction immediately after the completion of the synthesis allows us to obtain dense and non-porous composites. In all the studied composites, reinforcing phases are carbide particles TiC and diboride titanium TiB_2 . Those phases are formed as a result of exothermic reactions (1), which one can imagine as a combination of chemical reactions:



Grey-colored TiC particles are sized between 0.5 and 2 μm and resemble a shape of a sphere (Fig. 2, *a*). Black-colored TiB_2 particles are sized between 2–10 μm and resemble a shape of a cube (Fig. 2, *b*). In all studied composites, the distribution of reinforcing particles is uneven in volume: some areas contain mostly TiC particles, some others – TiB_2 . It is obviously associated with heterogeneous carbon (soot) and boron carbide B_4C distribution in the volume of the original powder mixture, despite thorough mixing of the initial powders before filling a steel casing. The reason for this is the tendency for multi-component powder mixtures, containing different particle shapes, dispersion and bulk density, for mechanical segregation. It is caused by external forces mostly. In our case, gravity is the main force (the process of pouring the powder mixture from the mixer into the pipe container) and pressure power compaction on a hydraulic press). In the areas with predominant soot content a chemical reaction, takes place (7), but in the areas with higher boron carbide concentration the following chemical reaction, takes place (8).

Under the same conditions of formation of *SHS*-composites, different chemical matrix composition affects the structure and nature of the distribution by volume of the material obtained. Depending on the chemical composition, matrix of composites consists of different phases. Composite Fe-Ti-C-B (obtained from powder mixture 1 in table 1) contains an austenitic matrix and reinforcing TiC and TiB_2 particles, which is characterized by hardness of 63-6 HRC and density of $R_{bm}30 = 800$ MPa. The microstructure of the composite is shown in the Figure 3, *a*.

Nickel addition to the matrix composition (powder mixture No. 2 in table 1) led to the formation of separate areas with eutectic structure $\gamma\text{-Fe} + Fe_2B$ in the composite with a particular skeletal structure (Figure 3, *b*). Conditions for the formation of such a structure were earlier described [25]. Dendritic axes are enriched in iron and contain boron. Interdendritic spaces are austenite (Figure 4 and table 3). Separate micropores sized no more than 5 μm are fixed in areas with eutectics $\gamma\text{-Fe} + Fe_2B$. Perhaps the relatively low bending strength of this composite $R_{bm} = 620$ MPa is associated with the formation of eutectic structures, the destruction of which requires a bit of work (see Table 2).

The basis of the metal matrix of the Fe-Ni-Cr-Ti-C-B composite system formed from a powder mixture of composition 3 (see Table. 1), is austenite (indicated by the number 1 in Fig. 5, *a*). In addition,

