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Effect of NbN doping on forming of the structure in diamond-(Fe-Cu-Ni-Sn) system and physico-mechanical properties of this composites

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In this study the changes in the structure of the transition zone of diamond – metal bond and the metal bond in composites diamond-(Fe-Cu-Ni-Sn-NbN), after sintering in an oven at 800C for 1 hour in dependence of the parameters of the hot mounting pressing and their influence on the mechanical and tribological properties, was investigated. It is shown that an increase in pressure from 100 to 200 MPa and with a duration of the hot mounting pressing from 2 to 3 minutes, the composition diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) provides an increase of 4,3 times in the wear resistance of the samples by improving the retention of the diamond grains of the metal bond and increases the strength limits upon compression from 758 to 890 MPa and upon bending from 754 to 880 MPa. This is consistent with a change in the phase composition, the morphology and the structure of the phase components of the composite. It was found that samples of composites diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) have higher wear resistance than the composites diamond-(51Fe-32Cu-9Ni-8Sn) of about 3,8 times and 2, 4 times higher regarding the samples diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂), obtained under the same conditions. Thus, in the composite metal bond with the addition of powder NbN, grinding of the elements of the structure occurs, which is accompanied by the disappearance of the pores at the interphase boundaries. It was elucidated the mechanism, which provides increase (enhances) the tribological properties of such composites.

Keywords: Diamond, iron, copper, nickel, tin, niobium nitride, metal bond, transition zone, phase, composite, pressure, temperature, structure, properties, wear resistance.

Досліджено зміни структури перехідної зони алмаз-металева зв'язка і металевої зв'язки в композитах алмаз-(Fe-Cu-Ni-Sn-NbN) після спікання в прес-формі в печі при температурі 800 °C впродовж 1 год залежно від параметрів гарячої допресовки та досліджено їх вплив на механічні і трибологічні властивості. Показано, що збільшення тиску від 100 до 200 МПа і тривалості гарячої допресовки від 2 до 3 хв композиту алмаз-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) забезпечує підвищення в 4,3 рази його зносостійкості за рахунок поліпшення утримання алмазних зерен металевою зв'язкою та підвищення границі міцності під час стиску від 758 до 890 МПа і згинання - від 754 до 880 МПа. Це добре узгоджується зі зміною фазового складу, морфологією фазових складових та структурою композиту. Встановлено, що зразки алмаз-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) мають зносостійкість вище, ніж зразки алмаз-(52Fe-32Cu-9Ni-8Sn) в 3,8 і 2,4 рази для зразків алмаз-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂), одержаних в однакових умовах. При цьому в металевій зв'язці композиту з добавкою порошку NbN відбувається подрібнення елементів структури, яке супроводжується зникненням пор на міжфазних границях. З'ясовано механізм, який забезпечує підвищення трибологічних властивостей таких композитів.

Ключові слова: алмаз, залізо, мідь, нікель, олово, нітрид ніобію, металева зв'язка, перехідна зона, фаза, композит, тиск, температура, структура, властивості, зносостійкість.

Изучены изменения структуры переходной зоны алмаз металлическая связка и металлической связке в композитах алмаз(Fe-Cu-Ni-Sn-NbN) после спекания в пресс-форме в печи при температуре 800 C в течение 1 ч в зависимости от параметров горячей допрессовки и исследовано их влияние на механические и трибологические свойства. Показано, что увеличение давления от 100 до 200 МПа и продолжительности горячей допрессовки от 2 до 3 мин композиция алмаз(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) обеспечивает повышение в 4,3 раза износостойкости образцов за счет улучшения удерживания алмазных зерен металлической связкой и повышения пределов прочно-сти при сжатии от 758 до 890 МПа и изгибе от 754 до 880 МПа. Это хорошо согласуется с изменением фазового состава, морфологии фазовых составляющих и структуры композита. Установлено, что образцы композитов ал-маз(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) имеют износостойкость выше, чем композиты алмаз(51Fe-32Cu-9Ni-8Sn) в 3,8 и 2,4 раза для образцов алмаз(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂), полученных в одинаковых условиях. При этом в металлической связке композита с добавкой порошка NbN происходит измельчение элементов структуры, которое сопровождается исчезновением пор на межфазных границах. Выяснено механизм, который обеспечивает повышение трибологических свойств таких композитов.

Ключовые слова: алмаз, железо, медь, никель, олово, нитрид ниобия, металлическая связка, переходная зона, фаза, композит, давление, температура, структура, свойства, износостойкость.

Introduction

At the present time, intensive research works in the diamond composite material (DCM) system diamond-(Fe-Cu-Ni-Sn), which are used for the manufacture of tools for stone processing and mining industries, are conducting, regarding the nature of the physical and chemical processes occurring during the formation of the composition, their influence to the structure and the properties [1, 2]. These processes are determined the structure-phase state of the composition in all steps of sintering and the mechanical properties of the DCM [3, 4]. Such DCM should have high hardness, strength, durability (wear resistance) and reliability, should have the ability to be self-sharpening and be chemically inert to the reactants of coolant. Specific results obtained in the study, have applied to the development of new DCM with predetermined properties [5-11].

In [12, 13] was shown that directed changes in the structure and phase composition of the transition zone diamond-metallic bond of the DCM diamond-(51Fe-32Cu-9Ni-8Sn) by varying with technological parameters of the hot mounting pressing after sintering in the mold in the oven, allow a significant impact on their physical and mechanical properties. In these works, is also noted that the presence in the microstructure of a transition zone of lamellar precipitates of graphite neighboring the diamond grains, it is a major cause of premature failure by a mechanism of intense cracking and falling of diamond grains with a metal bond, as well as the deterioration of the composite. The study of the carbon (non-diamond) interaction, formed in the surface graphitization of diamond grains, with the metal bond components during sintering compositions diamond-(51Fe-32Cu-9Ni-8Sn) in the mold in an oven at 800C for 1 hour followed by hot mounting pressing, showed that the formation of Fe₃C nanostructures in the transition zone allows higher values of the metal bond regarding hardness, the strength limits under (upon) compression and bending, as well as the wear resistance of the DCM in various conditions of their practical use [14]. In [14], it was noted that an increase in pressure from 100 to 200 MPa and hot mounting pressing from 2 to 3 minutes, provides complete decarburization in the transition zone diamond-metal bond and increases 2 times the wear resistance of the DCM.

Hence, correlations between the structure of the transition zone, phase composition and the physical-mechanical properties of DCM on the one hand and their wear resistance from the other were observed.

To improve the mechanical and performance properties of DCM, in the initial materials is introducing chromium diboride (CrB₂) additives in a small amount comparing with the basic components [15-18]. Thus, a necessary condition to provide the desired structure and properties of the DCM is not only a uniform distribution of the components in the bulk metal bond, but also the

realization of the interaction of carbon, formed during graphitization of the surface diamond grains in sintering process of the composition, with the solid phases α -Fe and CrB₂ during its hot mounting pressing. Borides, nitrides, carbides and silicides of the transition metals as activating materials [19] continue to play a major role in enhancing the durability and the performance properties of heterophase composites and, in the opinion of the lead experts, they will remain on such positions for many more years [20-23]. Often when using such additives, the transition zone is contaminated by the reaction products, which generates structural instability and degrades the DCM properties [15, 16]. Laws of structure formation in the system in the presence of solid additives, such as NbN, are not currently investigated.

