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Peculiarities of obtaining diamond-(Fe–Cu–Ni–Sn) composite materials by hot pressing

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Structure formation processes were investigated depending on sintering temperature in hot pressing of diamond-(Fe–Cu–Ni–Sn) composition and their impact on physical and mechanical properties of obtained composite materials. Properties of the composites, such as hardness, wear resistance and strength limits in compressing and folding were studied. It was found that the composite obtained by hot pressing in the temperature range of 20–1000°C and under a pressure of 0.5–40 MPa had the best properties. The results were compared with mechanical properties of composites obtained earlier by sintering in molds placed in a furnace with hydrogen environment at 800°C during 1 h with subsequent hot repressing under different pressures.

Keywords: diamond, iron, copper, nickel, tin, interaction, interlayer, metal-bindered, composite, wear resistance.

Проведены исследования процессов формирования структуры в зависимости от температуры спекания при горячем прессовании композитов алмаз-(Fe–Cu–Ni–Sn) и их влияние на физико-механические свойства полученных материалов. Изучены свойства композитов, такие как твердость, износостойкость, а также границы их прочности при сжатии и изгибе. Установлено, что композит, полученный горячим прессованием в диапазоне температур 20–1000°C и при давлении 0,5–40 МПа, обладает наилучшими свойствами. Результаты исследования сопоставляются с механическими свойствами композитов, полученных ранее путем спекания в пресс-формах в печи в среде водорода при 800°C в течение 1 ч с последующим горячим допрессовыванием при разных давлениях.

Особливості отримання композиційних матеріалів алмаз-(Fe–Cu–Ni–Sn) гарячим пресуванням. *Е.С.Геворкян, В.А.Мечник, М.О.Бондаренко, Р.В.Вовк, С.В.Литовченко, В.А.Чишкала, О.М.Мельник*.

Досліджено процеси структуроутворення в залежності від температури спікання при гарячому пресуванні композитів алмаз-(Fe–Cu–Ni–Sn) та їх вплив на фізико-механічні властивості одержаних композитів. Вивчено такі властивості композитів як твердість, зносостійкість, границі міцності під час стискання та згинання. Встановлено, що найкращі властивості має композит, який отриманий гарячим пресуванням в інтервалі температури 20–1000°C і тиску — 0,5–40 МПа. Результати зіставлено з механічними властивостями раніше отриманих композитів спіканням у прес-формі у печі в середовищі водню за температури 800°C впродовж 1 год. з наступним гарячим допресовуванням за різного тиску.

1. Introduction

Scientific and technical progress is in large part distinguished for application of the new composite materials with predetermined properties [1, 2]. This range of materials includes diamond-containing composite materials (DCM) which have the advantages of having high mechanical and performance properties, particularly hardness and wear resistance, as well as resistance to aggressive influences [3, 4]. A special place among the mentioned materials is occupied by DCM with metallic binders containing iron, copper, nickel, and tin. They are used to make a wide range of tools for stone processing industry [5–10]. This is why research on enhancement of wear resistance of current and development of the new DCM with a required complex of physical and mechanical properties is important today. Analysis of literature references [11–21] has shown that there are two ways to enhance wear resistance of DCM, viz:

— to apply hot repressing technologies which ensure interaction of carbon that precipitates in graphitization of diamond grain surfaces at the stage of sintering of the composition in a pressing mold, with α-Fe (it is inhibited by formation of the diamond-metal binder in the liquid (Cu–Sn) phase);

— to apply the hot pressing techniques to obtain the DCM, which makes it possible to substantially reduce time for sintering and, as a result, to reduce the amount of carbon precipitating in graphitization of diamond grain surfaces.

Consistent patterns of structure formation in diamond-(51Fe–32Cu–9Ni–8Sn)* (Hereinafter the composition is given in wt.% with respect to the metallic binder) system during furnace sintering in the molds in hydrogen environment for 1 hour with subsequent hot repressing are studied in [13, 14], while influence of process parameters of the hot pressing on the DCM structure and physical and mechanical properties have scarcely been studied. At the moment, no consistent patterns of influence of structural and phase transformations, which take place in the hot pressing, on formation of such composites have been established either. However, such technique looks economically sound and promising with regard to high mechanical properties of the composites [15–21]. Therefore, we studied a possibility to obtain this kind of DCM with improved properties by the hot pressing.

The objective of this work was to study influence of sintering temperature on structure formation processes and physical and mechanical properties of diamond-(51Fe–32Cu–9Ni–8Sn) composite materials obtained by the hot pressing in the pressure range of 0.5–40.0 MPa, as well as to compare obtained results with the data presented in paper [14].

2. Experimental

The objects of our experimental investigation were as follows: 51Fe–32Cu–9Ni–8Sn output mixture for metal binders before and after sintering, preliminarily ground DCM specimens (10 mm in diameter, 8 mm in thickness) and thin foils made of these specimens (thickness of 80–100 nm). As source materials for preparation of the DCM specimens, we used AC50 diamond powders with a grit size of 125/100, PZh1M2 iron powder, PNE nickel powder, PMS-1 copper powder, and PO-1 tin powder. Iron, copper, nickel and tin powders were mechanically treated in a tumbling mill by milling balls made of high density aluminum-oxide ceramics in a dry milling mode with the circulation velocity of the mill of 200 rpm, which ensured impact-and-shear effect on the powders. The weight ratio of the milling balls to the powder charge was 5:1, treatment time — 10 h. The powder mixtures for 51Fe–32Cu–9Ni–8Sn metal binder were mixed in alcohol media. We added diamond powder wetted with glycerin to them in amount of 1.54 carat per 1 cm^3 of furnace charge, which corresponds to the relative concentration of $K = 35$ %, and mixed it up without the milling balls [22, 23]. Sintering was carried out by hot pressing under pressure of 0.5–40 MPa in temperature ranges of 20–600; 20–800; 20–900 and 20–1000°C. Hot pressing of the DCM specimens was carried out by putting the time-variant flux through graphite pressing molds in vacuum [24]. Heating up to the holding temperature was carried out at a constant rate of 200 degrees/min. Sintering temperature changes served as criteria of change in the DCM structure and properties.

The microstructure of the metal binder and diamond-metal binder transition zone in the DCM specimens and corresponding diffraction pictures were studied using a transmission electron microscope SELMI TEM-125K microscope with resolving power of 0.18 nm. The quantitative composition of the metallic binder was calculated using the full-profile analysis method in MAUD package.

Thin foils for studies were polished in a solution of 20 % $HClO_4$ + 30 % HNO_3 + H_2O. X-ray diffraction patterns were obtained by DRON 4.13C diffractometer in copper anode radiation in the Bragg-Brentano geometry in the range of angles $20° \leq 2\theta \leq 90°$. Processing of the X-ray diffraction patterns was carried out using X-powder software [25]. The diffraction spectrum of the specimens as a set of specified values of interplane distance d_i of the phase crystal lattice and comparative intensity of reflexes of this phase I_i was identified by comparing it with the reference spectrum using JCPDS (ASTM) files [26]. The transition zone surface morphology and its chemical composition were studied using SELMI SEM-106M scanning electron microscope at an acceleration potential of 20 kV. The quantitative calculations of the chemical composition were carried out using ZAF correction techniques and Magelanes 3.1 software package. The composition analysis errors were as follows: ~ 0.01 % for heavy elements and ~ 1 % for light elements (by weight). Microhardness of the specimens was measured using PMT-3 unit and Vickers indentor at 4.9 N loading. Impressions were made in phases which have no diamond grains. The print sizes were measured at a 25-fold magnification. The hardness was calculated according to the following formula: $H_b = 0.4636P/x^2$, where P is the load and x is the diagonal of impression. The bending and crushing strengths were studied using the standard technique (error ≤ 5 %). The wear rate was based on loss of weight in the specimens. The specimens were weighed on VLA-20g-M analytical weighing scale with a precision of ±4 mg.