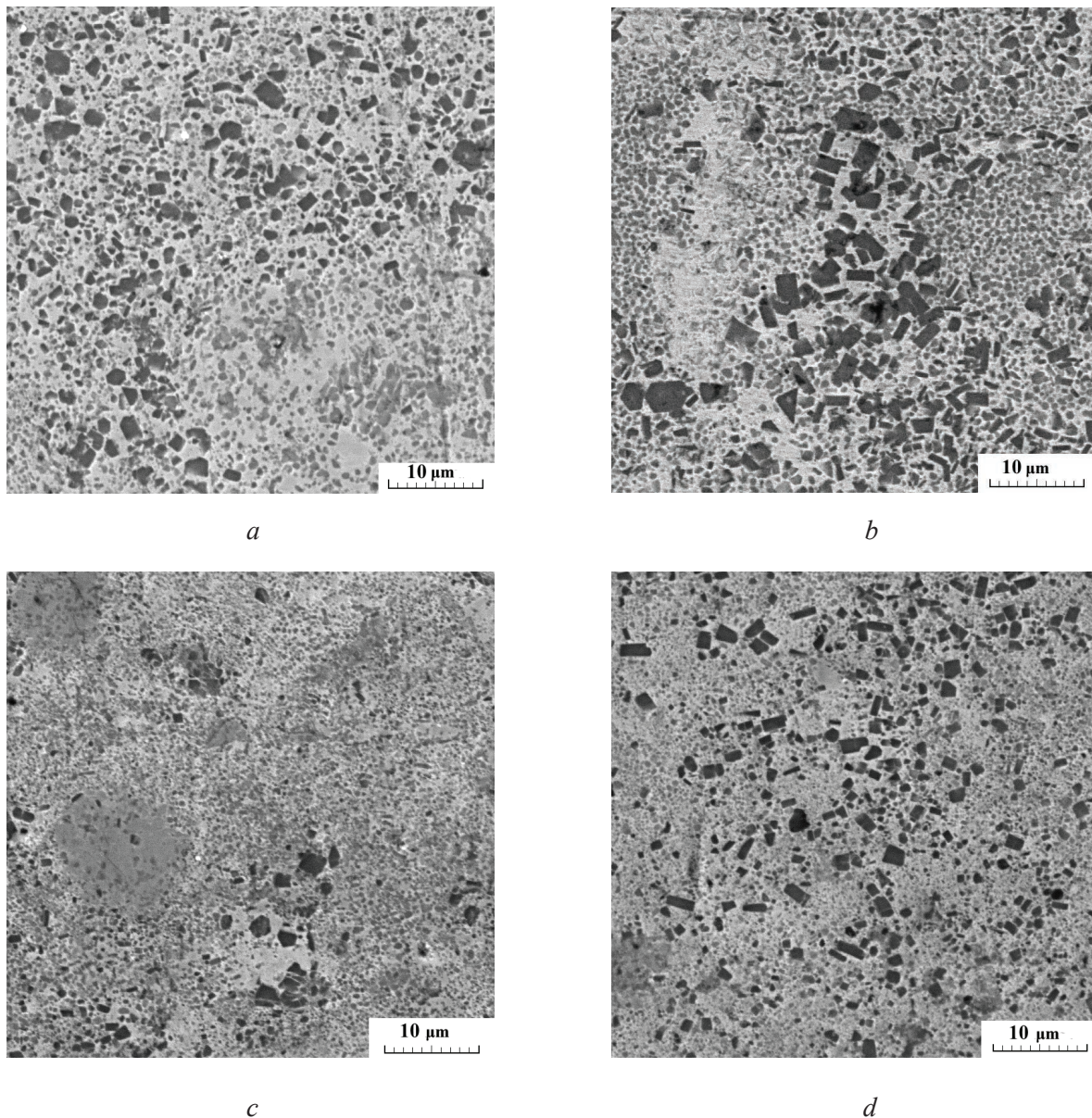


Fig. 2. Morphology and distribution of particles of strengthening TiC and TiB_2 phases in the studied SHS composites:

a – Fe matrix; *b* – Fe + Ni; *c* – Fe + Ni + Cr; *d* – Cu

areas of chromium ferrite were formed in the matrix (indicated by the number 2 in Fig. 5, *a*). Chromium is also involved in the formation of Cr_{23}C_6 carbides, which are separated along the boundaries of austenitic grains (thin layers enriched with chromium, in Fig. 5, *c*). Titanium carbide and diboride particles are formed mainly in a solid solution based on Ni (Fig. 2, *b* and 5, *d*, *e*). The composite is quite dense, single micropores with a maximum size of 5 μm are recorded, located mainly between clusters of TiB_2 particles. Phase X-ray diffraction analysis showed the presence of Ni_3Ti intermetallide, as in the composite of composition 2. The hardness of the composite of composition 3 was 66-72 HRC, and the bending strength $R_{bm30} = 670 \text{ MPa}$ (Table 2).

Melting temperature of copper (1,083 °C) is significantly lower than iron (1,538 °C), chromium (1,907 °C) and nickel (1,455 °C). In this regard, with the same thermal effect of the SHS process of all the studied powder compositions (the content of TRC in the mixture is the same), we should expect significantly different conditions for the formation of the composite structure. A micro-X-ray spectral study of a composite with a copper matrix obtained from a powder mixture of composition 4 (Table 1) revealed the presence of

