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INFLUENCE OF REINFORCEMENT BY THE WHISKERS OF Si_3N_4 AND $Mg_2B_2O_5$ ON THE PROPERTIES OF cBN-BASED COMPOSITES

Three types of cBN-based composites (without whiskers, reinforced with whiskers of Si_3N_4 and reinforced with whiskers of $Mg_2B_2O_5$) have been obtained by High Pressure-High Temperature (HPHT) sintering. Density, Young modulus, hardness, Poisson ratio and fracture toughness have been measured for all samples. cBN-based composites, that were reinforced by the whiskers of Si_3N_4, are characterized by better mechanical properties (hardness, fracture toughness) than non-reinforced cBN-based composites. But reinforcement by $Mg_2B_2O_5$ whiskers was insufficient, because $Mg_2B_2O_5$ whiskers have low thermochemical stability.
***Keywords**: whiskers, reinforcement, cBN, fracture toughness*

Introduction

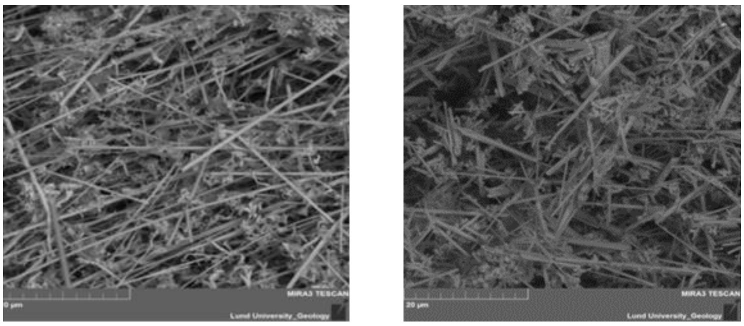
Despite the development of high-performance conventional technology of forming (eg, injection molding, laser machining), cutting materials stay the most universal and popular way of forming machine-building products. It is well-known that during operations the cutting tool is subjected to the influence of temperature and pressure, which causes wear. Therefore, in the perfect case, the material of its cutting part of the tool must meet many requirements: have high hardness, durability, wear resistance, heat resistance, crack resistance (fracture toughness), adhesion resistance and cyclic strength, thermodynamic strength, heat capacity, thermal conductivity, low affinity for machining material, etc. cBN-based materials occupied a special place among the cutting materials due to such characteristics, like high strength, chemical stability over a wide temperature range [1]. HPHT sintering is traditional preparation method of cBN-based materials, which allow to obtain high-density materials with fine microstructure (consequently, these materials have high level of hardness and fracture toughness). Despite on this, the fracture toughness BL group stays unsatisfactory (about 2,5–5 $MPa{\cdot}m^{1/2}$, which is significantly lower than the fracture toughness of BH group – 9–10 $MPa{\cdot}m^{1/2}$) and this leads to a reduction in the service life of tools based on them [2–3]. Therefore, the problem of increasing the fracture toughness of this group of materials becomes obvious while maintaining the chemical stability of these materials [4]. One of the well-known ways to solve this problem is whisker reinforcement [5], which was widely used for other groups of cutting materials, but almost never used for this group [6].