3. Results and discussion

The results of the X-ray diffraction investigation of DCM specimens with composition of diamond-(51Fe–32Cu–9Ni–8Sn), which were obtained by hot pressing under pressure of 40 MPa at temperatures of 600 and 800°C are presented in Fig. 1. In the X-ray pattern for the DCM specimen obtained at 600°C (Fig. 1a), the registered diffraction peaks due to planes (111), (200), (220), and (311) for copper phases (lattice parameter $a = 0.3615$ nm); (110), (200), and (211) for iron phases ($a = 0.28664$ nm); (111), (120), and (220) for diamond phases ($a = 0.35667$ nm); (551), (660), and (844) for Cu_9NiSn_3 phases ($a = 1{,}801$ nm), correlate with the data in the ASTM files [26] and corresponding the experimental data

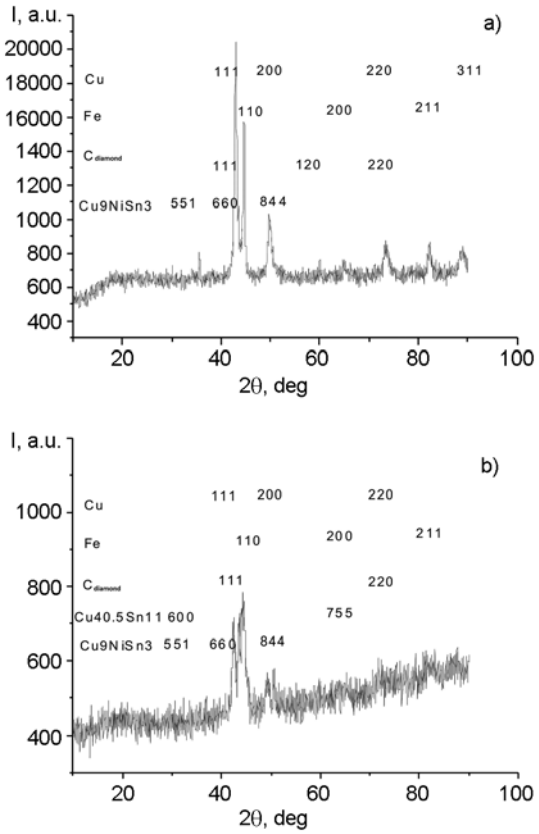

Fig. 1. X-ray pattern from surface of DCM specimens obtained from the diamond-(51Fe–32Cu–9Ni–8Sn) charge by hot pressing under pressure of 40 MPa. (a) At temperature of 600°C; (b) at temperature of 800°C.

for copper and iron from [27]. Analysis of the obtained data shows that during hot pressing of the studied composition, one can observe interaction of components (copper, nickel, and tin) with created Cu_9NiSn_3 intermetallic compounds already at the temperature of 600°C. As shown in the X-ray pattern (Fig. 1b), when the temperature is increased from 600 to 800°C, apart from diffraction peaks due to planes (111), (200), and (220) for copper phases; (110), (200), and (211) for iron phases; (111) and (220) for diamond phases; (551), (660), and (844) for Cu_9NiSn_3 phases, we also found diffraction peaks due to planes (600) and (775) for $Cu_{40.5}Sn_{11}$ phases, which agrees with reference data of [26] and suggests influence of the sintering temperature on phase formation during formation of the composition by the hot pressing.

The diffraction data (interplane distance, relative intensity, angles 2θ), phase angles and crystal lattice indices (*hkl*), which correspond to the X-ray diffraction peaks), are

Table 1. Characteristics of DCM specimen obtained at 600°C

2θ, deg	Intensity, I_i	d_i [nm]	Phase	*hkl*
35.600	396.17	0.2521	Cu_9NiSn_3	551
42.8727	1740.52	0.2109	Cu_9NiSn_3, Cu, C	660, 111, 111
44.6182	761.82	0.2030	Fe	110
49.8545	489.58	0.1829	Cu_9NiSn_3, Cu	844, 200
59.9394	335.76	0.1543	Cu_9NiSn	120
64.9818	359.80	0.1435	Fe	200

Table 2. Characteristics of DCM specimen obtained at 800°C

2θ, deg	Intensity, I_i	d_i [nm]	Phase	*hkl*
29.5879	616.63	0.30192	Cu_9NiSn_3, $Cu_{40.5}Sn_{11}$	551, 600
42.4848	838.69	0.21278	$Cu_{40.5}Sn_{11}$	600
43.5515	917.95	0.20781	$Cu_{40.5}Sn_{11}$	111
44.2303	988.82	0.20478	Cu_9NiSn_3, Cu	111
46.6182	761.82	0.20351	C	110
49.46678	489.58	0.18290	Fe	844
50.6303	678.86	0.18030	Cu_9NiSn_3	200, 755
59.6485	335.76	0.15501	Cu, Cu_9NiSn	120
81.9515	391.59	0.11757	Fe	211

presented in Tables 1 and 2. The analysis of the obtained results shows that in the process of DCM specimens formation, an interaction of elements takes place, which, depending on the hot pressing temperature, results in creation of solid-state solutions based on iron and copper. This is supported by the change of parameters in the crystal lattice compared to relatively pure elements for iron (a = 0.2864 nm) and copper (a = 0.3615 nm), and intermetallides Cu_9NiSn_3, Cu_9NiSn, $Cu_{40.5}Sn_{11}$.

For the DCM specimen obtained at 900°C, the registered diffraction peaks due to planes (111), (200), and (220) for copper phases (lattice parameter a = 0.3615 nm); (110) and (211) for α-Fe phases (a = 0.28664 nm); (101) and (103) for γ-Fe phases (a = 0.3615 nm); (111) and (220) for diamond phases (a = 0.35661 nm); (400), (660), (755), and (888) for $Cu_{40.5}Sn_{11}$ phases (a = 1,80.011 nm), correlate with the data in the ASTM files of [26] and corresponding experimental data for copper and iron from [27].

The study of this specimen showed that it was heterophased and consisted of Cu, α-Fe and γ-Fe, $C_{diamond}$, and $Cu_{40.5}Sn_{11}$ phases. At increase of temperature up to 1000°C, we found diffraction peaks due to planes (111), (200) and (220) for copper phases; (100), (101), and (103) for γ-Fe phases (a = 0.3615 nm); (111) and (220) for diamond phases (a = 0.35661 nm); (400), (660), (840), (755), (888), and (144) for $Cu_{40.5}Sn_{11}$ phases (a = 1,80011 nm). One can see that the obtained results well correlate with the data in the ASTM files [26] and the corresponding experimental data for copper and iron from [27]. A distinctive feature of the obtained data is that at 1000°C of hot pressing, the polymorphic transformation of the volume-centered lattice to the face-centered lattice of iron takes place.

The diffraction data ((interplane distance, relative intensity, angles 2θ), phases and crystal lattice indices (*hkl*), which correspond to the X-ray diffraction peaks), for the DCM specimens obtained at 900 and 1000°C are presented in Tables 3 and 4, accordingly. The interplane distance and relative intensity values obtained from the performed X-ray diffraction studies well correlate with the data in the ASTM files [26]. The diffraction data analysis (Table 3) showed that in the process of DCM specimen formation by the hot pressing at 900°C, the interaction of elements resulted in creation of $Cu_{40.5}Sn_{11}$ intermetallides and solid-state solutions based on iron and copper.

