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The effect of the introduction of B_4C on the adhesive and cohesive properties of self-fluxing coatings

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Abstract. Wear and tear of technical equipment elements is a serious problem that negatively affects the productivity of production processes. The work analyzed the strength of cohesive and adhesive bonds of wear-resistant coatings obtained by detonation gas-dynamic spraying method based on a self-fluxing alloy, depending on the introduction of B_4C particles into the composite composition. The chemical composition of the cohesive bonds of the resulting coatings and the effect of boron-containing elements on their strength were analyzed. The method of transverse scratching of transverse sections of thick coatings is a qualitative assessment of adhesive and cohesive properties and helps to determine the mechanisms of their destruction. The purpose of the work is to study the effect of the introduction of B4C particles on the adhesive and cohesive properties of coatings obtained by detonation gas-dynamic spraying based on self-fluxing NiCrBSi alloy. Methods for studying coating samples obtained by detonation gas-thermal method: scanning electron microscopy, energy dispersive analysis, optical metallographic microscopy, scratch testing. Previously, wear-resistant coatings based on self-fluxing NiCrBSi-B4C alloy have proven their effectiveness as protective coatings for centrifugal beet cutter knives. This study substantiates the feasibility of obtaining a composite based on such coatings with the introduction of boron carbide to improve its strength characteristics.
Keywords: detonation gas-dynamic spraying method, thermal spraying, self-fluxing alloys, wear-resistant coatings

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1. INTRODUCTION

As a rule, detonation gas-dynamic spraying of NiCrBSi-B4C composite coatings on the surface of centrifugal beet cutter knives significantly increases their service life [1]. However, carrying out full-scale tests gives only a general idea of the effectiveness of wear-resistant coatings. Quantitative

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assessment of the characteristics of such coatings can be realized by various methods, such as corrosion resistance test [2, 3], tribological characteristics test [4, 5], hardness test [6, 7], ductile fracture test [8, 9], etc. [10-12]. The most important properties that wear-resistant coatings should have are adhesion and cohesion, since the low strength of the coating bond with the substrate material or the boundaries of the material itself, other characteristics are of no importance.

There are also some methods for measuring adhesion and cohesion characteristics. Standard ones include tensile and flexural tests. In this paper, the method for assessing the adhesion and cohesion of a coating by scratching was used, which allows for observations of the nature of the destruction of adhesive and cohesive bonds, and the method is also simple and easy to use [13-15]. This method is standardized in ISO 27307 [16] and is used to analyze coatings obtained by thermal spraying methods, with a thickness of 50 μm to 1000 μm. Thus, using the example of [1], obtaining a composite coating based on a self-fluxing NiCrBSi alloy containing B4C particles can positively affect the hardness of the coating [17, 18], while negatively affecting the adhesive and cohesive properties [19, 20]. Therefore, it is important to study the effect of introducing B4C particles as a filler that increases the hardness of the coating on the adhesive and cohesive properties of composite coatings based on self-fluxing alloys obtained by the detonation gas-dynamic method. This paper presents the results of studying coatings with two compositions: without adding boron carbide – NiCrBSi and with adding boron carbide particles - NiCrBSi-B4C, obtained by the detonation gas-dynamic method. Comparisons of the adhesive and cohesive properties of these coatings were made according to the ISO standard (ISO 27307). The nature of cohesive and adhesive destruction of the coatings was analyzed.

2. METHODS AND MATERIALS

NiCrBSi (PR-NH17SR4, 0-40 μm, Polema, Tula) and B_4C (F1200 FEPA, 20-40 μm, Polema, Tula) powders were used as precursors to obtain the coatings. The granulometric composition of the powders was analyzed using a laser particle size analyzer (Analysette 22 NanoTec Plus Frisch, Germany). The NiCrBSi-B_4C powder composition in a ratio of 90/10 wt. %, respectively, was mixed in a Fritsch Pulverisette 6 planetary mill at a mass ratio of balls and mixture of 2:1 at a speed of 250 rpm for 5 min. The coatings were formed by the high-speed detonation spraying method on medium-carbon steel 40G based substrates with dimensions of 40 by 40 mm using a robotic complex for detonation spraying of coatings (BSTU named after V. G. Shukhov) [1, 21], which is part of a unique scientific installation. The parameters of the spraying process are presented in Table 1.

Table 1. Spraying process parameters.