Experimental technique

The starting materials were powders of cubic boron nitride (*d* = 2–6 μm), aluminum (*d* = 1 μm), tantalum nitride (*d* = 5–7 μm), and also whiskers of refractory compounds (Si_3N_4 and $Mg_2B_2O_5$) (with a diameter of 1–2 μm and length around 15–20 μm). The grinding and mixing of powders was carried out in a Fritch Pulverisette 7 planetary ball mill in isopropyl alcohol, which allows to intensify the process. First of all, in order to reduce the grain size of tantalum nitride powder, two grinding cycles of 1 min duration at a speed of 1100 rpm were carried out. To prevent tantalum nitride powder from self-igniting, a pause of 10 minutes was made between the grinding cycles. After that, tantalum nitride powder [7, 8] was mixed with powders of boron nitride and aluminum for 20 minutes. The rotation speed was 250 rpm. [8] After grinding and mixing operations, ultrasonic mixing of the obtained mixture of powders with whiskers of refractory compounds was carried out. Thus, mixtures of powders with different contents (0; 5; 10; 15 vol.%) of Si_3N_4 and $Mg_2B_2O_5$ – whiskers were obtained, which were then precompressed and placed in graphite crucibles for vacuum annealing in order to remove undesirable impurities and excess oxygen. After that, the obtained blanks were sintered in a high-pressure “toroid” type apparatus (HPA) [9, 10]. In order to investigate the effect of temperature, sintering of samples was carried out at different temperatures (1600; 1800; 2000; 2150 °C) and pressure of 7,7 GPa. After sintering, the samples were ground to a round plate. Before further research, the surface of the obtained plates was polished with diamond paste (9 μm and 1 μm), as well as with a colloidal solution of silicon oxide (0,04 μm) until a mirror surface was obtained. The elastic constants of the obtained samples (Young's modulus, Poisson's ratio) were measured by measuring the velocities of ultrasonic waves in a material [11] with the help of Olympus 38DL Plus. The microstructure of the obtained samples was studied using a Hittachi SU8010 Cold Field Emission high-resolution scanning electron microscope, and phase analysis was performed using X-ray diffractometry using CuKα – radiation. The Vickers microhardness of the obtained samples was determined as an average value of 4-5 indentations with a load of 9.8N and an exposure of 15 seconds using a Sematic Durometer. Fracture toughness (K_{1C}) was determined by the method of indentation using the Vickers indenter at higher loads – 24,5 and 49,0 N.

The discussion of the results

The results of measurements of relative density showed that increasing of Si_3N_4 whiskers volume content leads to a deterioration of samples compressibility, despite on the less developed morphology of Si_3N_4 whiskers compared to the $Mg_2B_2O_5$ whiskers morphology (Fig. 1): the relative density of the material with the addition of $Mg_2B_2O_5$ whiskers was 47–48% , the relative density of the material with the addition of Si_3N_4 whiskers was 35–45%) (Fig. 2).



a *b*

Fig. 1. Morphology of whiskers of $Mg_2B_2O_5$ (a) and Si_3N_4 (b) before mixing

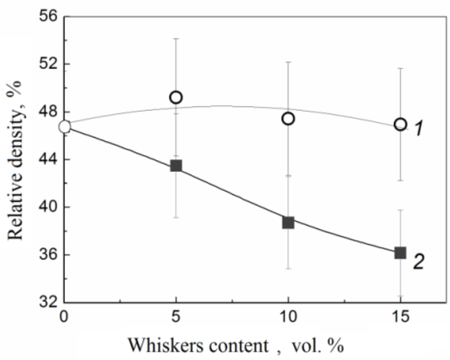
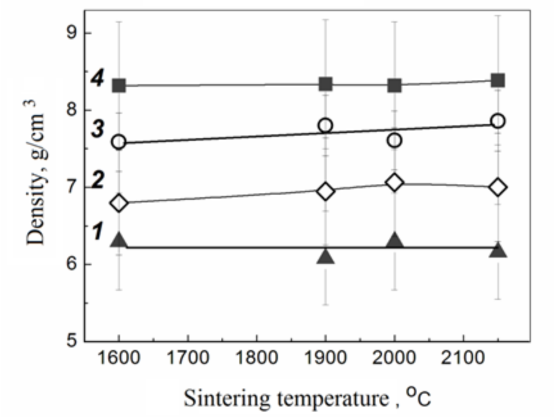


Fig. 2. The dependence of the relative density of green compact samples on the volume content of whiskers of $Mg_2B_2O_5$ (1) and Si_3N_4 (2)

The study of the microstructures of the obtained samples showed that the length of the whiskers in the material was 15–25 microns. This indicates that ultrasonic mixing made it possible to mix whiskers effectively without breaking them, unlike traditional mixing. No significant grain growth up to the sintering temperature of 1900 °C (Fig. 3) was observed.