Table 3. Characteristics of DCM specimen obtained at 900°C

2θ, degrees	Intensity, I_i	d_i [nm]	Phase	*hkl*
35.5030	413.60	0.25286	$Cu_{40.5}Sn_{11}$	400
42.3879	996.21	0.21326	$Cu_{40.5}Sn_{11}$, Fe	660, 100
43.4545	852.16	0.20825	Cu	111
44.2303	629.21	0.20478	C, Fe	111, 110
49.2727	624.71	0.18494	γ-Fe	101
50.5333	545.48	0.18062	Cu, $Cu_{40.5}Sn_{11}$	200, 755
72.3515	524.27	0.13061	$Cu_{40.5}Sn_{11}$	888
74.3879	548.53	0.12753	γ-Fe	110
82.3394	548.53	0.11711	Fe	211
87.6727	523.85	0.11131	γ-Fe	103

Table 4. Characteristics of DCM specimen obtained at 1000°C

2θ, degrees	Intensity, I_i	d_i [nm]	Phase	*hkl*
35.6000	222.94	0.25219	$Cu_{40.5}Sn_{11}$	400
42.4848	984,29	0.21278	$Cu_{40.5}Sn_{11}$, Fe	660, 100
43.3576	343.99	0.20870	Cu	111
44.0364	245.24	0.20564	C	111
44.5212	294.45	0.20351	$Cu_{40.5}Sn_{11}$	840
46.0727	197.89	0.19701	Cu	200
49.2727	314.93	0.18494	γ-Fe	101
72.4485	262.84	0.13046	$Cu_{40.5}Sn_{11}$	888
74.2909	215.31	0.12767	γ-Fe	110
78.0727	220.77	0.12241	$Cu_{40.5}Sn_{11}$	144
87.7697	265.13	0.11121	γ-Fe	103

This is supported by the change of parameters in the crystal lattice compared to the relatively pure elements for iron (a = 0.2864 nm) and copper (a = 0.3615 nm). At increase of the hot pressing temperature from 900 to 1000°C, the first-kind phase transition takes place — γ-modification with the face-centered cubic arrangement is formed (Table 4). The obtained results demonstrate that to obtain the DCM, which is composed of diamond-(51Fe–32Cu–9Ni–8Sn), with improved mechanical characteristics, the hot pressing need to be carried out in the range of temperature from 20 to 1000°C under a pressure of 40 MPa.

Thus the X-ray diffraction studies analysis suggests that the obtained DCM of the same composition are very much different by their structural features depending on the hot pressing temperature. An increase of temperature up to 1000°C resulted in the active interaction of elements with formation of γ-Fe and $Cu_{40.5}Sn_{11}$ phases and the solid-state solutions based on copper.

Results of the structural investigation of metallic binder areas by applying scanning electronic microscope (SEM) investigation techniques to the DCM specimens obtained from exit charge depending on the hot pressing temperature are presented in Fig. 3. The binder structure is heterophase and is composed of grains in dark, grey and light colors, and depends on the sintering temperature (Fig. 3, a–d). At a sintering temperature of 600°C, discontinuities and pores at interphase boundaries (Fig. 3a) are observed, which deteriorates the mechanical and performance properties of such DCM. If the sintering temperature is increased up to 800°C, the metallic binder structure is visibly improved (Fig. 3b). If the temperature is further increased, there are absolutely no

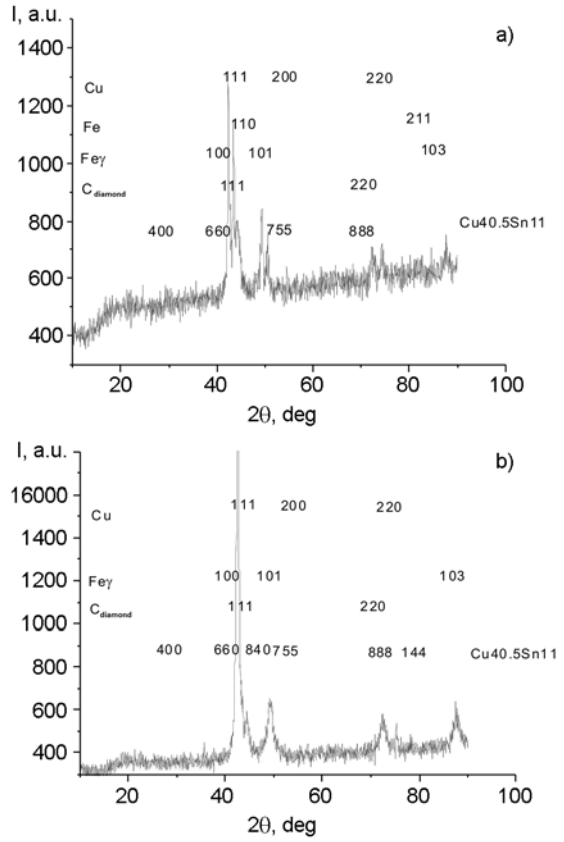

Fig. 2. X-ray pattern from surface of DCM specimens obtained from the diamond-(51Fe–32Cu–9Ni–8Sn) charge by hot pressing under pressure of 40 MPa. (a) At temperatures of 900°C; (b) at temperatures of 1000°C.

pores, and the grain boundaries are thin and sharply defined, and have a hard contact (Fig. 3,c,d), which improves the mechanical and performance properties of these DCM. The formed phases have a variety of configurations and sizes. Thus, the elements of the metallic binder structure are considerably different in their element composition, and some elements of its structure are much dispersed without any clear discontinuities, which is due to the influence of the hot pressing temperature on the interaction of elements in the process of composite formation. This may produce a significant economic effect in producing of the DCM and tools on their basis.

The structural investigation results of the obtained DCM specimens by applying the scanning electronic microscope investigation techniques are presented in Fig. 4. It can be seen that the metallic binder structure surrounded by diamond grains is composed of copper phases, and the diamond-metallic binder contact is hard, the grain boundaries are thin, sharply defined, and have no visible pores or cracks (Fig. 4a), which helps to improve the diamond-holding and performance properties. In this case the metallic binder structure has the granular material structure (copper phase) with inclusions of another phase both at the grain boundaries and regularly distributed in the grain matrix. One can clearly see the copper phases on the ring electron diffraction patterns of this specimen (Fig. 4b). The grains of this phase are (200), (111) and (220)-textured. Diffraction peaks on the ring patterns suggests the presence of texture in the specimen under investigation.

It was found that the dislocation picture of the metallic binder is heterogeneous, with definite peaks and troughs in etch pit density, which are identified as emergence of dislocations on the copper grains surface (Fig. 4c). In addition, the intense generation of defects takes place.

The electron microscopic studies demonstrate practically straight slip bands and parts of another phase (Fig. 4c). It is worth noting that slip bands in the adjacent grains have different orientation. This goes to prove that every grain is deformed in a different way shifting relatively to one another. Breaking of the connection between them leads to cracking. The properties that define a distinguishing feature of the metallic binder microstructure is presence of Cu_9NiSn_3 phases (Fig. 4e) and numerous dislocation loops which are formed under conditions of interaction of the elements in sintering. The density of the formed (Cu_9NiSn_3) intermetallides is a bit higher close to the finest particles of iron. Analyzing the microstructure of the obtained specimens, it is worth noting that the metallic binder is characterized by absence of pores on the interphase boundaries, which helps to improve their thermophysical and mechanical properties. One can clearly see the presence of copper (Fig. 4b,d), iron and Cu_9NiSn_3 phases (Fig. 4f) on the ring electron diffraction patterns (EDF) of this specimen, which suggests a three-phase structure of the metallic binder. The copper phase grains are (111) and (220)-textured. The iron phase grains are (111)-textured, and Cu_9NiSn_3 phase grains are (511)-textured.