Powder type	Fuel mixture components consumption (m^3/hour)			Powder consumption (g/hour)	Spraying distance (mm)
	Air	Oxigen	Propan		
NiCrBSi	1.56*/1.39**	3.13*/3.25**	0.71*/0.75**	243	55
NiCrBSi-B_4C	1.56*/1.39**	3.13*/3.25**	0.71*/0.75**	214	55
* Cylindrical combustion chamber; ** Annular combustion chamber					

To analyze the microstructure, the obtained coating samples were cut into 4 parts measuring 20x20 mm. To determine the adhesion and cohesion parameters, 5x20 mm transverse sections were additionally prepared using an IsoMet 5000 precision cutting machine. To prepare the transverse sections, the obtained samples were filled with epoxy resin (universal two-component epoxy adhesive brand EDP, "EKOKLASS", Technical Specifications 2252-003-62517430-2020) into a tablet. The resulting tablet was polished with a BUEHLER polishing machine (Vector POWER HEAD) using diamond discs with a decrease in the diamond grain size in the following order: 75, 54, 18, 6, 3, to achieve a surface roughness of Ra 0.03 μm. The microstructure of powder precursors, their mixtures, coatings and the nature of cohesive failure were studied by scanning electron microscopy using a Mira 3 LMU scanning electron microscope (Tescan, Czech Republic). To obtain high-quality SEM images, a detector of reflected electrons was used in high-resolution mode at an accelerating voltage of 15 kV.

The elemental composition of the coating samples was studied by energy-dispersive spectroscopy (EDS) in an AZtec 3.1 microanalysis system using an X-Max 50 detector (Oxford Instruments NanoAnalysis, High Wycombe, England). The accumulation of EDS spectra and elemental composition distribution maps was performed at an accelerating voltage of 15 kV, the beam current was selected so that the signal level was about 4000-5000 pulses per second. Scratch tests of transverse sections of the obtained coatings were performed using a multi-functional tribometer (MFT 2000A). The study of scratches on the coatings in the visible field was carried out by an MT-24RF optical metallographic microscope (SIAMS, China) using the SIAMS 800 software package (SIAMS, Russia). According to the ISO 27307 standard, two types of failure can be defined: within the coating (cohesion) and at the coating-substrate boundary (adhesion). The types of such failures are schematically shown in Fig. 1.

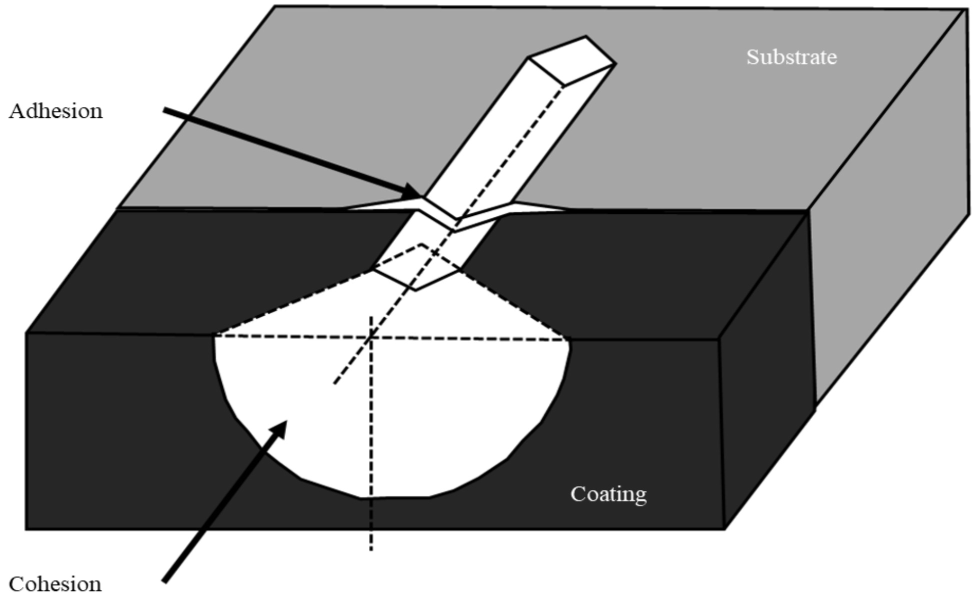


Fig. 1. Adhesive and cohesive failure in cross-sectional scratch.

The scratch test was performed under constant load by moving the indenter from the substrate, through the substrate/coating interface, and then through the entire coating to the tablet edge. The following test parameters were used to obtain a scratch on each sample: Rockwell diamond indenter with a radius of 200 μm, constant normal loads of 3, 5, 10, 13, 15, 20 and 23 N, scratch length of 2 mm, indenter speed of 6 mm/min. After visual inspection, the geometric values of the side lengths of the resulting cone projection Lx, Ly were measured using a metallographic microscope. Using the obtained values, the area Acn = Lx * Ly was calculated. The cone projection area (Acn) was chosen as the most objective result for comparing the adhesion strength values, since it has a monotonic dependence on the scratch load [16]. Analysis of the obtained area of the cone projection allows evaluating the cohesion of the lamellas of the obtained coatings, where the larger the area, the lower the adhesion strength between the lamellas.