The aim of this work is to study the impact of the NbN additive in the composition diamond-(51Fe-32Cu-9Ni-8Sn), as well as the effect of the technological modes of the hot mounting pressing on the structural-phase state of the transition zone and to the metal bond, the mechanical and tribological properties of sintered at 800 C for 1 h in the mold in the oven composites and to identify the mechanism for the improvement of their structure, to increase the wear resistance and, as well, to compare the results with the data obtained in [14, 17, 18].

Experimental methods

The objects of the experiment were: powder mixtures 51Fe-32Cu-9Ni-8Sn and diamond-(51Fe-32Cu-9Ni-8Sn). From these, were obtained pellets at room temperature and at a pressure of 100 MPa, as well as samples of metal bond and DCM (10 mm in diameter and with thickness of 8 mm) and thin foils (with thickness 80-100 nm). To produce DCM samples, was used powders of diamond AS160T, graininess 400/315 (TU 2-37-344-85), iron PZH1M2 (GOST 9849-74), copper ICP-1 (GOST 4960-75), nickel GNU (GOST 9722-79), tin PO-1 (GOST 9723-73), chromium diboride (CrB₂) (70.62% wt. Cr and 29.30% wt. B) and niobium nitride (NbN) (86.82% wt. Nb and 13.10% wt. N), produced by the Donetsk plant of chemical reagents. The methodology in batch processing, the process parameters to obtain DCM samples and the experimental methods of their tribological properties are described in detail in [14]. To study the microstructure and properties of the DCM samples, it was taken into account the data regarding the dependence of the wear of the Cutting Diamond Segmented Circles (CDSC), 320 mm in diameter in the cutting process of the granite (Janevski deposit, Ukraine), with a thickness of 30 mm at a speed of longitudinal flow of 100 cm/min and cutting capacity of 300 cm²/min, from the content of the NbN in the batch diamond-(51Fe-32Cu-9Ni-8Sn), obtained by us (Table 1).

The work elements (40,0 mm x 12,0 mm x 3,2 mm) were sintered in the mold in a muffle furnace at 800 C for 1

Table 1
Dependence of the wear of the CDSC from the NbN content in the composition of DCM

Circle	NbN content	Specific consumption of diamonds, ct / m ² of cutting area
1	0	1,80
2	0,5	1,18
3	1,0	0,95
4	1,5	0,68
5	2,0	0,35
6	2,5	0,81
7	3,0	1,63
8	4,0	2,34
9	5,0	3,26
10	6,0	4,00

hour, followed by hot mounting pressing under pressure of 200 MPa. The most resistant to abrasion proved CDSC (5), of which the work elements contained in the composition of the initial batch 2% (wt.) NbN. This became the basis for determining the optimal composition of the batch and the study of the structure and the properties of the DCM. The microstructure of the metal bond and of the transition zone diamond-metal bond of DCM samples and the associated diffraction patterns were examined with the transmission electron microscope SELMI TEM with a potential acceleration of 125 kV at a resolution of 0,18 nm. The quantitative phase composition of the metallic bond was calculated using the package MAUD. Thin foil specimens from DCM for the study of the microstructure of the transition zone, and metal bond obtained by polishing in the electrolyte 20% HClO₄ + 30% HNO₃ + H₂O.

The diffraction patterns from the NbN powder and the sample of the metal bond with composition 49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN obtained by the diffractometer DRON 4.13S in the emission of copper anode in Bragg-Brentano geometry in the range of angles 20° ~ 2θ ~ 80°. The X-ray phase analysis was performed by standard methods using the software package X - powder [24]. The diffraction spectrum of the samples as a set of revised values of the interplanar spacing of the crystal lattice phase d_i and the relative intensities of the reflexes of this phase I_i were identified by comparing with the references from the catalogs ASTM-ICPDS [25]. The morphology of the surface of the transition zone and its chemical composition was studied in a scanning electron microscope SELMI SEM - 106M, with a potential acceleration of 20 kV. Quantitative calculations of chemical composition performed using ZAF correction method and the software package magelanes 3.1. Determination error: for heavy elements ~ 0.01% (wt.), for light elements ~ 1% (wt.).

The microhardness of the samples was determined by

the PMT- 3 device using a Vickers indenter at a load of 4,91 N. Print sizes were measured at a magnification of 25 times. The hardness was calculated by the formula $H_b = 0,4636P / x^2$, where P - load; x - indentation diagonal. The limits of the compressive strength and bending strength was investigated by the standard method (error ~ 5%). The tribological tests were performed on samples of QAM multi-functional apparatus for testing materials of friction without lubrication scheme disc (corundum counterface of medium hardness SM-2 vitrified 7K15 diameter of 60 mm and a width of 8) -palets (sample KAM), which was established on the basis of serial car IMASH- 20-75 [26]. The normal load on the sample at the disc was 500 g, the disc rotation speed was 300 rev/min and the duration of steps was: burnishing works and testing, 0,5 and 4,0 hours, respectively, which corresponds to the friction path (L) 1,7 and 13,6 km. During the tests, it was recorded the friction coefficient f , the massive wear Δm , defined by the weight loss of the samples in weighing analytical balances VLA-20g-M (accuracy ±4 mg) and the mass wear intensity $I_m = \Delta m/L$.

Results and discussion

Structure of NbN and the condition of the initial materials before and after sintering

X-ray and diffractometric studies shown, that the niobium nitride (NbN) in the initial state has a two-phase

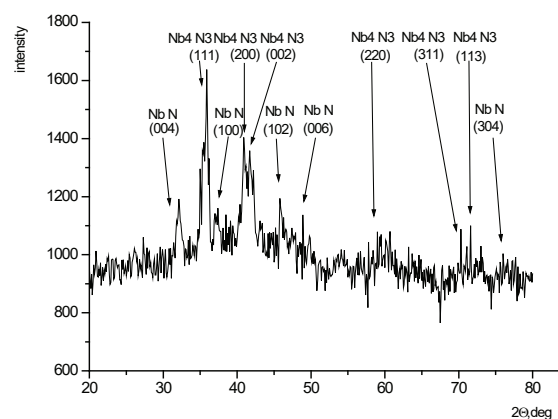


Fig. 1. The X-ray diffraction diagram of NbN in the initial state.

structure. Thus, on the X-ray diffraction diagram of the NbN powder (Fig. 1), are recorded interference reflexes from the planes, (111), (200), (002), (220), (311), (113) for phase Nb₄N₃ (tetragonal) with a crystal lattice period $a = 0,4382$ nm, $c = 0,4316$ nm, $V = 0,8288$ nm³ and (004), (100), (102), (006), (304) for the NbN phase (hexagonal) with a crystal lattice period $a = 0,2956$ nm and $c = 1,1270$ nm, $V = 0,8531$ nm³, which is consistent with the bibliography [25] and the experimental data [19].