Increase of the hot pressing temperature up to 900°C resulted in some structural phase changes in the DCM specimen structure (Fig. 5). For example, $Cu_{40.5}Sn_{11}$ (Fig. 5a), γ-Fe and Cu (Fig. 5c) phases, as well as γ-Fe (Fig. 5e) phases were found in the metallic binder structure. The grain bounda-

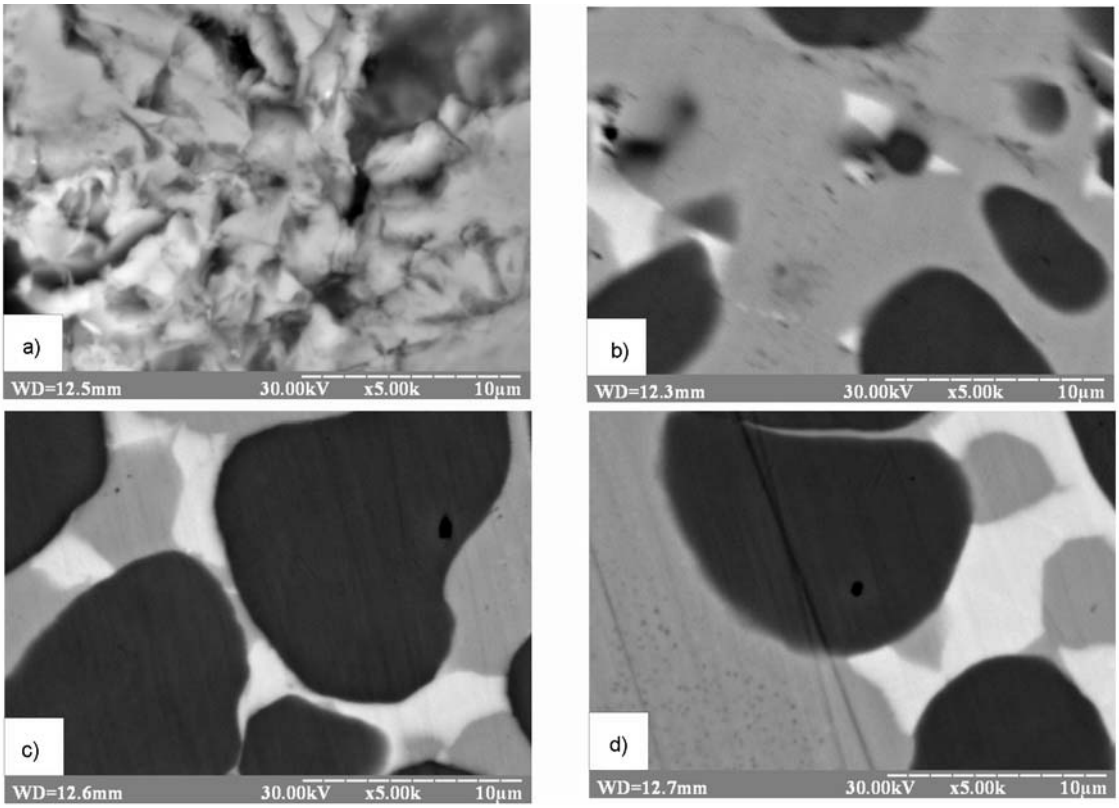

Fig. 3. Microstructure of areas of metallic binder in DCM specimens obtained by hot pressing at different temperatures. (a) 600°C; (b) 800°C; (c) 900°C; (d) 1000°C.

ries are thinner, sharply defined, have a the hard contact and no visible pores. One can clearly see Cu and γ-Fe phases (Fig. 5d, f) on the ring electron diffraction patterns of the metallic binder areas. The copper phase grains are (111) and (220)-textured, and the γ-Fe phase grains are (111) and (200)-textured. Continuing the analysis of the structure formation in the process of composition formation at 900°C, it should be noted that no Cu_9NiSn_3 and Cu_9NiSn brittle intermetallides were formed unlike the composition formed at 800°C. The considerable difference of the metallic binder structure of the DCM specimen obtained at 900°C from the metallic binder structure of the DCM specimen made at 800°C is that there are no defects on the interphasal boundaries (see Fig. 5c, e and Fig. 4c, e). In this case defects are either closed up under the temperature impact by falling into dislocation loops or grow under tensile stress releasing the internodal loops. The obtained results suggest active interaction of the elements in the process of composition formation, which depends on temperature, and this helps to influence on the phase formation and physical and the DCM mechanical properties.

The main difference of the DCM specimen structure obtained at 1000°C from the DCM specimen structures obtained at lower temperatures (800 and 900°C) is that it has absolutely no defects, inconsistencies and pores both on the diamond-metallic binder interface (Fig. 6a) and interphasal boundaries in the composition metallic binder (Fig. 6c, e). In this case the metallic binder structure is composed of γ-Fe (Fig. 6a), Cu (Fig. 6c) and $Cu_{40.5}Sn_{11}$ (Fig. 6e) phases. One can clearly see γ-Fe (Fig. 6b), Cu (Fig. 6d) and $Cu_{40.5}Sn_{11}$ (Fig. 6f) phases on the ring electron diffraction patterns (EDF) of the metallic binder areas. The γ-Fe phase grains are (111) and (200)-textured, and the Cu phase grains are (111), (200) and (220)-textured.

Diffraction peaks on the ring patterns suggest presence of texture in the specimen under investigation. It should be noted that the obtained data show no graphite inclusions in the metallic binder structure which produce a negative impact on formation of DCM mechanic properties including wear resistance [13, 14]. Below, the influence of techniques and process parameters of obtaining of DCM under investigation on their