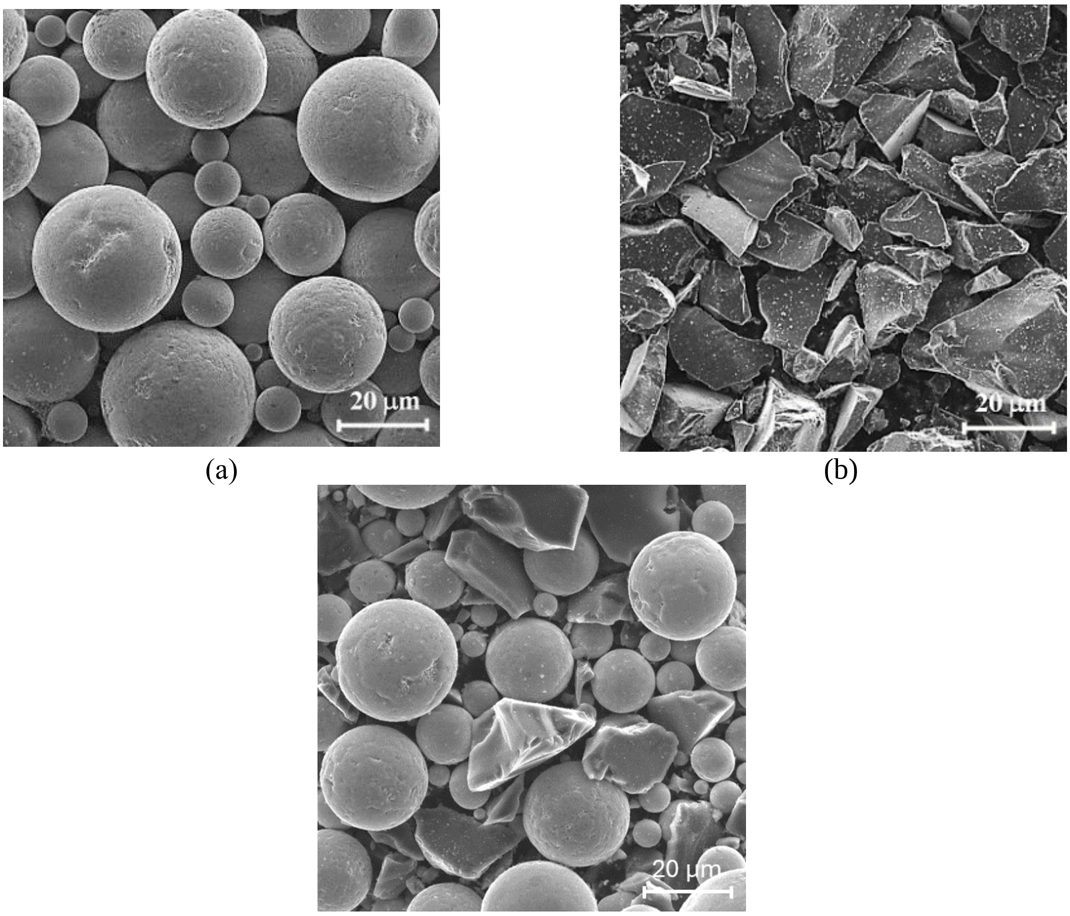
3. RESULTS AND DISCUSSION

The results of granulometric analysis of powders are presented in Table 2. The initial NiCrBSi powder has a dispersion of mainly 20 - 40 μm, which corresponds to the declared characteristics of the manufacturer. NiCrBSi-B_4C powder has a reduced particle fraction due to the volume content of B_4C of fine dispersion.

Table 2. Powders used for the coatings production.

Powder type	Granulometry		
	d(10)	d(50)	d(90)
NiCrBSi	10.29	23.23	44.03
NiCrBSi-B_4C	3.66	16.86	36.1
B_4C	0.29	3.08	13.17

The main purpose of mixing the initial powders is to evenly distribute and introduce finely dispersed B_4C into the spherical particles of NiCrBSi. Also, when spraying, this can have a positive effect on the amount of material in the resulting coating by reducing the required thermal energy for melting or deformation of the sprayed mixture. Fig. 2 shows the results of SEM-analysis for powder precursors and their mixtures.



(a) (b)

(c)

Fig. 2. Microstructure of the powder precursors: (a) NiCrBSi, (b) B_4C and their mixtures: (c) NiCrBSi-B_4C.