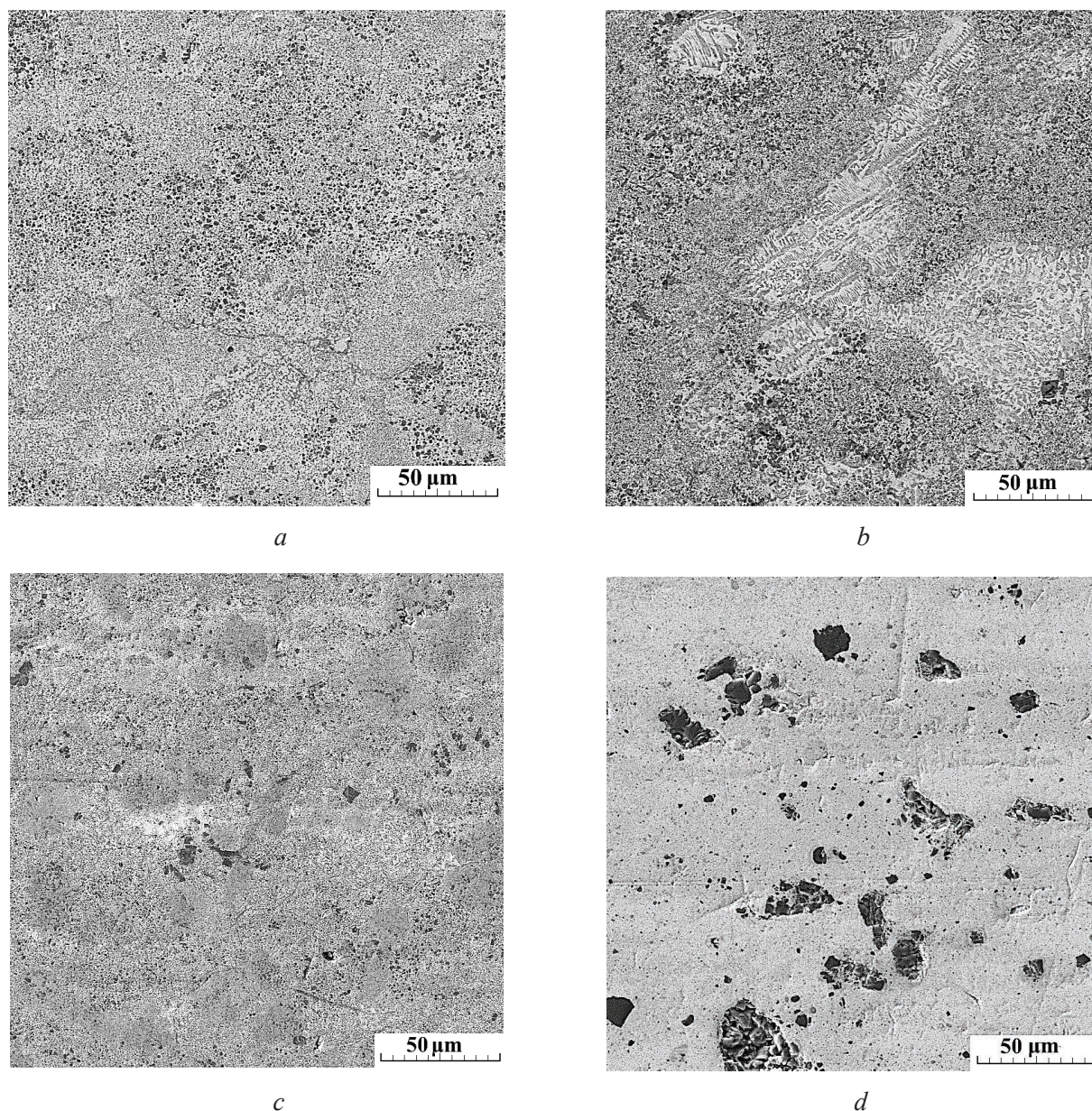


Fig. 3. Microstructure of SHS composites with a matrix of different chemical composition:
a – Fe matrix; *b* – Fe + Ni; *c* – Fe + Ni + Cr; *d* – Cu

separate clusters of boron carbide particles that did not have time to fully react according to the reaction (7). Probably, the following reaction took place:



Particles of B_4C and boron were recorded based only on the results of SEM analysis on separate surface areas of the thin section of the composite system Cu-Ti-C-B (Fig. 6, Table 4). Nevertheless, the strength of this composite turned out to be the maximum of all the studied $R_{bm} = 830 \text{ MPa}$ (see table 2).

Abrasion resistance of all explored SHS-composites turned out to be about the same (table 5). The relative wear resistance was calculated in comparison with indicators of the structural steel 40Cr samples, thus its' $\varepsilon = 1$. Silicon carbide is harder (3,000-3,200 HV) in comparison to electro corundum (2,000-2,300 HV) [28], hence for all tested composites, the relative wear ε when tested with silicon carbide is higher. The coefficient of friction is at the level of 0.5 for all the studied composites. The minimum surface roughness is typical for a composite with a copper matrix.