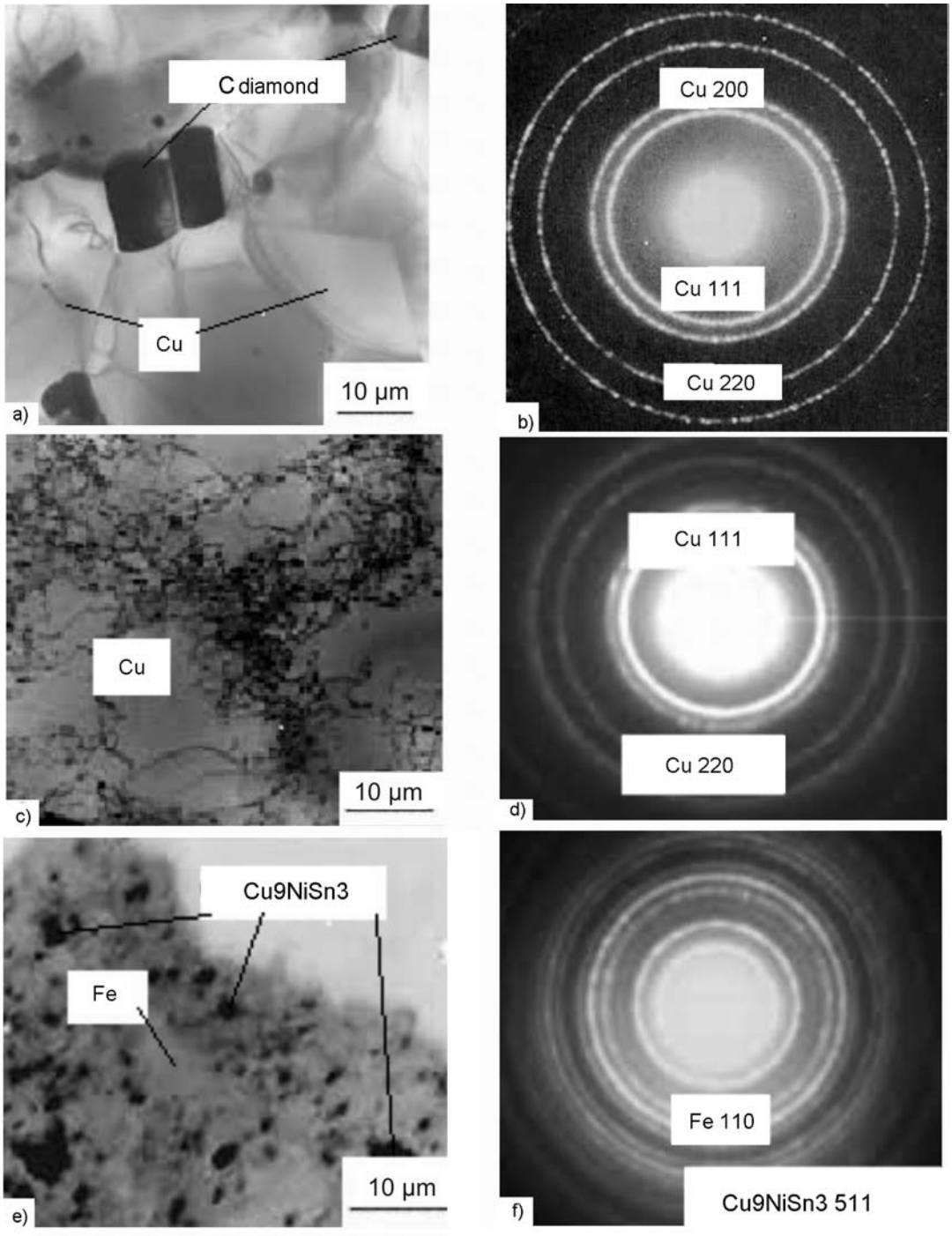

Fig. 4. DCM specimens obtained from the exit charge by hot pressing in the range of temperatures from 20 to 800°C under pressure of 40 MPa. (a, c, e) Electron microscopic images of surfaces; (b, d, f) microelectron diffraction patterns of its fragments.

physical and mechanical properties, and wear resistance are investigated.

Comparison of peculiarities of structure formation in the diamond-(Fe–Cu–Ni–Sn) system in the process of hot pressing and furnace sintering in the pressing mold with hot repressing. Comparison of peculiarities of the structure formation in the diamond-(51Fe–32Cu–9Ni–8Sn) composition in the process of hot pressing (first type specimens) with peculiarities of the structure formation in similar composition in the process of its formation by sintering in molds in a furnace in hydrogen media at

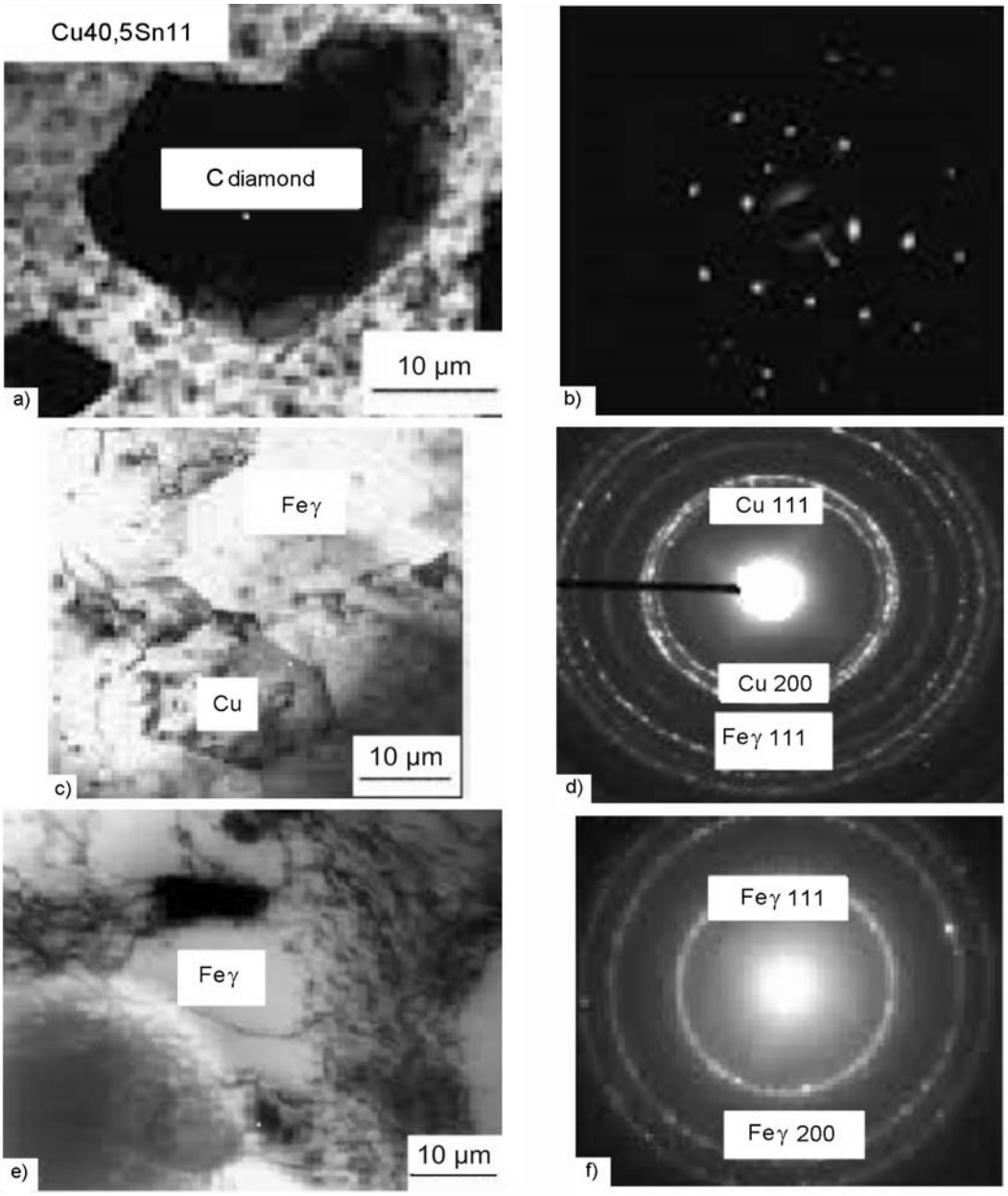

Fig.5. DCM specimens obtained from the exit charge by hot pressing in the range of temperatures from 20 to 900°C under pressure of 40MPa. (a, c, e) Electron microscopic images of surfaces; (b) dark area in the diamond reflection; (d, f) microelectron diffraction patterns of fragments of DCM specimen.

800°C during 1 h with consequent hot pressing (second type specimens) [14] showed both similarity and fundamental differences. In particular, it was shown that the metallic binder structure of the first and second type DCM specimens obtained at 800°C is composed of solid-state solutions based on iron, copper and Cu_9NiSn_3 intermetallides. One should note the presence of $Cu_{40.5}Sn_{11}$ phases in the metallic binder structure of the first type, and presence of Ni_3Sn phases in the metallic binder structure of the second type.