The presented SEM images of the microstructure of the powder precursors and their mixture show that NiCrBSi (Fig. 2.a) has a spherical structure; B_4C powder (Fig. 2.b) is presented in the form of fragments; NiCrBSi-B_4C powder (Fig. 3.c) characteristically combines the structures of the original powders. The results of the electron microscopic images are consistent with the results of granulometric studies. However, in the resulting mixture, mechanical introduction of fragmented B_4C particles into spherical NiCrBSi particles did not occur; in general, the resulting mixture consists of uniformly dispersed particles, where spheres predominate. Fig. 3 shows the results of SEM analysis for transverse sections of the obtained coatings.

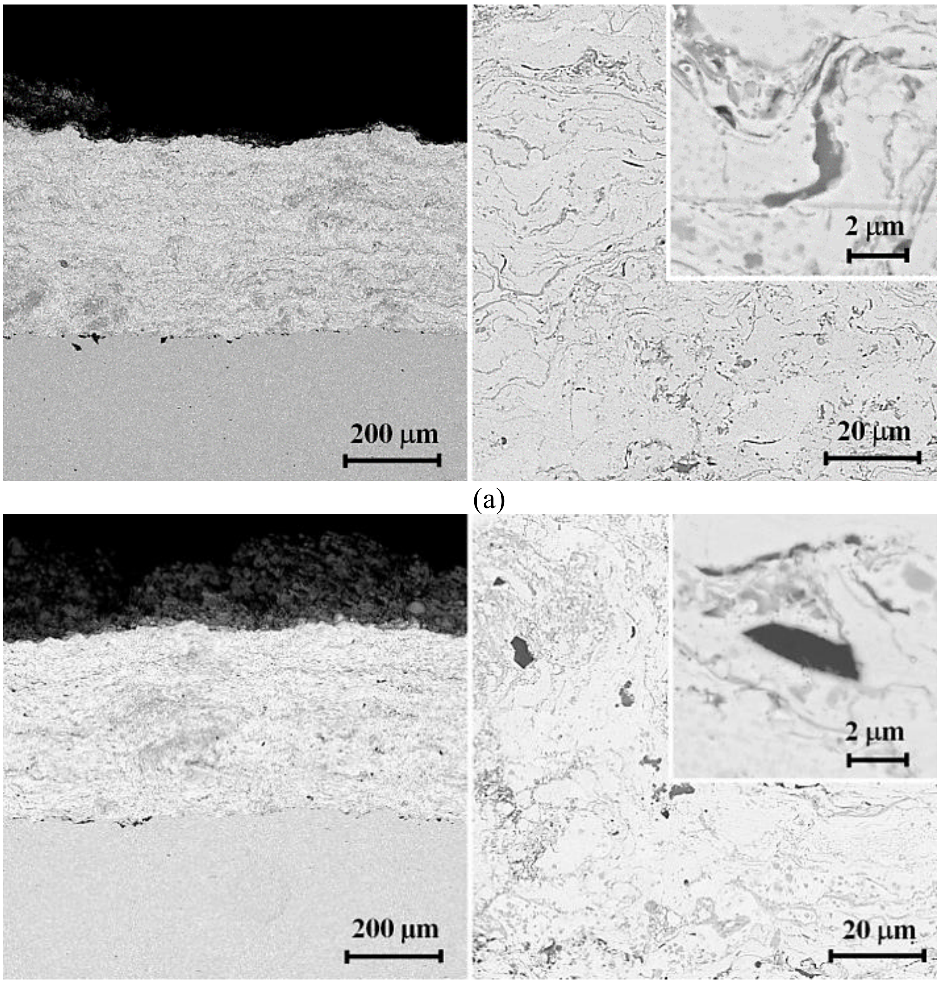


Fig. 3. Microstructure of transverse sections of coatings: (a) NiCrBSi, (b) NiCrBSi-B_4C.