Table 2

Phase composition, hardness and density of SHS composites

No.	Matrix elements	Phase composition	HRC	ρ , kg/m ³	R_{bm} 30, MPa
1	Fe	γ -solid solution Ti in FCC lattice of Fe, TiC, TiB ₂	63–68	6500	800
2	Fe-Ni	γ -solid solution Ni in FCC lattice of Fe, Fe ₂ B, TiC, TiB ₂ , Ni ₃ Ti	66–70	7000	620
3	Fe-Ni-Cr	γ - solid solution Ni in FCC lattice of Fe, α - solid solution Cr in bcc lattice of Fe, TiC, TiB ₂ , Ni ₃ Ti, Cr ₂₃ C ₆	68–72	6900	670
4	Cu	solid solution Ti in FCC lattice of Cu, TiC, TiB ₂ , B ₄ C, B	65–70	6700	830

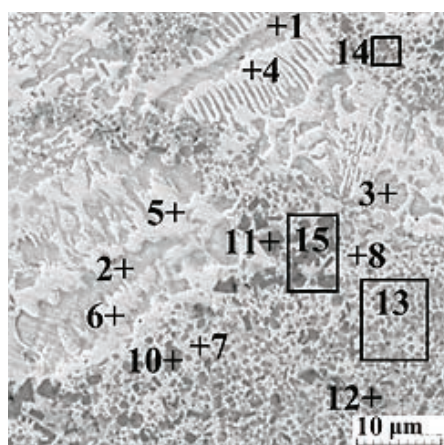
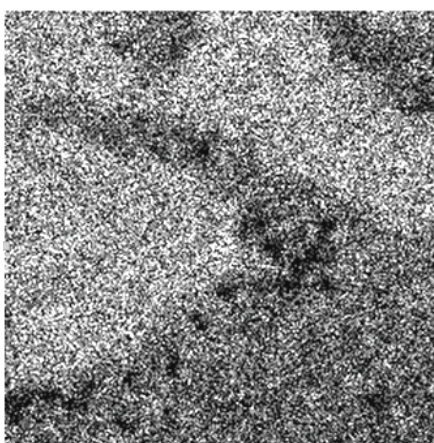
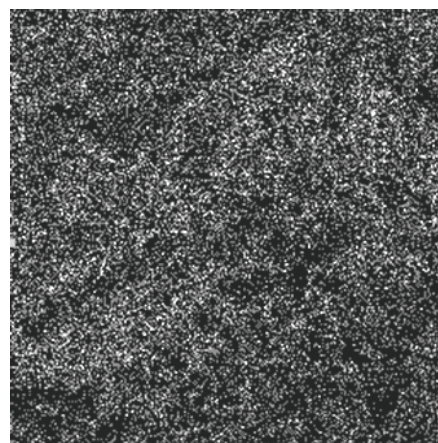
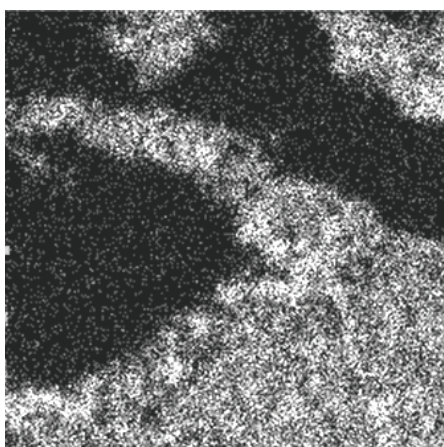
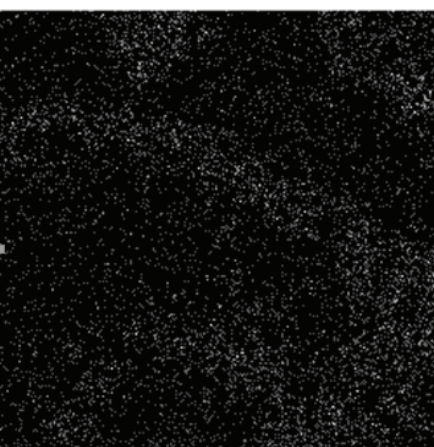
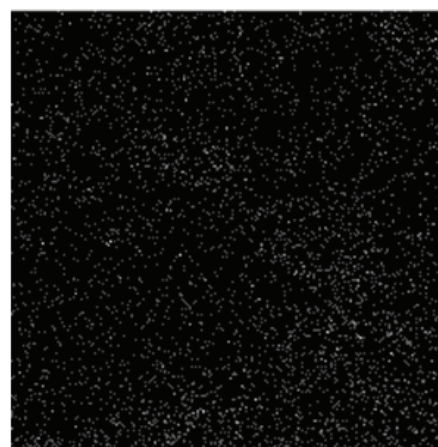
*a**b**c**d**e**f*