Analyzing the peculiarities of phase formation in the investigated composition, notice that depending on techniques and process parameters of obtaining the DCM specimens, interaction of elements occurs in different ways, which is the main reason for changing their structures and properties. The results obtained by the hot pressing at 900 and 1000°C demonstrate a significant improvement of the DCM specimens struc-

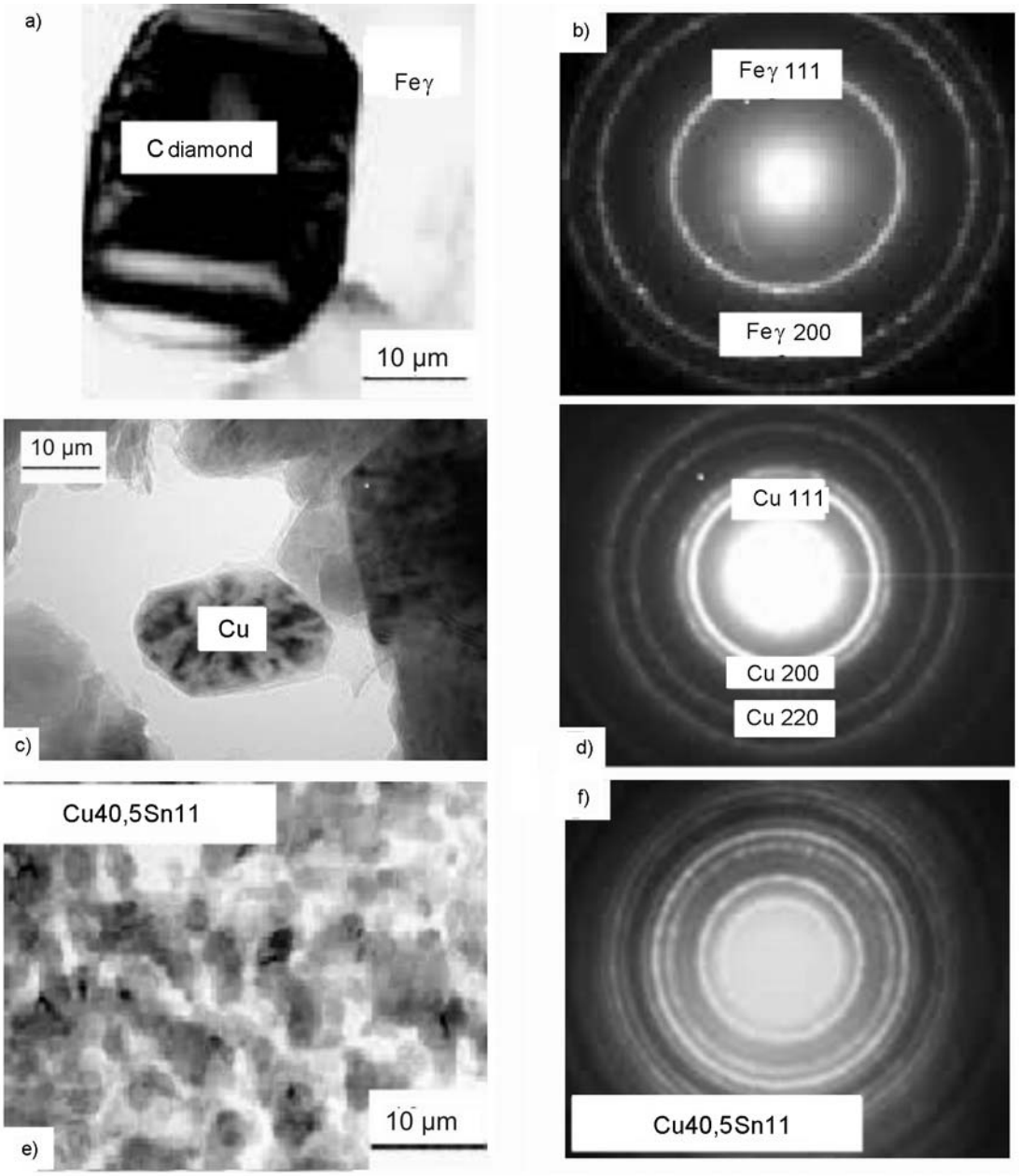

Fig. 6. DCM specimens obtained from the exit charge by hot pressing in the range of temperatures from 20 to 1000°C under pressure of 40 MPa. (a, c, e) Electron microscopic images of surfaces; (b, d, f) microelectron diffraction patterns of its fragments.

ture through formation of γ-Fe phases in the metallic binder (polymorphic transformation of the volume-centered lattice to the face-centered lattice of iron), which could not be formed at the lower temperatures (see Fig. 2, 5, and 6) or through decomposition of the brittle Cu_9NiSn_3 phase.

In accordance with the change in the elements interaction in the composition in the process of the second type DCM specimens, the substantial changes in their structures and physical and mechanical properties take place. The most significant difference in the structure of these specimens is the presence of a transition zone on the diamond-metallic binder boundary which affects their mechanical properties (Fig. 7) [14]. Analysis of the said investigation showed that the structure of the transition zone on the diamond-metallic binder boundary differs from the metallic binder structure because it formation is affected by the carbon deposed during graphitization of the diamond grains surfaces at the sintering stage. One can see