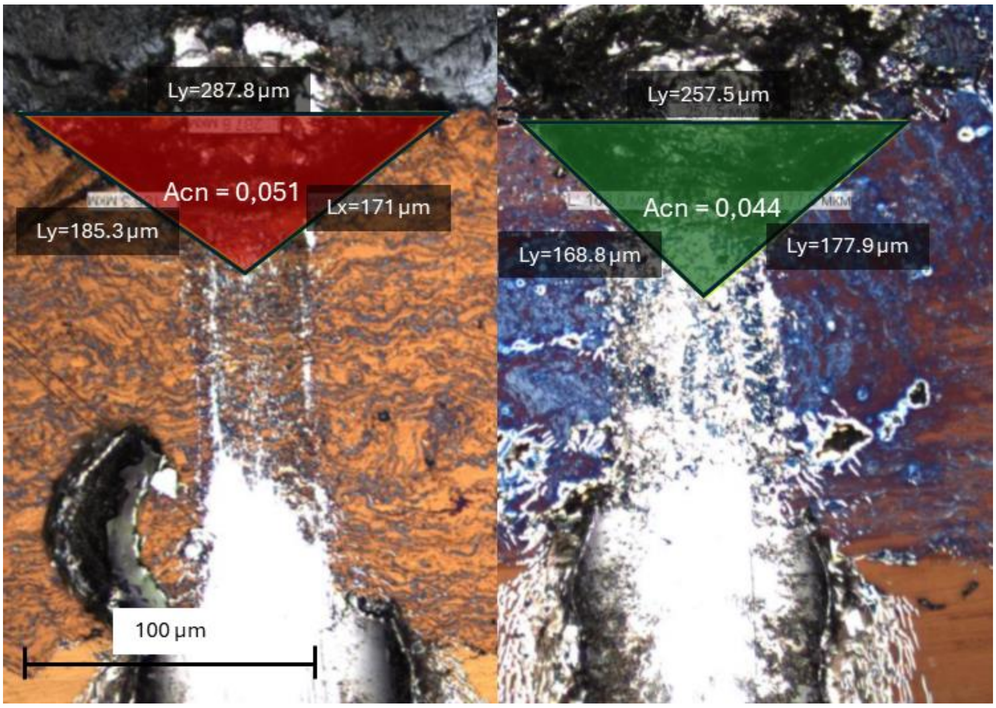
The coating based on the self-fluxing NiCrBSi powder (Fig. 3.a) has a pronounced lamellar structure, about 300 μm thick and without cracks. There are pores at the coating/substrate interface, the reason for their formation is the presence of a high relief difference in the substrate in this area, formed because of sandblasting. The pores were formed because of the coating spraying process at a deviation from the normal and the formation of a "shadow", such defects, as a rule, are a special case in "problem" zones.

The coating based on the NiCrBSi-B_4C powder (Fig. 3.b) has the same lamellar structure as NiCrBSi, however, in the volume of the coating, particles of boron carbide with a dense interface between their surface and the lamellas of the self-fluxing alloy are clearly visible with a dense interface between their surface and the plates of the self-fluxing alloy. A significant difference between the obtained coatings is the difference in the B_4C content, because of the additive and its morphology in the coating. This can be verified by paying attention to the lamellar boundaries, where the coatings with the additive have a clearly expressed particle of the added powder with the original structure. While in the coating without the additive, boron is present in the form of a solid solution formed because of melting the finely dispersed sphere of NiCrBSi-B_4C powder. The data obtained from the scratch tests on the substrate/coating cross-section is presented in Table 3.

Table 3. Results of scratch test for the coatings.

Powder type	Load, N	L_x		$L_{x\text{-cp}}$	L_y	Acn
NiCrBSi	10	166.6	167.2	166.9	217., 6	0.036
	13	145.1	168	156.55	239	0.037
	15	164	177	170.5	246	0.041
	20	163.2	178.7	170.95	264.6	0.045
	23	171	185.3	178.15	287.8	0.051
$NiCrBSi\text{-}B_4C$	10	152.3	151	151.65	205.5	0.031
	13	142.4	145.8	144.1	227.4	0.032
	15	157.1	168.8	162.95	230.2	0.037
	20	161.5	164.9	163.2	255.9	0.041
	23	168.8	177.9	173.35	257.5	0.044

According to the test results, a cohesive tear in the form of a cylinder was formed on all coating samples. Table 3 shows the range of loads from 10 to 23 N. The narrowing of the range of the given data is characterized by the fact that when scratching with the application of loads from 3 N to 10 N, the tearing of the coatings was not observed. The images of the metallographic optical microscope and the measurement of the cone projection area with the applied load of 23 N are shown in Fig. 4.



(a) (b)

Fig. 4. Measuring the projection area of the cone on the coating samples: (a) NiCrBSi, (b) $NiCrBSi\text{-}B_4C$.

Based on the obtained images of the scratch test results, it can be said that the cone fracture projection area for the sample with the addition of B_4C decreased over the entire comparison of the applied indicators. Fig. 5 shows a diagram of the dependence of the cone projection area on the applied load.