Fig. 4. Chemical elements distribution in the regions with the dendritic structure of the composite Fe-Ni-Ti-C-B:

a – image in secondary electrons; *b* – in the characteristic X-ray radiation Fe; *c* – Ni; *d* – Ti; *e* – C; *f* – B

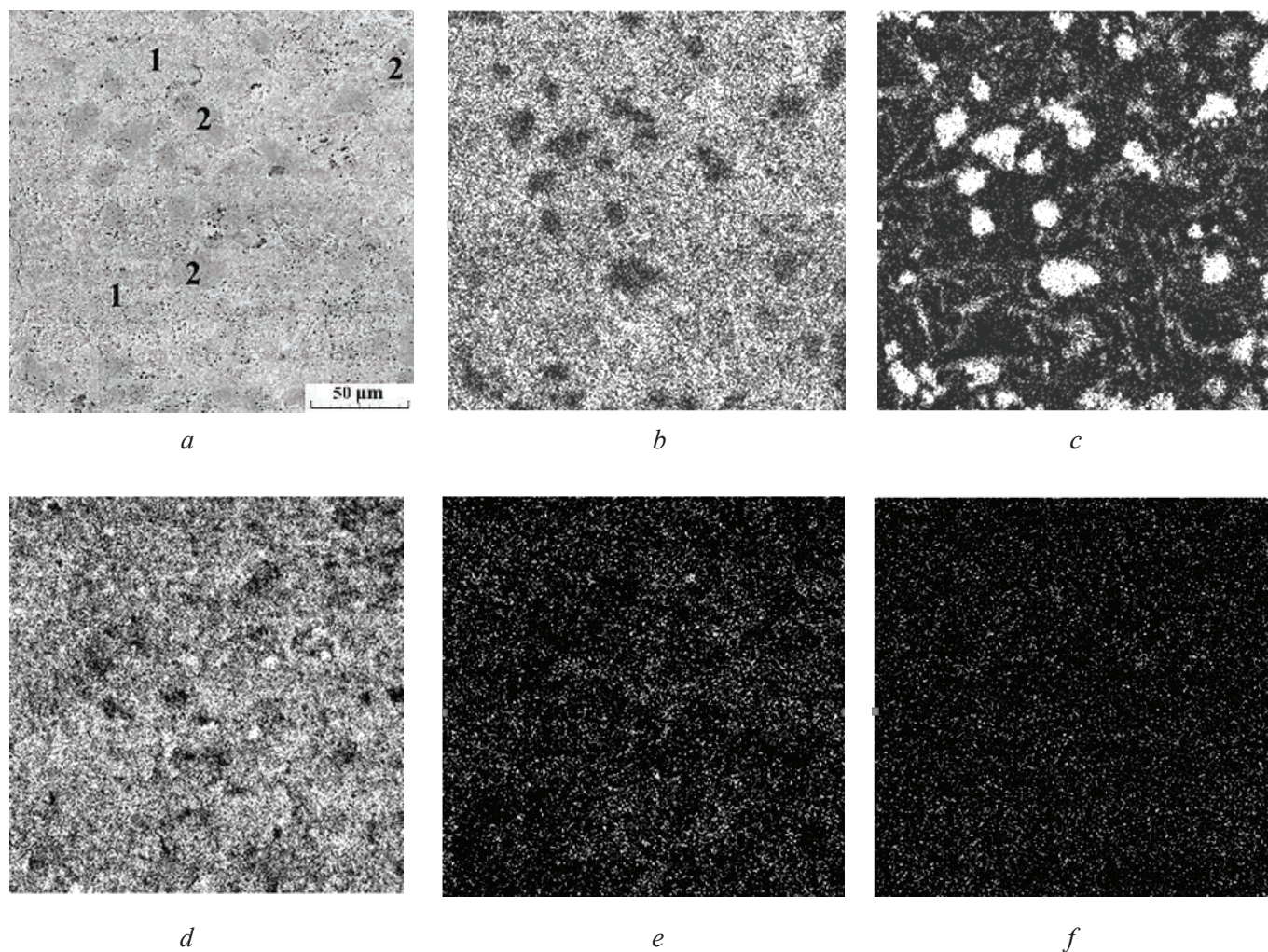


Fig. 5. Chemical elements distribution in the composite Fe-Ni-Cr-Ti-C-B:
a – image in secondary electrons; *b* – in the characteristic X-ray radiation Fe; *c* – Cr; *d* – Ti; *e* – C; *f* – B