Factual studies have established that NbN powder in the initial (bulk) state consists of irregularly shaped

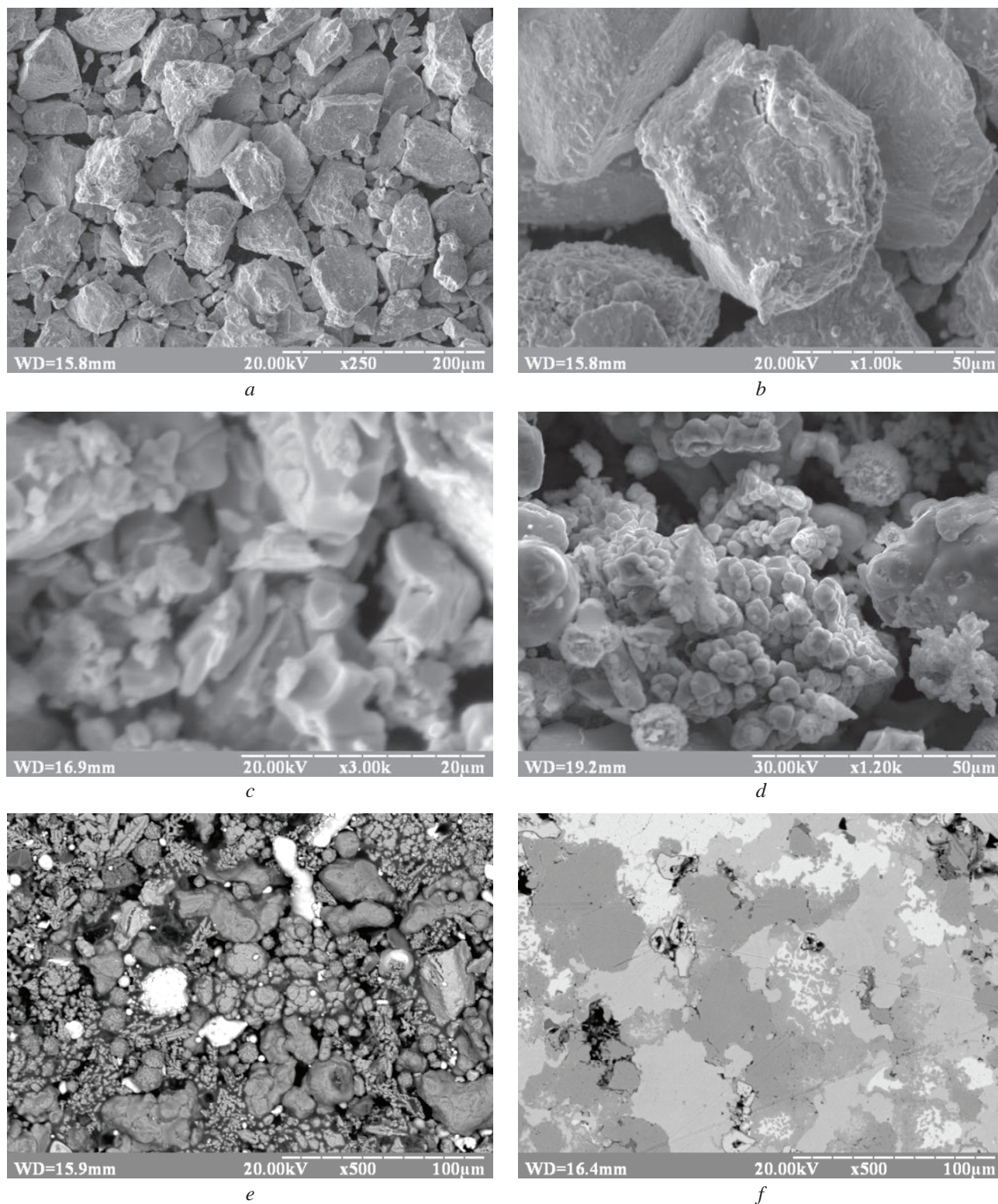


Fig. 2. SEM images of NbN particles in the initial state (a, b) and cold pressed fractogram of the NbN powder at a pressure of 100 MPa (c); photomicrographs of the initial mixture 49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN (d), the briquette, obtained from the initial mixture at a pressure of 100 MPa, (d) and bond sample (e) obtained at a temperature of 800 C for 1 hour with hot mounting pressing at a pressure of 100 MPa.

particles, the average size of which ranges between 10-30 μm (Fig. 2a), although we meet also some larger particle size (up to 50 μm) (Fig. 2b) with a developed surface. It is characteristic that particles larger than 40 μm have an irregular shape with relief with irregularities, the number

of which increases by increasing the particle size. In the particles smaller than 20-30 μm, by reducing further the size, the irregularities are smoothed out. When compacting at room temperature, the NbN particles do not change the shape, between them is observed gaps, that predetermined

by the insufficient compaction pressure (100 MPa). The strength of the compact in this case is determined by the mechanical clamping of particles (Fig. 2c). The shape and the dimensions of powders of iron, copper, nickel, tin, and niobium nitride NbN to the resulting mixture, in comparison with separate particles in the initial state, has not changed (Fig. 2d). During briquetting the mixture, i.e. in the process of cold compaction at a pressure of 100 MPa, the shape of particles of iron, copper, nickel and niobium nitride does not change (Fig. 2e), although, compared with the initial state, they are compacted more strongly (Fig. 2a, b, d). However, in the process of pressing the mixture, all particles are deformed due to their contact each other. Thus, there is a relatively uniform distribution of all components.

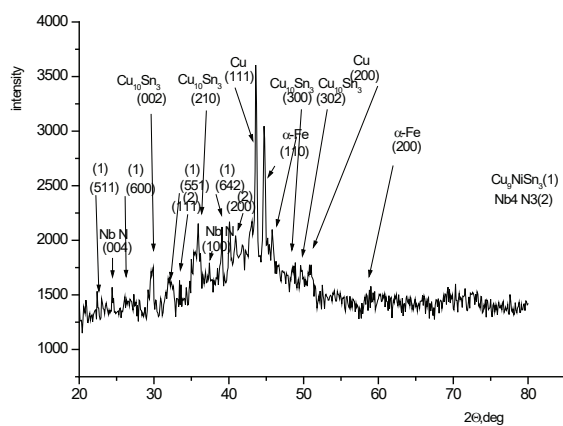


Fig. 3. The XRD diagram of the surface of the sample with a metallic bond of composition 49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN, obtained by sintering in the mold in an oven at 800 C for 1 hour with hot mounting pressing at a pressure of 160 MPa for 3 min.

During sintering the briquette, the compaction of the bond components occurs by adjusting the surfaces of adjacent particles to one another, which contributes to a compact contact at the interphase boundaries in the composite bunch (Fig. 2f). In addition, the sintered metal bond sample is characterized by a homogeneous structure throughout the volume, which is an advantage and points to prospects for its use in DCM technology. All this affects the structure and properties of the DCM.

Table 2.