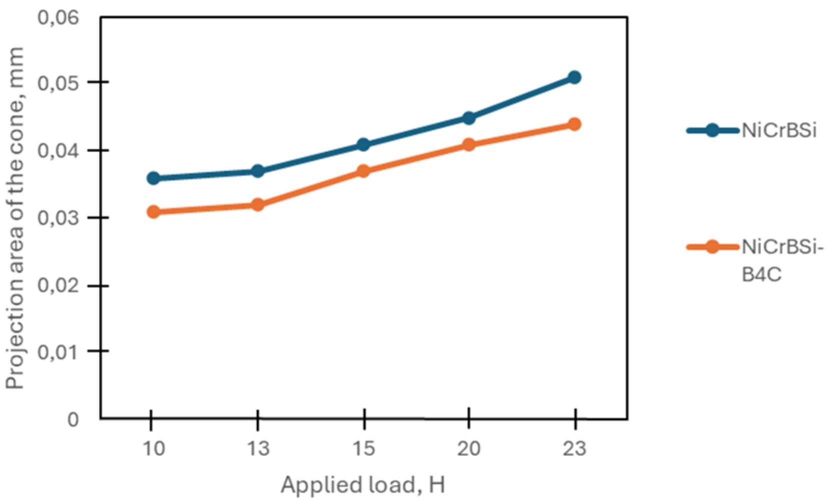
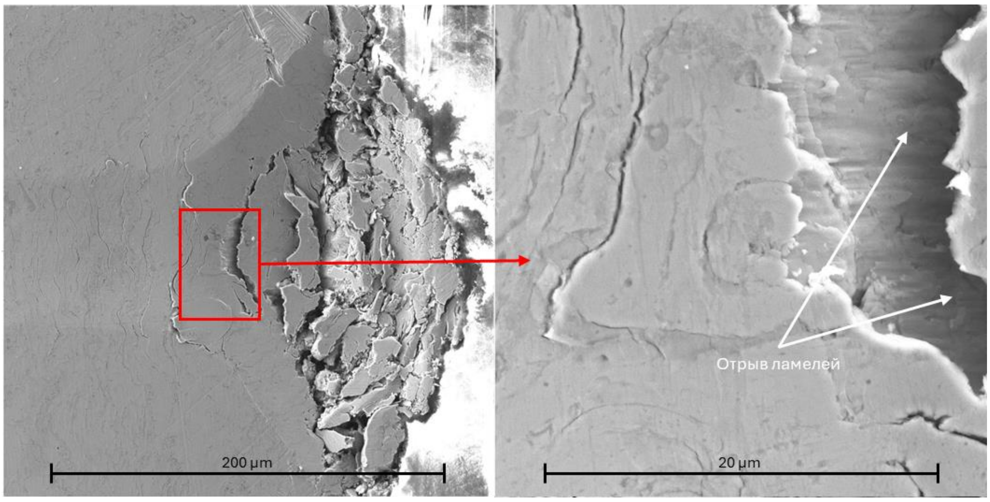


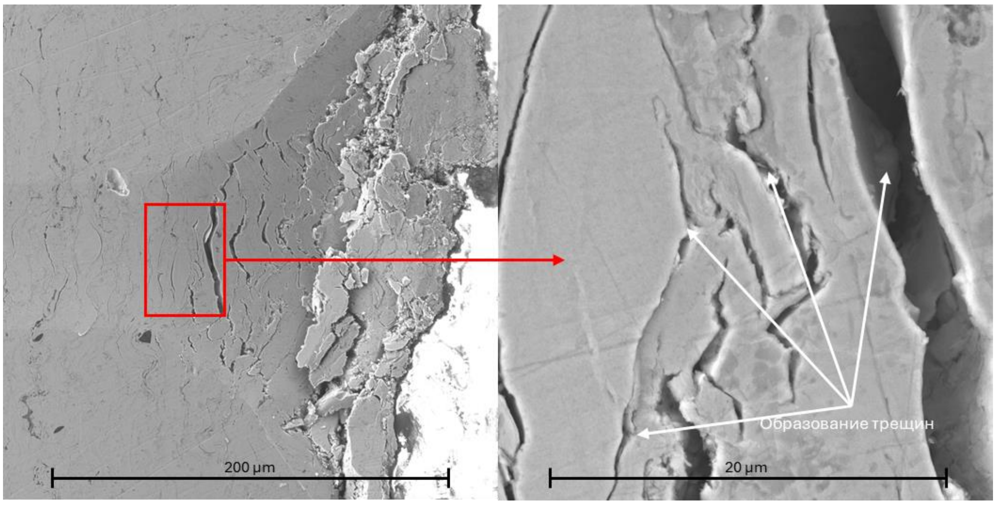
Fig. 5. Dependency diagram of the projection area of the cones versus the applied scratching load.

Comparative analysis of the cohesive properties of the obtained coatings showed that the NiCrBSi-B_4C based sample demonstrates an increase in cohesive strength by 25% for all applied loads from 10 N to 23 N. Thus, the positive effect of introducing B4C particles on the strength of cohesive bonds of self-fluxing coatings takes place.