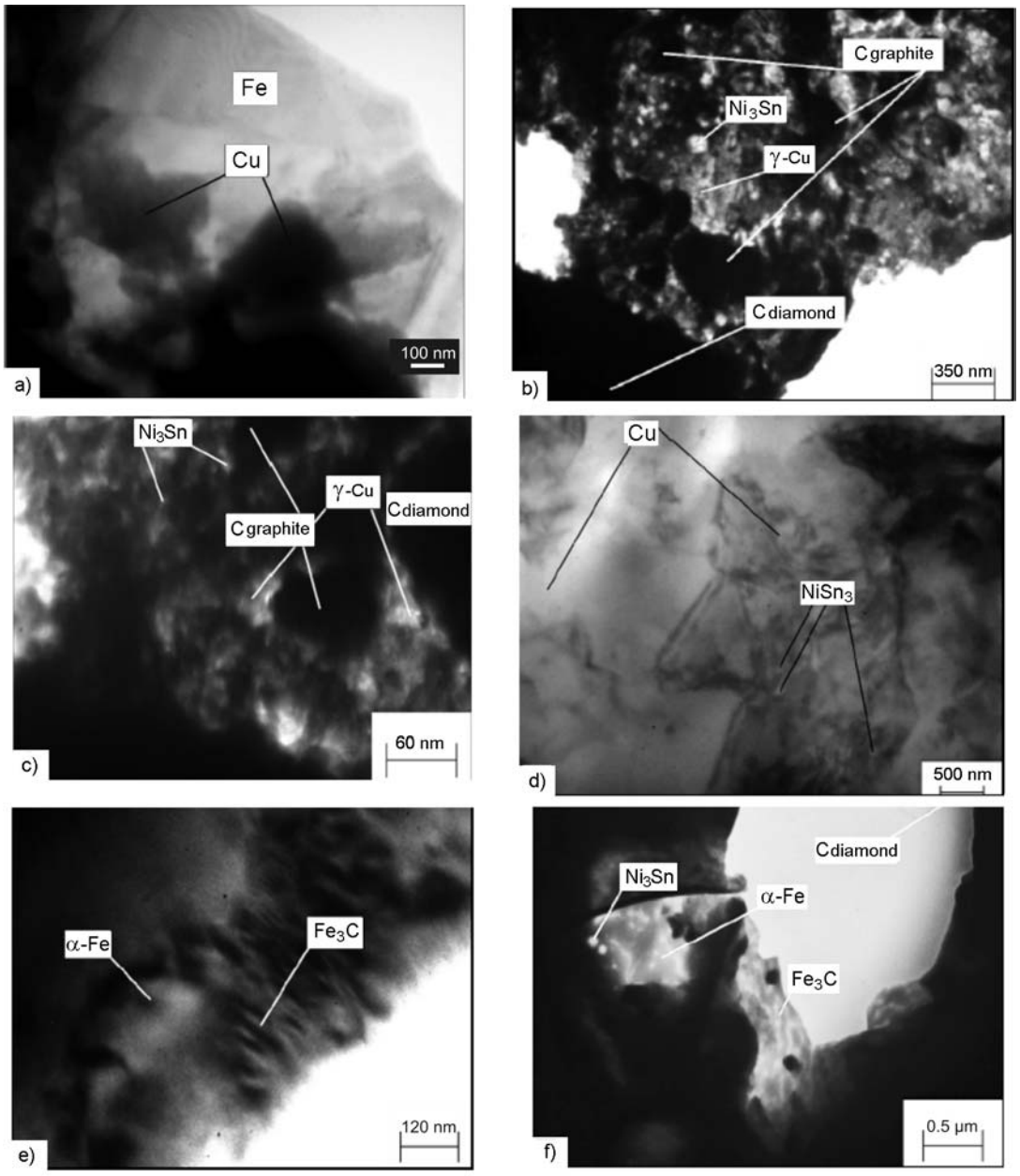

Fig. 7. Electron microscopic images of areas of the metallic binder (a, d) and the diamond-binder transition zone (b, c, e, f) of DCM specimens obtained from the exit charge by sintering in molds in a furnace at 800°C during 1 h with hot repressing under the following pressures: (a, b) — p = 100 MPa, t = 3 min; (c) p = 160 MPa, t = 3 min; (d, e) p = 200 MPa, t = 2 min; (f) p = 200 MPa, t = 3 min.

(Fig. 7a, d) that irrespective of the process parameters of hot repressing of the diamond-(51Fe–32Cu–9Ni–8Sn) composition, the metallic binder structure is composed of the solid-state solutions based on copper and iron as well as Ni_3Sn and Cu_9NiSn_3 compounds. Then the grain boundaries are thin, sharply defined, have the hard contact and have no visible pores or cracks. However, the structure of the transition zone in the DCM specimens obtained under insufficient pressure (below 200 MPa) and at insufficient time of the hot pressing (less than 3 min.) is composed of Cu, Ni_3Sn phases with graphite inclusions (Fig. 7b, c), which is the reason for its early fracturing by the intensive cracking mechanism (Fig. 8a) and falling out of the diamond grains from the binder (Fig. 8b, c), which results in rapid wear of the DCM. It should be noted that in the process of formation of these DCM, the eutectic liquid in the Cu–Sn system is in

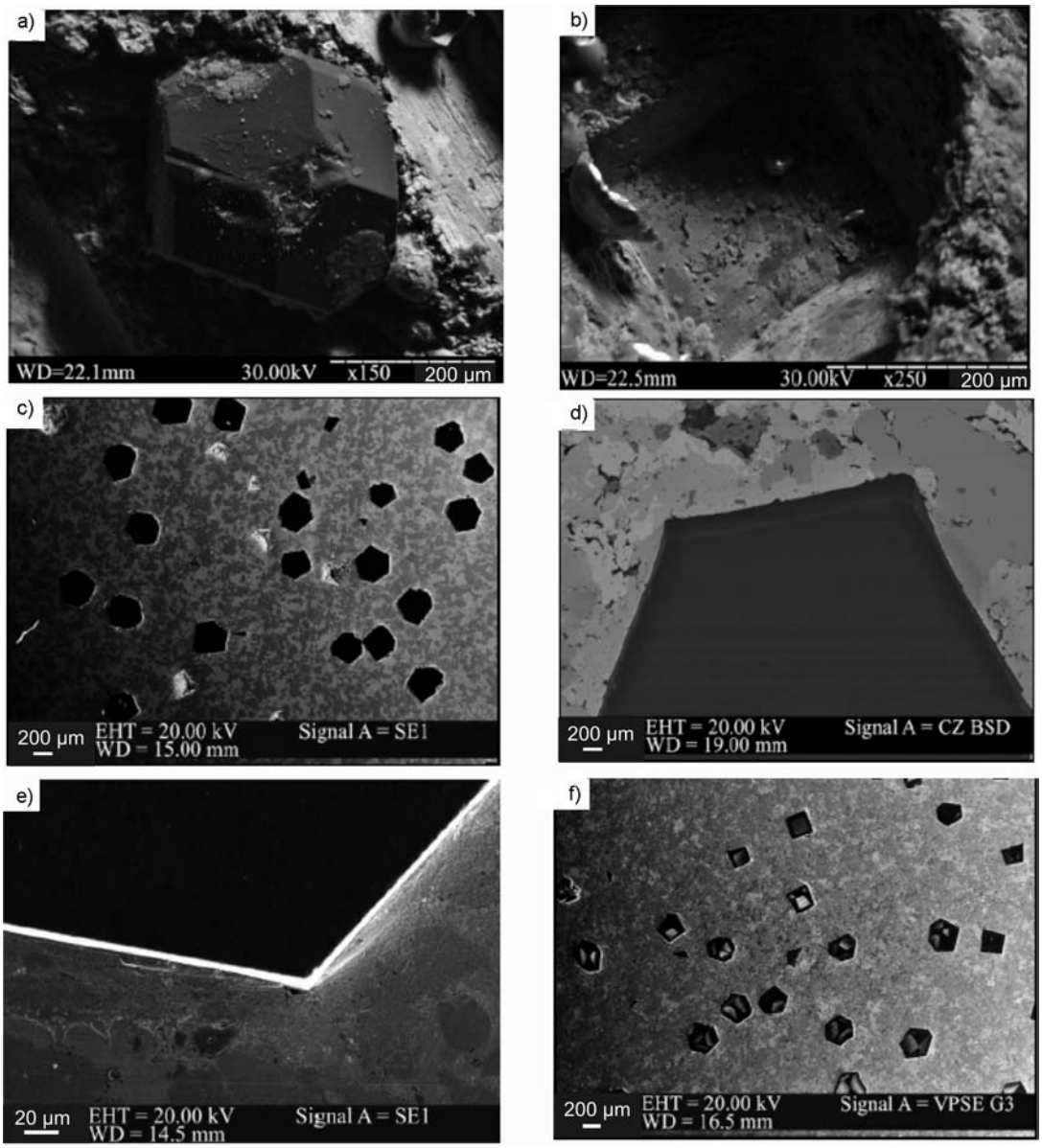

Fig. 8. Microscopic images of surfaces of DCM specimens obtained from the exit charge under conditions similar to those shown in Fig. 7. (a, b, d, e) SEM images; (c, f) cathodoluminescence topographs.

contact with the diamond grains and prevents interaction of carbon, which is deposed during graphitization of diamond grains surfaces at the sintering stage, with the α-Fe solid-state phase. All this gives rise to formation of graphite inclusions in the transition zone, which impairs the DCM mechanical properties.

The main difference of the transition zone structure of the DCM specimens obtained under pressure of 200 MPa from the transition zone structure of the DCM specimens made under the lower pressure (100 and 160 MPa) is that the former have Fe_3C nanoscale thickness layers and have no graphite inclusions (Fig. 7e, f). Improvement of the structure of such DCM occurs because during the composition hot repressing, the carbon deposed during graphitization of diamond grains surfaces interacted with α-Fe and formed iron carbides, which significantly improves diamond-holding (Fig. 8d–f) and enhances performance properties of the DCM. In this case the height of diamond grains in the composite during its performance (cutting of granite with a fluid-cooled segmented diamond cutting wheel) exceeds half of their diameter

Table 5. Mechanical properties of obtained DCM specimens

Pressure, MPa	Process time, min	Microhardness H_v, GPa				Strength, MPa	
		γ-Cu	Ni_3Sn	α-Fe	γ-Fe	Compression, σ_c	Bending, σ_b
Hot pressing							
20	5	2.88	–	4.18	5.96	842	790
40	5	2.90	–	–	6.10	845	810
Sintering in molds in a furnace with hot repressing [14]							
100	2	2.60	2.76	2.93	–	730	620
160	2	2.70	2.82	3.46	–	750	645
160	3	2.80	3.03	3,98	–	780	655
200	2	2.92	3.48	4.12	–	840	675
200	3	2.99	3.63	4.34	–	846	680

(Fig. 8d, e). Thus, under the optimum pressure and at the optimum time of hot repressing of the composition, the carbon deposed during graphitization of the diamond grains surfaces in sintering becomes an additional source for improvement of the structure and mechanical properties of such DCM.