Conclusions

In all the studied composites, the main reinforcing phases are particles of titanium carbide TiC and titanium diboride TiB₂, formed as a result of the chemical interaction of the initial powders and unevenly distributed in the volume of the composites. The matrix of composites consists of solid solutions based on crystal lattices of Fe, Cr, Ni or Cu. The more metals are included in the matrix, the more heterogeneous the composite is in chemical composition. The Fe-Ni-Cr-Ti-C-B composite system is characterized by the maximum chemical heterogeneity.

The introduction of Ni into the iron matrix led to the formation of regions with the eutectic structure of γ -Fe + Fe₂B and an additional strengthening phase-the intermetallic Ni₃Ti.

The matrix of the Fe-Ni-Cr-Ti-C-B composite system consists of two solid solutions: based on austenite (Fe-Ni) and based on ferrite (Fe-Cr). Chromium carbide Cr₂₃C₆ particles formed along the boundaries of austenitic matrix grains were added to the reinforcing particles TiC, TiB₂ and Ni₃Ti.

When forming a composite of the Cu-Ti-C-B system, the synthesis reaction of titanium carbide and diboride was not fully realized. In addition to the TiC and TiB₂ phases, particles of unreacted initial boron carbide B₄C were recorded in separate volumes of this composite, around which a thin layer of a Cu-based solid solution with boron particles was formed.

SHS composites with a matrix consisting of a solid solution with a FCC crystal lattice showed the greatest strength when tested for transverse bending. The abrasive wear resistance of all the studied composites is at the same level. The minimum roughness after testing was recorded for a composite with a copper matrix.

Table 3

The chemical composition of the composite in the areas marked in Fig. 4 a, at. %

No.	B	C	Ti	Fe	Ni	Phases
1	31.83	0	0.48	66.81	0.88	Fe ₂ B
2	32.15	0	0.43	66.80	0.62	
3	31.98	0	0.54	66.20	0.78	
4	0	5.74	0.50	65.54	28.22	γ -Fe
5	0	5.50	0.46	64.04	30.00	
6	0	5.22	0.73	66.40	27.65	
7	0	15.90	12.25	51.65	23.50	γ -Fe, TiC
8	0	22.36	11.93	55.85	21.56	
9	17.50	20.85	22.24	33.65	5.76	γ -Fe, TiC, TiB ₂
10	56.78	0	27.34	11.14	4.74	TiB ₂
11	62.64	0	24.68	9.70	2.98	
12	60.73	0	25.24	10.85	3.18	
13	0	28.65	24.26	35.11	11.98	γ -Fe, TiC
14	43.90	12.00	16.50	32.83	7.06	γ -Fe, TiB ₂ , TiC
15	47.75	11.65	19.42	30.31	5.92	