The result of the SEM analysis of the nature of the destruction of cohesive bonds of the tested coatings is shown in Fig. 6.



(a)

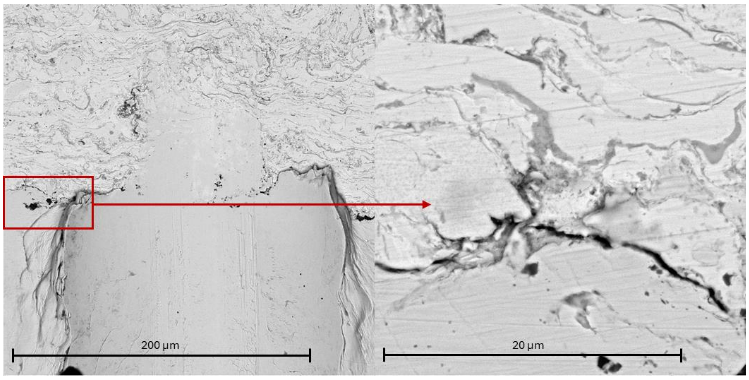


(b)

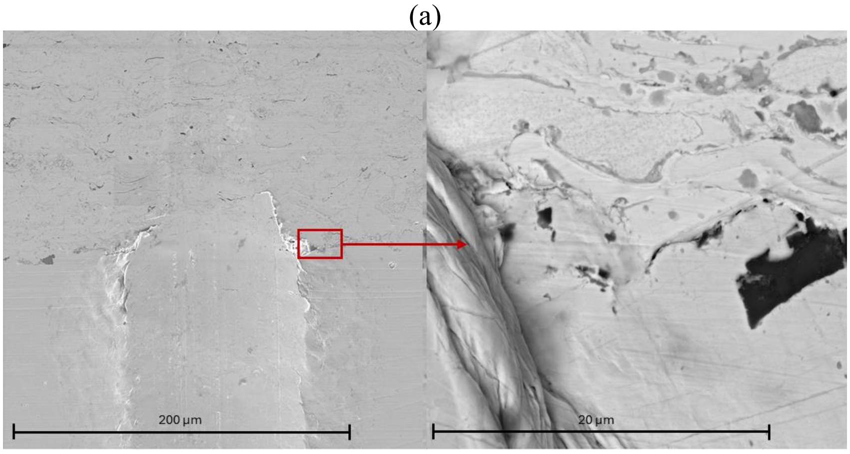
Fig. 6. The nature of cohesive destruction of the coatings under load of 23 N: (a) NiCrBSi, (b) NiCrBSi-B_4C.

SEM analysis showed that the process of destruction in both types of coatings occurs through the formation of cracks in the cohesive interlamellar bonds with subsequent chipping of the boundary areas. In the NiCrBSi coating, their separation occurs at a significantly greater depth (more than 100 µm), while the NiCrBSi-B_4C coating demonstrates greater cohesive strength and chipped areas are observed at a depth of 50-60 µm, which is clearly demonstrated in Fig. 6.

The result of the SEM analysis of the nature of the destruction of adhesive bonds is shown in Fig. 7.



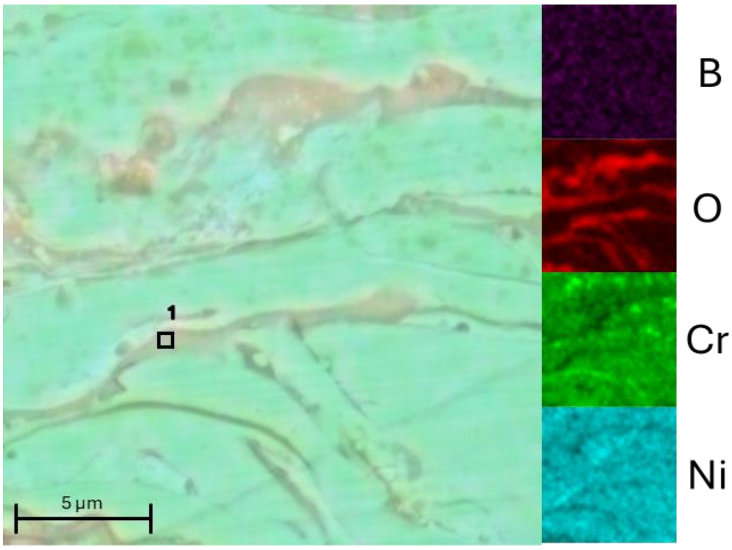
(a)