The diffraction data for the metal bond sample

d_i nm	2θ , deg	I_i	Phase	hkl
0,33594	26,5333	464,12	Cu ₉ NiSn ₃	511
0,3284	29,5934	574,53	NbN	004
0,30192	29,5879	546,64	Cu ₉ NiSn ₃	600
0,25074	35,8172	561,99	Cu ₁₀ Sn ₃	002
0,23046	39,0861	579,46	Nb ₄ N ₃	111
0,22482	40,1091	658,25	Cu ₉ NiSn ₃	511
0,22465	40,1409	588,10	Cu ₁₀ Sn ₃	210
0,22131	40,7733	533,36	NbN	100
0,22036	40,9576	522,45	Cu ₉ NiSn ₃	642
0,21090	42,8822	579,03	Nb ₄ N ₃	200
0,20727	43,6727	949,84	Cu	111
0,20278	44,6909	901,66	α-Fe	110
0,19798	45,8348	571,58	Cu ₁₀ Sn ₃	300
0,18793	48,5764	476,25	Cu ₁₀ Sn ₃	302
0,18375	49,6121	543,15	Cu	200
0,18001	50,7152	529,04	α-Fe	200

Fig. 3 shows a diffractogram of the metallic bond sample 49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN, obtained by sintering in the mold in an oven at 800 C for 1 hour, followed by hot mounting pressing at a pressure of 160 MPa for 3 min. The interference maxima on the X-ray XRD from the (111) and (200) for phase for copper (lattice parameter $a = 0,3608$ nm); (110) and (200) for iron ($a = 0,2863$ nm); (002) (210) (300) (302); (211) Cu₁₀Sn₃; (511) (600) (551) (642) Cu₉NiSn₃ ($a = 1,801$ nm); (111) (200) and (220) Nb₄N₃ ($a = 0,4382$ nm) and (004), (100) NbN ($a = 0,2956$ nm, $c = 1,1274$ nm) are consistent with [25] and the corresponding parameters for copper, iron, and niobium nitride [19]. Diffraction data (interplanar spacing, relative intensity, the angles 2θ , phase and lattice indices (hkl), corresponding to the interference maxima), are shown in Table 2. Crystal structure and quantitative phase composition of the bond sample are shown in Table 3. Analysis of the results showed that during the formation of the metal bond sample occurs elements' interaction that forms solid solutions on the basis of copper and iron

Table 3.

The crystal structure and phase composition of the metal bond of DCM

Phase	PG	Lattice parameter, nm			Concentration phase, % (wt.)	
		A	ϵ	C	SEM	XRD
Cu	$Fm3m$	0,3608	–	–	41,8	44,2 ± 2,3
α-Fe	$Im3m$	0,2857	–	–	38,5	34 ± 1,8
Cu ₉ NiSn ₃	$F-43m$	1,8010	–	–	19,7	12,1 ± 1,9
Cu ₁₀ Sn ₃	$hP26$	–	–	–	–	7,1 ± 1,3
NbN	$C6/m2$	0,2952	–	1,1271	–	2,2 ± 0,9
Nb ₄ N ₃	–	0,4381	–	0,4312	–	0,4 ± 0,7

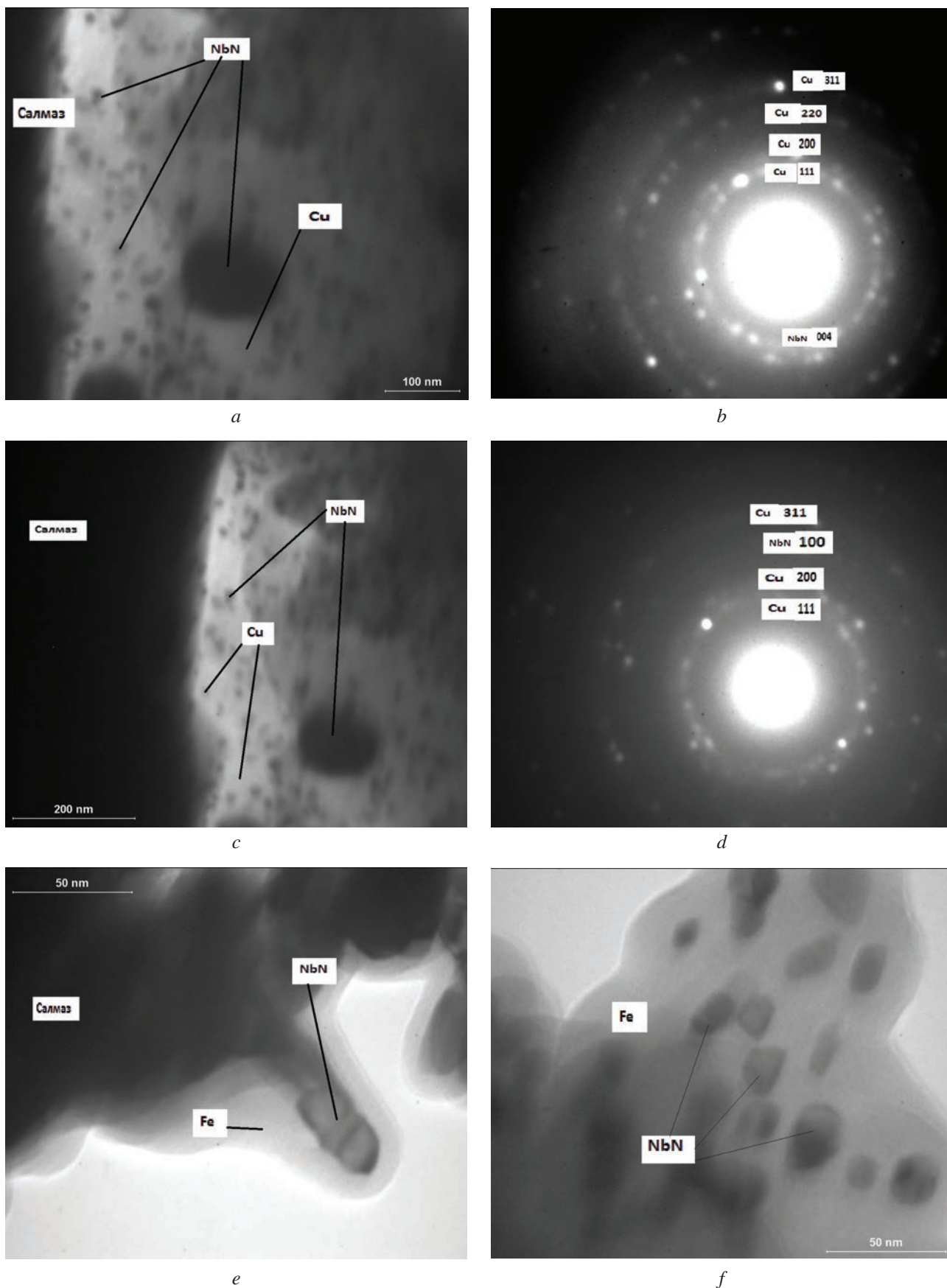


Fig. 4. TEM images of the sections of the transition zone diamond-metal bond of the samples 1 (a), 2 (b), 3 (e), 4 (f) and microelectronographs of its fragments for samples 1 (b), 2 (d): (a, b) - $p = 100$ MPa, $t = 2$ min; (c, d) - $p = 100$ MPa, $t = 3$ min; (e) - $p = 200$ MPa, $t = 2$ min, (f) - $p = 200$ MPa, $t = 3$ min.