Thus, the obtained results show the significant differences in the structure of DCM specimens of the first and second types. These differences lay in the construction of the metallic binder surrounded by diamond grains, which depends on techniques and process parameters of obtaining of the DCM specimens due to the fact that interaction may take place in the different ways. When the sintering temperature is increased from 600 to 1000°C in the process of formation of the first type specimens, the structural phase transformations occur. These transformations are accompanied by formation of $Cu_{40.5}Sn_{11}$ and γ-Fe phases, which contributes to the increase of hardness, compression resistance and wear resistance of such DCM. Whereas during formation of the second type specimens with an increase of the pressure of hot repressing from 100 to 200 MPa, structural phase changes occur which lead to creation of Fe_3C nanostructures on the diamond-metallic binder boundary (see Fig. 7e, f), which prevents the carbon deposition in the form of graphite inclusions and has beneficial effect on the enhancement of wear resistance of these DCM.

The results of testing of the investigated DCM specimens for microhardness, compression and bending resistance limits are presented in Table 5. Its analysis has shown that mechanical properties of the DCM specimens depend both on techniques and process parameters of their obtaining. For example, when the hot-pressing pressure is increased from 20 to 40 MPa, one can see insignificant increase of microhardness of the metallic binder areas in Cu, α-Fe and γ-Fe phases. Moreover, maximum compression resistance of the obtained specimens hardly changes, but the limit of strength during bending increases from 790 to 810 MPa. Depending on pressure, the values of microhardness and maximum compression resistance for DCM of the second type change more significantly. However, unlike the specimens of the first type, the maximum bending resistance of these specimens has much lower values.

Improvement of mechanical properties of the first type specimens compared to the second type specimens suggests that in the process of the DCM obtaining, the interaction of elements occurs in the different ways, and first of all it depends on techniques and process parameters of their obtaining, which influence the processes of structure formation, and physical and mechanical properties.

Testing of the DCM specimens for wear resistance was carried out on a special workbench by polishing a quartz sandstone with vertical load of about 10 kg and a sliding velocity of 4 m/sec. during 600 sec. Ordinary water was used as a coolant. Wear resistance of the specimen was defined by weighing. The testing results are presented in Table 6. The research shows that the mass wear of the DCM specimens, similarly to mechanical properties, depends on the techniques and process conditions of their obtaining (Table 6). For example, the comparative analysis of the results shows that the increase of the hot pressure temperature from 600 to 1000°C results in decrease of

Table 6. Wear resistance tests results of the obtained DCM specimen

Technological parameters of obtaining DCM specimens			Mass wear I_m, g
Temperature, °C	Pressure, MPa	Process time, min	
Hot pressing			
20–600			1.50
20–800	40	5	0,39
20–900			0.24
20–1000			0.20
Furnace sintering in molds for 1 h with consequent hot repressing			
	100	2	0,96
	160	2	0.84
800	160	3	0,75
	200	2	0,68
	200	3	0.54

the mass wear I_m from 1.50 to 0.20 g after polishing the quartz sandstone in similar conditions. This can be explained as follows. The structure of the metallic binder of the DCM specimen obtained at 600°C is composed of the solid-state solutions based on copper and iron as well as brittle phases Cu_9NiSn_3 and Cu_9NiSn (see Fig. 1 and Table 1), while on the interphasal boundaries one can see inconsistencies and pores (see Fig. 3a), which decreases the wear resistance. The structure of the DCM specimen obtained at 1000°C is composed of γ-Fe, Cu and $Cu_{40.5}Sn_{11}$ phases, and there are no inconsistencies or pores either on the diamond-metallic binder boundary (Fig. 6a) or interphasal boundaries in the metallic binder of the composite (Fig. 6c, e), which is the reason for increase of the wear resistance.

A similar trend of dependence of wear on pressure remains true for the DCM specimens obtained by the sintering in molds with the consequent hot repressing. It was found that after polishing the quartz sandstone in similar conditions, during the hot repressing, in result of pressure increase from 100 to 200 MPa, the mass wear I_m decreases from 0.96 down to 0.54 g (Table 6). This effect is related to the fact that during hot repressing of the composition, the carbon which was developed due to graphitization of the diamond surfaces at the stage of furnace sintering in molds, interacted with α-Fe and developed nanostructure filled with Fe_3C in the transition zone (see Fig. 7e, f). At that the wear resistance of the DCM specimens obtained by hot pressing in contract to the wear resistance of the DCM specimens obtained by furnace sintering in the molds with hot repressing, has much higher values. The reason for this is that the binder structure surrounded by the diamond grains in the first type specimens is composed of γ-Fe phases (see Fig. 6a), while the binder structure surrounded by diamond grains in the second type specimens is composed of α-Fe phases and interlayers with Fe_3C (see Fig. 7e, f).

Thus the obtained results suggest that the applied technology ensures improvement of the structure and mechanical properties of the diamond-(Fe–Cu–Ni–Sn) composites by forming the metallic binder with increased strength parameters. The determined consistent patterns provide up-to-date and important data both for theoretical science, because they give us a better understanding of already known approaches with regard to forecast of the physical state of diamond-containing compositions during their formation, and for technology, because they make it possible to influence the interaction of elements in required direction and obtain the DCM with new useful properties.

4. Conclusions

We carried out investigation of the structure and properties of diamond-(51Fe–32Cu–9Ni–8Sn) composite materials obtained by hot pressing under pressure of 0.5–40 MPa in the temperatures ranges of 20–600, 20–800, 20–900 and 20–1000°C and by furnace sintering in the mold in hydrogen media at 800°C during 1 h with hot repressing under various pressures. We found the following advantages of the hot pressing as compared to the furnace sintering in molds with the hot repressing:

– It has been proved that with the change of temperature from 600 to 1000°C in the process of hot pressing of the diamond-(51Fe–32Cu–9Ni–8Sn) composition, incremental structure and phase transformations take place, which make it possible to increase the DCM wear resistance by 7.5 times. This well correlates with the change of the phase composition, morphology of the phase components and the composite structure.

– The main reasons for enhancement of wear resistance in the process of hot pressing of the composite at 1000°C are transformation of α-Fe into γ-Fe and formation stable $Cu_{40.5}Sn_{11}$ intermetallides, which ensures high adhesion of diamond grains to the metallic binder, as well as strengthening of the metallic binder and improvement of mechanical properties of the composite materials.

– It has been shown that the key factor in manufacturing the commercially promising DCM by sintering in the molds in a furnace at 800°C during 1 hour is application of the hot repressing (with a pressure not lower than 200 MPa and process time not less than 3 min), which ensures decarburization and formation of nano-interlayers with Fe_3C in the diamond-metallic binder transition zone.

–Bend resistance and wear resistance of the specimens of the composite diamond-containing materials obtained by the hot pressing are correspondingly 1.2 and 2.7 times higher than of those obtained by the sintering in molds in a furnace with the hot repressing.

Thus, application of hot pressing makes it possible to obtain the composite diamond-containing materials for stone processing industry with much better physical and mechanical properties than similar materials manufactured by sintering in the molds in a furnace with consequent hot repressing.

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