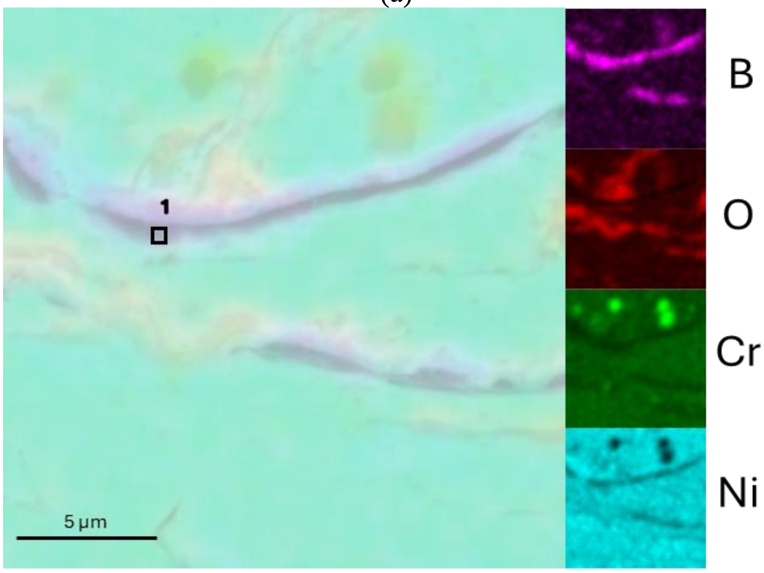
(b)

Fig. 7. Nature of adhesive destruction of coatings: (a) NiCrBSi, (b) NiCrBSi-B_4C.

Analysis of the obtained images shows that scratching a transverse section of the NiCrBSi coating (Fig. 7.a) results in the formation of a crack less than 1 μm thick in the coating/substrate interface zone. On NiCrBSi-B_4C coatings (Fig. 7.b), adhesive failure in the coating/substrate interface zone is absent under the same conditions. The phenomenon of strengthening of cohesive and adhesive bonds can be explained as follows: the introduction of up to 10% (wt.) of B_4C particles into the self-fluxing powder leads to the formation of boron-containing compounds between the lamellas, which significantly improve their strength properties and prevent the formation of brittle oxides. To prove this hypothesis, transverse sections of the obtained coatings were examined using energy-dispersive spectrometry (EDS). Fig. 8 shows maps of the distribution of chemical elements in the coating and the area of individual sections on the edge of the lamellas.



(a)



(b)

Fig. 8. Energy dispersive spectrometry of coatings: (a) NiCrBSi, (b) NiCrBSi-B_4C.

Table 4 presents the results of the EDS analysis, giving the percentage content of elements in the highlighted areas, designated as 1 for each map in Fig. 8.

Table 4. Results of EDS analysis in selected areas.

Spectrum, at. %	B	C	O	Si	Cr	Fe	Ni
NiCrBSi+10B_4C - 1	61.69	21.48	0.75	0.62	1.94	0.58	12.95
NiCrBSi - 1	0.00	21.04	29.51	1.82	10.09	2.71	34.83

According to the data in Table 4, it is evident that boron-containing compounds in cohesive bonds replace brittle oxides, increasing their strength.

4. CONCLUSIONS

The study examined wear-resistant coatings based on self-fluxing NiCrBSi alloy obtained by detonation gas-dynamic spraying method. SEM analysis of the obtained coatings showed that they have a lamellar structure with no cracks. The introduction of B_4C particles into the self-fluxing powder leads to the formation of a composite with a dense structure in cohesive bonds. Analysis of the cohesive properties by scratching showed that the adhesion strength of the coating to the substrate is higher than the adhesion strength of the coating lamellas. The introduction of B_4C particles reduces the area of coating separation during scratching over the entire range of applied loads. Improvement of the cohesive characteristics of the coating occurs due to the strengthening of cohesive bonds with boron-containing compounds. The dispersion of B_4C powder particles ranged from 3.66 to 36.1 μm. In the composition of the coating, both large particles not included in the solid solution and boron-containing compounds within the boundaries of cohesive bonds can be observed. The energy resulting from the detonation gas-dynamic spraying process is enough to melt the fine fraction of B_4C particles with the subsequent formation of boron-containing compounds. Particles of the large fraction of B_4C are partially melted, as evidenced by the density at the interface of the introduction of these particles into the composite.

Analysis of the adhesive properties showed that the introduction of B_4C strengthens the adhesive bonds of the coating with the substrate, preventing the formation of cracks and further detachment of the coating, thereby increasing its reliability.

Previously, wear-resistant coatings based on the self-fluxing NiCrBSi-B_4C alloy proved their effectiveness as protective coatings for centrifugal beet cutter knives. This study substantiates the feasibility of creating a composite based on such coatings with the introduction of boron carbide to improve its strength characteristics.

5. ACKNOWLEDGEMENTS

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