(main phases), which is confirmed by the change in lattice parameters compared to the pure elements for copper ($a = 0,3615 \text{ nm}$) and iron ($a = 0,2864 \text{ nm}$), as well as the formation of new phases - Cu_9NiSn_3 and $\text{Cu}_{10}\text{Sn}_3$.

Comparison of the results with the data of [14] shows that the introduction of NbN in the amount of 2% (wt.) in the composition of the metal bond sample 51Fe-32Cu-9Ni-8Sn affects the phase formation in the process of its formation. Furthermore, the content of the sintered sample of the formed phases Cu_9NiSn_3 and $\text{Cu}_{10}\text{Sn}_3$ is respectively 12.1% (wt.) and 7.1% (wt.), which can affect the mechanical and tribological properties of the DCM.

The structure of transition zone diamond-metal bond of the sintered samples DCM

The results of the structural studies of the transition zone diamond-metal bond of DCM samples obtained from the batch diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN), sintering in the mold in an oven at 800 C for 1 hour, in dependence from p - t parameters of the hot mounting pressing, is shown in Fig. 4. The microstructure of the transition zone diamond-metal bond of obtained DCM samples (Fig. 4, a, c, e, f) is radically different from the above described microstructure of sintered samples of the metal bond. The structure of the transition zone diamond-metal bond of the sample 1, obtained at a pressure of 100 MPa, under hot mounting pressing for 2 minutes, is composed of copper phase and niobium nitride, which are uniformly distributed in the absence of graphitic inclusions (Fig. 4a). The grain size in the transition zone does not exceed 100 nm. Furthermore, niobium nitride NbN is present as inclusions in spherical shape, average particle size is from 10 to 100 nm, which is considerably smaller than the niobium nitride in the initial state (10-50 μm) (see Fig. 2a, b). As a result, the microstructure of the transition zone of the sintered DCM sample includes both nano-sized crystals of copper separated by almost invisible boundaries and nanoscale inclusions of niobium nitride. In this case, the contact diamond-metal bond is very tight; the boundaries between the grains are thin, clearly formed, with no visible pores and cracks, which has a positive effect on the performance properties of the DCM.

At the roundabout prints of the mikroelektronographs of this sample are clearly visible the interference reflexes for the phases of Cu and NbN (Fig. 4b). Copper phase grains are textured in the directions (311), (220), (200) and (111) and niobium nitride phase grains in the direction (004). Expansion of the diffraction peaks on circular (ring) imprints indicating a low degree of crystallinity of these phases. By increasing the duration of the hot mounting pressing from 2 to 3 minutes, the structure of the transition zone remains practically unchanged (sample 2) (Fig. 4c). In this case, the copper phases grains textured in the directions (311), (200) and (111) and niobium nitride phase grains in the direction (100). Expansion of the diffraction peaks in the ring imprints on mikroelektronographs of this sample once more confirms the low degree of crystallinity of the phases of Cu and NbN (Fig. 4d). Diffraction data (interplanar spacing and phase identification codes of the crystal lattice (hkl)) for samples 1 and 2 are shown in Tables 4 and 5. The calculated values d_i for the phases are coincide with the data in [18], indicating the reliability of the results. Thus, our experimental data on the structural state of the transition zone of the investigated composites and their comparison with previous experimental data [5-7, 9-11] suggest that the use of the NbN in the composition diamond-(51Fe-32Cu-9Ni-8Sn) completely eliminates the phase transformation diamond \rightarrow graphite at the sintering stage, which is very important to improve the structure and the working (exploitation) properties of such DCM

It was revealed that when the pressure of hot mounting pressing increases from 100 to 200 MPa in samples 3 and 4 is formed a solid nanograin structure of the transition zone with uniformly distributed phase α -Fe and NbN (Fig. 4, e, f). A characteristic feature of these results is the absence of both the graphite inclusions in the transition zone and the formation of carbide phases, which reduces its brittleness and, as a consequence, is very important to improve the structure and improve service properties such DCM.

Below are the studies that were carried out regarding the effect of the structure of the transition zone of the obtained DCM samples to their physical, mechanical and tribological properties.

Peculiar properties of the transitional zone diamond-

Table 4

The diffraction data of the transition zone for the sample DCM 1

d_i (calculation), nm	d_i (experiment), nm	Phase	hkl
0,2570	0,2580	NbN	400
0,2079	0,2080	Cu	111
0,1811	0,1808	Cu	200
0,1280	0,1270	Cu	220
0,1090	0,1090	Cu	311

Table 5

The diffraction data of the transition zone for the sample DCM 2

d_i (calculation), nm	d_i (experiment), nm	Phase	hkl
0,2079	0,2080	Cu	111
0,1810	0,1808	Cu	200
0,1661	0,1470	NbN	110
0,1092	0,0900	Cu	311