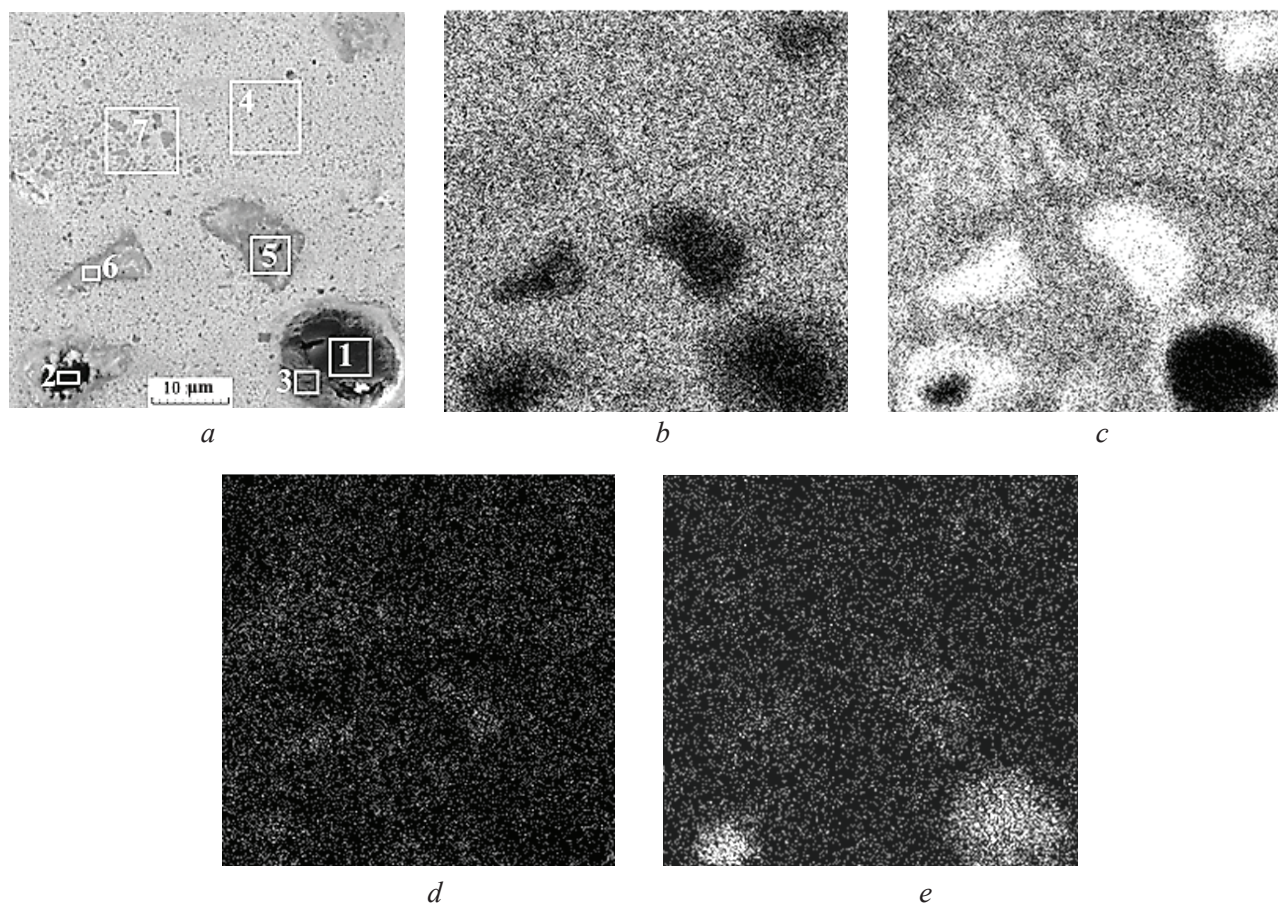


Fig. 6. Chemical elements distribution in the composite Cu-Ti-C-B:

a – image in secondary electrons with the microanalyses regions; *b* – in the characteristic X-ray radiation Cu;
c – Ti; *d* – C; *e* – B

Table 4

The chemical composition of the structural components of the Cu-Ti-C-B composite shown in Fig. 6 a, at. %

No.	B	C	Ti	Cu	Phases
1	78.7	22.3	0	0	B ₄ C
2	84.8	21.2	0	0	
3	88.2	0	0	11.8	B + Cu
4	17.9	18.4	17.5	46.2	Cu + TiB ₂ + TiC
5	35.5	24.6	33.5	6.4	
6	21.2	21.4	31.6	25.8	
7	32.40	17.28	35.15	15.17	

Table 5

Abrasion Resistance Test Results

№	The composition of the matrix	$\Delta m/m_0$, %	ε	K	R_a , μm
1	Fe	0.7 ¹	1.84	0.51	1.53
		1.3 ²	0.87	0.57	
2	Fe-Ni	1.8	1.95	0.52	1.53
		2.5	1.39	0.55	
3	Fe-Ni-Cr	0.4	3.16	0.45	1.49
		1.0	1.24	0.50	
4	Cu	0.6	1.96	0.49	1.1
		1.2	0.99	0.56	
5	Steel 40Cr	1.3	1	0.65	2.0
		1.3		0.66	

¹ tests with electro corundum; ² tests with silicon carbide

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Conflicts of Interest

The authors declare no conflict of interest.

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