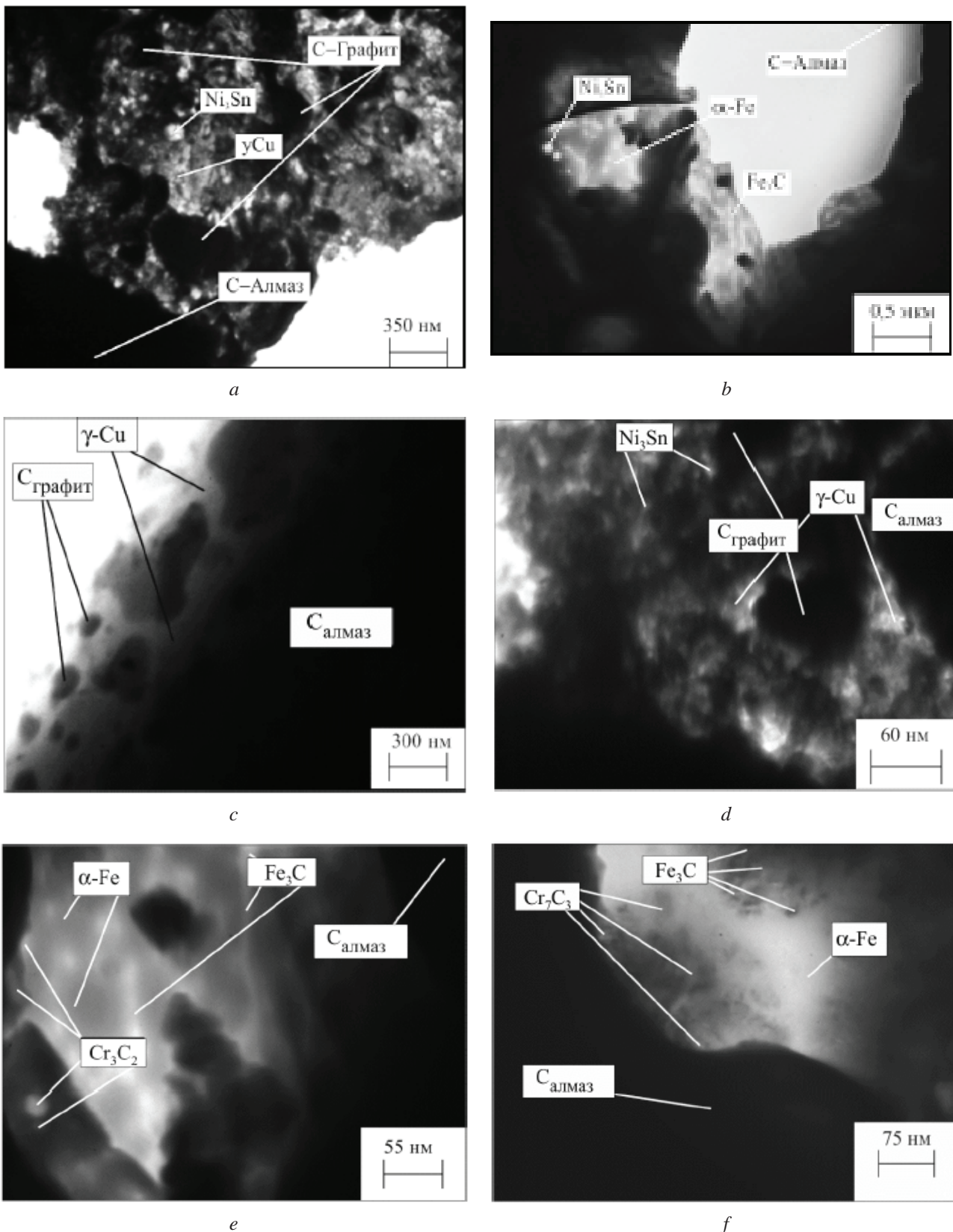


Fig. 5. TEM images of sections of the transition zone diamond-bond samples 5 (a), 8 (b), 9 (c), 10 (d), 11 (e), 12 (f), obtained by sintering in a mold in an oven at 800 C for 1 hour, followed by hot mounting pressing at: a) $p = 100$ MPa, $t = 2$ min; b) $p = 100$ MPa, $t = 3$ min; c) $p = 100$ MPa, $t = 2$ min; d) $p = 100$ MPa, $t = 3$ min; e) $p = 200$ MPa, $t = 2$ min; f) $p = 200$ MPa, $t = 3$ min.

metal bond in composites: diamond-(Fe-Cu-Ni-Sn-NbN) (samples 1-4), diamond-(Fe-Cu-Ni-Sn) (samples 5-8), diamond-(Fe-Cu-Ni-Sn-CrB2) (samples 9-12) and their impact on the tribological properties.

The comparison of the peculiar properties of the structure of the investigated samples diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) (samples of the first type) and their analogues, diamond-(51Fe-32Cu-9Ni-8Sn) [7] (samples of the second type) and diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB2) (samples of the third type) [10, 11], obtained by sintering in the mold in an oven at a temperature of 800 C for 1 hour, followed by hot mounting pressing, shows that exist common identities as well as fundamental differences.

In particular, it is shown that the main phases of the metallic bond of DCM samples of the first, second and third types, obtained under identical conditions, are solid solutions based on iron and copper intermetallics Cu₉NiSn₃. It should be noted that the other phases in the metal bond samples of the first type are Cu₁₀Sn₃, NbN and Nb₄N₃, and Ni₃Sn and Ni₃Sn, CrB₂ in second and third type, respectively. This means that the formation of phases in the system depends on the composition of the initial materials.

A fundamental difference of the structure of the DCM samples is the construction of the transition zone diamond-metal bond. Thus, the structure of the transition zone diamond-metal bond of the samples of the first type in dependence on the parameters of the hot mounting pressing, is consisting of phases Cu, NbN (see Fig. 4 a-d) and (or) α -Fe and NbN (see Fig. 4e,f) with complete absence of graphitic inclusions, cracks and micropores. Whereas, the structure of the transition zone diamond-metal bond of DCM samples of the second [14] and third [17, 18] types, differs substantially from the structure of the metallic bond, because to its formation affects the interaction of carbon which is obtained by the surface graphitization of diamond grains at the stage of sintering, with the solid phases. Thus, the structure of the transition zone of the sample 5, manufactured from the batch diamond-(51Fe-32Cu-9Ni-

8Sn) at a pressure of 100 MPa and a holding time under pressure for 2 minutes, consisting of Cu, Ni₃Sn phases with graphite inclusions (Fig. 5a). This is the reason for its destruction and premature loss of diamond grains with a metal bond, which reduces the wear resistance of DCM.

A distinctive feature of the structure of the transition zone in the sample 8, obtained from the same batch, at a pressure of 200 MPa, is the presence of Fe₃C nanoscale layers and no graphite inclusions (Fig. 5b). This effect is due to the fact that the carbon which is formed in the surface graphitization of diamond grains at the stage of sintering the composition interacted with the solid phase α -Fe during its hot mounting pressing, by forming iron carbides. This in turn, provides the decarburization in the transition zone of the DCM sample, which can prevent premature failure of the transition zone and deposition (falling out) of diamond grains from the metal bond of the composite.

The transmission electron microscopy (TEM) data (presented in Fig. 5c-f), shows that the structure of the transition zone diamond-metal bond of DCM samples of third type (samples 9-12) also depends on technological parameters of the hot mounting pressing. Thus, the structure of the transition zone in the DCM samples (samples 9 and 10) obtained under insufficient pressure values (lower than 200 MPa) and with duration of the hot mounting pressing less than 3 minutes, is consisting of Cu, Ni₃Sn phases with graphite inclusions (Fig. 5c, d), which serves the cause of its premature failure of the mechanism of intensive cracking and loss of diamond grains from the bond of the composite.

Whereas, the structure of the transition zone diamond-metal bond of DCM samples (samples 11 and 12) obtained under insufficient pressure values (lower than 200 MPa), consists of layers of carbides Fe₃C, Cr₃C₂ and Sr₇C₃ with a nanometer thickness (Fig. 5e, f). Thus, in the transition zone of these samples there are no graphite inclusions, micro-cracks and pores. The mechanism of formation of

Table 6

The mechanical properties of the DCM samples

Sample	Pressure p , MPa	Duration of hot mounting pressing t , min	Microhardness H_v , GPa			Strength limits upon compressive R_{cm} , MPa	Strength limits upon bending R_{bm} , MPa
			Cu	Ni ₃ Sn	α -Fe		
1	100	2	2,82	–	3,67	758	754
2		3	2,84	–	3,80	790	781
3		2	2,95	–	4,20	856	830
4	200	3	3,35	–	4,40	890	880
5		2	2,50	2,63	2,93	710	610
6		3	2,61	2,67	3,16	720	625
7	200	2	2,70	3,05	3,42	800	635
8		3	2,79	3,33	3,74	826	650
9		2	2,60	3,76	2,93	730	640
10	100	3	2,70	2,82	3,46	750	645
11		2	2,80	3,03	3,96	780	655
12		3	2,92	3,48	4,12	840	675

these layers is explained due to the fact that carbon obtained by graphitizing the surface layer of diamond grains in the sintering compositions interacted with α -Fe and CrB₂ under hot mounting pressing, forming iron and chromium carbides.

Thus, as a result of the conducted research, it was found that several factors affect to the formation of nanostructures in the transition zone diamond-metal bond sintered in mold in the oven at 800 C for 1 h of the composites diamond-(51Fe-32Cu-9Ni-8Sn). The main factors are: The composition of DCM, including the existence of hardening additives niobium nitride and chromium diboride, pressure and the duration of hot mounting pressing. It should be noted some advantages of this method for producing DCM compared with other methods. Firstly, the metal components of the composite in the initial state have sizes ranging ~ 5–50 μ m, i.e. in our case the burnishing of powder mixtures do not require the use of nanoparticles in their composition, that reduces the possibility of oxidation of the initial materials in the process of batch burnishing at one hand, and on the other, it significantly reduces the cost of DCM.

Secondly, in the case of samples of the second and third type, that is, when occurs phase transformation diamond \rightarrow graphite, carbon, which is formed in the surface graphitization of diamond grains under sintering the composition, ceases natural sources in improving mechanical and tribological properties of the DCM. Thirdly, the correct choice of introducing additives and technological parameters of hot mounting pressing is the key for obtaining DCM with useful new properties. Furthermore, during the formation of the DCM samples, occurs significant milling of the structural elements of the transition zone diamond-metal bond. To confirm these conclusions, studies regarding mechanical and tribological properties of the DCM, was carried out.

The results of determination of microhardness (Hv),

strength limits upon compressive (Rcm) and upon bending (Rbm), of sintered at 800 C with a duration of 1 h in the mold in an oven followed by hot mounting pressing composites diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) (samples of the first type) and their analogues, diamond-(51Fe-32Cu-9Ni-8Sn) (the samples of the second type) [14] and diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂) (examples of the third type) [17, 18], sintered in the same conditions as the samples of the first type, are shown in Table. 6. Analysis of the data showed, that the mechanical properties of the DCM samples depending from both, composition and technological parameters of their hot mounting pressing. Thus, by increasing pressure of hot mounting pressing of the composition diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) (samples of the first type) from 100 to 200 MPa and the duration of the process from 2 to 3 minutes, is observed an increase in microhardness in the sections of the metal connection for the phase Cu from 2,82-2,84 GPa (samples 1 and 2) to 2,95-3,35 GPa (samples 3 and 4) and for the phase α -Fe from 2,67-3,80 GPa (samples 1 and 2) to 4,20-4,40 GPa (samples 3 and 4). This increases the value of strength limits upon compressive from 758 to 890 MPa and upon bending from 754 to 880 MPa. The mechanism of this improvement is likely consisting of the compacting of the structural components of the composite and, as a consequence, of the disappearance of pores at the interfaces boundaries, as well as in the boundary of the section diamond-metal bond.

A similar tendency in the dependencies on the mechanical characteristics and the duration of hot mounting pressing is preserved for the DCM samples of second type (samples 5-8), but their mechanical properties have smaller values than the samples of the first type. When administered chromium diboride to the composition diamond-(51Fe-32Cu-9Ni-8Sn), is observed a slight increase in strength properties (samples 9-12) in comparison to the samples

Table 7

The tribological properties of the DCM samples

Sample	Coefficient of friction, <i>f</i>		Massive wear Δm , g		Wear intensity I_m , g/Km	
	Burnishing	Testing	burnishing	Testing	burnishing	Testing
1	0,207	0,092	0,040	0,106	0,0236	0,0078
2	0,211	0,096	0,039	0,095	0,0229	0,0070
3	0,220	0,105	0,018	0,027	0,0106	0,0020
4	0,223	0,108	0,017	0,025	0,0101	0,0018
5	0,203	0,088	0,089	0,184	0,0518	0,0135
6	0,206	0,089	0,080	0,168	0,0471	0,0124
7	0,211	0,094	0,052	0,111	0,0306	0,0082
8	0,216	0,098	0,041	0,094	0,0241	0,0069
9	0,209	0,096	0,083	0,170	0,0488	0,0125
10	0,209	0,108	0,080	0,160	0,0470	0,0118
11	0,227	0,112	0,038	0,074	0,0223	0,0054
12	0,229	0,116	0,032	0,060	0,0188	0,0044

5-8.

The results of the tribological tests of the obtained DCM samples (coefficient of friction f , massive wear Δm , wear intensity I_m) paired with an abrasive circle of electrocorundum are given in Table 7. The analysis showed that the tribological properties of the DCM samples, as well as the mechanical properties, are depending from the composition of the initial materials and the technological parameters of the hot mounting pressing. We note some differences in the nature of changes in the tribological properties of the DCM samples in dependence from their composition and technological parameters of hot mounting pressing and the duration of the experiment. It can be seen that after a period of burnishing (friction path 1,7 km), all DCM samples (samples 1-12) exhibit a relatively high coefficient of friction f (0,207-0,223), compared with values of the coefficient of friction f (0,092-0,116) after the testing period (friction path 13,6 km). This is due to the fact that the step of the burnishing of DCM characterized with an increased wear due to intense friction with the metal bond with the abrasive circle, since the diamond grains have no enough time to be exposed to a sufficient amount.

It was revealed that the tribological characteristics of the DCM samples after testing in two stages are depending on the parameters of hot mounting pressing. Thus, when pressure is increased from 100 to 200 MPa and a holding time under pressure from 2 to 3 minutes, there is observed some increase in the coefficient of friction f , from 0,092 to 0,108 and the reducing of the mass wear Δm from 0,040 to 0,017 g, as well as (is observed) the reducing of the I_m from 0,0236 to 0,0101 g/km, for the samples of the first type after the burnishing period (friction path 1,7 km) (Table 7, samples 1 and 4). These data are in good agreement with the classical theory of wear, according to which the wear intensity of DCM naturally decreases by increasing the hardness of the metal bond. However, according to our data from TEM (Fig. 4) results that the main reason for increasing the wear resistance and strength properties of the samples of the first type is the distinction in the structure of the transition zone diamond-metal bond. Thus, from Fig. 4a, b could be clearly seen that the structure of the transition zone of the sample 1 is composed of Cu and NbN phases, and the structure of the transition zone of the sample 4, of α -Fe and NbN phases (Fig. 4 f). In this case we observed increase in the strength limits upon compressive R_{cm} from 758 to 890 MPa and upon bending R_{bm} from 754 to 880 MPa (tab. 6, samples 1 and 4) and the increase of the wear resistance of the composite is 2,3 times (Table 7, samples 1 and 4). This in turn provides an improved retention of the diamond grains and the metal bond and, as a consequence, increases the wear resistance of the DCM. With increasing friction paths from 1,7 to 13,6 km, it is observed a decrease in the coefficient of friction f and in the wear intensity I_m , at the testing, for the DCM samples.

This process is most notably expressed at the sample 4 for which the coefficient of friction f , the mass wear Δm and the wear intensity I_m are reaching the values 0,088, 0,025 and 0,0018, respectively. These data, once more, show that the structural phase state of the transition zone diamond-metal bond is the main reason for the increase of the wear resistance of the DCM.

The schematic picture of wear of the DCM 5-8 samples (samples of the second type) under friction, implemented in our experiments, is not changed comparing to DCM 1-4 samples (samples of the first type). However, the deterioration (wear) of these DCM is developing more intensively. Thus, by increasing the pressure of the hot mounting pressing of the composition diamond-(51Fe-32Cu-9Ni-8Sn) from 100 to 200 MPa and the duration of the process from 2 to 3 minutes, is observed a decrease of mass wear Δm and in wear intensity I_m for the burnishing stage, from 0,089 to 0,041 g and from 0,0518 to 0,00241 g/km respectively (Table 7, samples 5 and 8). With increasing friction path from 1,7 to 13,6 km it was also observed a decrease in the values of the mass wear Δm and the wear intensity I_m from 0,184 to 0,094 g, and from 0,0135 to 0,0069 g/km, respectively. Such a process of deterioration is also related mainly to the structure of the transition zone diamond-metal bond. Analysis of Fig. 5b shows that the structure of the transition zone of sample 8 obtained at a pressure of 200 MPa, consist of the phases α -Fe and layers Fe₃C of nanoscale thickness in the absence of graphitic inclusions. Whereas the structure of the transition zone of the samples 5 and 6 are obtained at a lower pressure of hot mounting pressing (100 MPa), consists of the phases Cu and Ni₃Sn with graphite inclusions (Fig. 5a), which is the reason for its premature failure of the mechanism of intensive cracking and deposition of diamond grains from the metal bond.

For the samples of third type (samples 9-12) effect of reducing the mass wear Δm and the wear intensity I_m when the pressure of the hot mounting pressing increases, is expressed more strongly in comparison with the samples of the second type (samples 5-8). Thus, by increasing the pressure of the hot mounting pressing from 100 to 200 MPa and the holding time under pressure from 2 to 3 min, is observed a decrease of the wear mass Δm and of the wear intensity I_m for DCM samples after their testing stage at burnishing step from 0,083 to 0,032 g and from 0,0488 to 0,0188 g/km, respectively (Table 7, samples 9 and 12). With increasing friction path from 1,7 to 13,6 km, the values of the wear mass Δm and of the wear intensity I_m decreases from 0,170 to 0,060 g and 0,0125 to 0,0044 g/km, respectively (Table 7, samples 9 and 12). The effect of improving the wear resistance of the sample 12 as compared with sample 9, is explained due to the presence of nanostructure Fe₃C, Cr₃C₂ and Sr₇C₃ without graphite inclusions in the first (Fig. 5e, f), which is providing the increase of the strength

limits upon compressive (Rcm) from 730 to 840 MPa and upon bending (Rbm) from 640 to 675 MPa. Whereas the structure of the transition zone of samples 9 and 10 consists of phases Cu, Ni₃Sn with graphite inclusions (Fig. 5c, d), which reduces the mechanical properties of such DCM (Table 7). The mechanism of formation of the nanostructure in the transition zone of DCM samples 11 and 12, is explained due to the fact that the carbon which is formed during the surface graphitization of the diamond grains at the stage of sintering of the composition, is interacted with the solid phase α -Fe and CrB₂ during its hot mounting pressing, forming carbides of iron and chromium. This in turn, leads to decarburization of the transition zone and improves the mechanical properties of DCM.

Thus, the analysis of the data show that the introduction of additives CrB₂ into the composite diamond-(51Fe-32Cu-9Ni-8Sn) leads to lower wear intensity I_m of DCM from 0,0069 to 0,0044 g/km (Table 9, samples 8 and 12, respectively), which provides an increase of the wear resistance by 1,6 times. Even more pronounced effect of enhancing the wear resistance (by 2,6 times) is observed when we use NbN additives, which is associated with the formation of two-phase structure of the transition zone (α -Fe and NbN phases) with reduced fragility parameters and more milled grains, as well as the formation of a metal bond structure with increased strength parameters. The observed important effect of the formation of a special structure of the transition zone in the DCM system diamond-(Fe-Cu-Ni-Sn), points out the necessity to continue these studies, in order to establish the generality of this effect for DCM of other systems, to obtain direct evidence of the effect of additives NbN, CrB₂ and (or) other additives of transition metals on the formation of the structure, with which are mainly related physical-mechanical and operational properties of the DCM. It should be noted that is observed correlation between the structure of the transition zone diamond-metal bond and the wear resistance of DCM [7].

Conclusions

In this work, the changes in the structure of the transition zone diamond-metal bond and metal bond in the compositions diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN), diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂) and diamond-(51Fe-32Cu-9Ni-8Sn) after sintering in the mold in the oven at 800 C for 1 hour, in dependence from the p-t parameters of hot mounting pressing and their influence on physical -mechanical and tribological properties of sintered composites was investigated.

1. It was found that the metal bond of the composite diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) regardless of the technological parameters of hot mounting pressing, consists of solid solutions based on iron and copper of variable composition and phases Cu₁₀Sn₃; Cu₉NiSn₃, Nb₄N₃, NbN, and the transition zone diamond-

metal bond in dependence from the hot mounting pressing may consist of phases Cu; NbN and (or) α -Fe; NbN, as well as of the combination of these phases.

2. It was established that by increasing the pressure from 100 to 200 MPa and the hot mounting pressing from 2 to 3 minutes of the composition diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) after sintering in the mold in an oven at 800 C for 1 hour, is providing a 4,3 times increase of the wear resistance of the composites due to the increase of the microhardness of the bond sections for the phases Cu from 2,85 to 3,35 GPa, for the α -Fe from 3,67 to 4,40 GPa, for the strength limit upon compressive from 758 to 890 MPa and for the strength limit upon bending, from 754 to 880 MPa. This is in agreement with the change in the phase composition, the morphology of the phase components and the structure of the composite.

3. It is shown that composite sample diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN) have abrasive wear resistance higher than composites diamond-(51Fe-32Cu-9Ni-8Sn) 3,8 and 2,4 times more than the samples diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2CrB₂), obtained in the same conditions. Herewith in the composite of metal bond occurs milling of the elements of the structure, which is accompanied by the disappearance of the pores at the interphase boundaries.

4. Different character of the wear of the studied DCM is explained due to the fact that the interaction of the elements in dependence from the type of additive in the process of the formation of the composition takes place in different ways and has different effects on the structure and properties:

- The increase of the wear resistance of DCM samples of second and third types is due to the formation of the transition zone diamond-metal bond of nanostructure respectively from Fe₃C and Fe₃C, Cr₃C₂ and Sr₇C₃, as the result of the interaction of the carbon, which is released at the surface graphitization of diamond grains in the sintering phase of the composition with solid phases α -Fe and CrB₂ during the hot mounting pressing, which helps to improve the adhesion properties at the boundary of the diamond-bond section.

- The increase of the wear resistance of DCM samples of the first type (samples diamond-(49,98Fe-31,36Cu-8,82Ni-7,84Sn-2NbN)) is explained by the formation of a longer lasting (stronger) contact on the boundary diamond-metal bond section by the hard compression of the diamond grains by α -Fe and NbN particles whereas milling the elements of the structure to nanoscale sizes. The mechanism of this process has not yet been studied, which is of interest for further research.

Legend (Notation)

P, is pressure of the hot mounting pressing; t, is the duration of the hot mounting pressing; a, b, c are the

lattice parameters; (hkl) , is lattice indices; d_i , is interplanar distance; θ , is the diffraction angle; I_i , is the relative intensity of reflexes; H_v , is microhardness; R_{cm} , is the strength limit upon compressive; R_{bm} , is strength limit upon bending; f , is the coefficient of friction; L , is the friction path; Δm , is the mass wear; I_m , is the wear intensity

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