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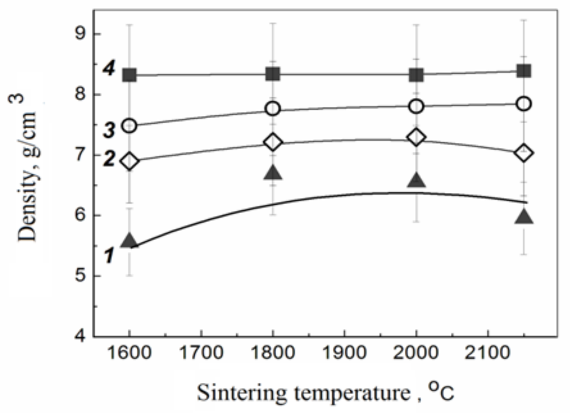


b

Fig. 3. The dependence of the density of cBN-based sintered materials, reinforced 0; 5; 10 and 15 vol. %, (4, 3, 2, and 1, respectively) whiskers of Si_3N_4 (a) and $Mg_2B_2O_5$ (b) versus sintering temperature

At the maximum sintering temperature of 2150 °C, slight recrystallization is observed (Fig. 3), which leads to a slight decrease in hardness (Table).

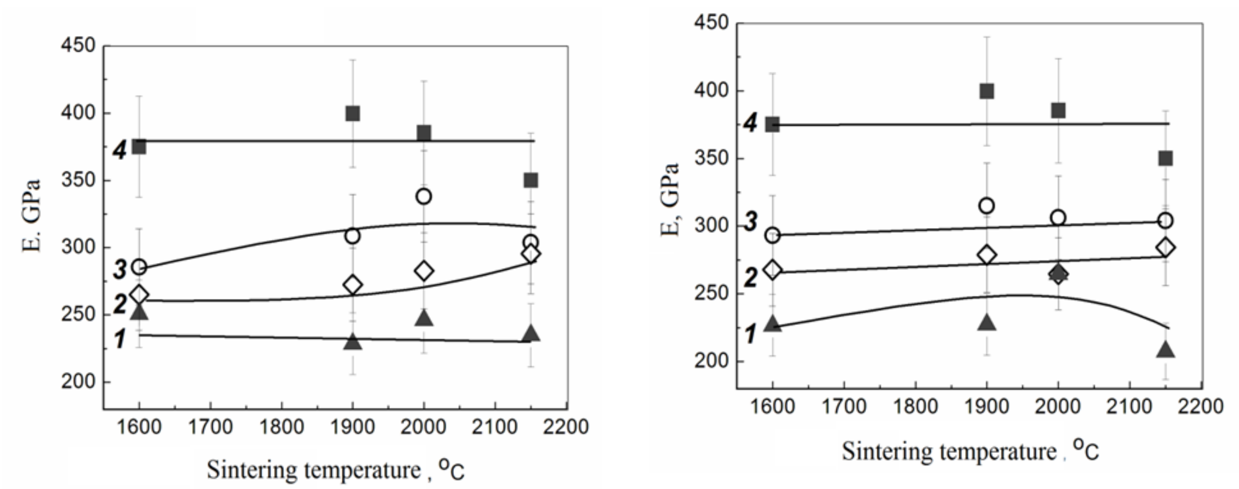
Properties of reinforced and unreinforced cBN-based materials

Chemical composition	*T* sinte-ring, °C	Den-sity, g/cm^3	Poisson ratio, μ	You-ng's modu-lus, GPa	Hardness at different indentation load			Fracture toughness at different indentation load , MPa·m$^{-1/2}$	
					$HV_{9,8}$	$HV_{24.5}$	HV_{49}	K_{1C} (24,5H)	K_{1C} (49H)
1	2	3	4	5	6	7	8	9	10
50% cBN – 5%Al – 45% TaN	1600	8,32	0,37	319	24,00	23,93	22,69	2,79	2,76
	1800	8,34	0,36	325	28,01	26,06	23,51	2,14	3,35
	2000	8,32	0,35	315	28,80	24,27	29,62	2,25	4,08
	2150	8,38	0,35	-	-	-	-	-	-
50% cBN – 5%Al – **5% Si_3N_4** – 40%TaN	1600	7,59	0,38	303	25,00	26,35	25,96	3,35	6,74
	1800	7,80	0,37	308	24,28	32,24	28,74	2,62	4,86
	2000	7,61	0,37	294	28,62	24,72	28,92	3,28	4,49
	2150	7,86	0,40	304	29,89	23,39	28,27	3,20	4,86

End of the table

1	2	3	4	5	6	7	8	9	10
50% cBN-5%Al – **10% Si_3N_4** – 35%TaN	1600	7,59	0,39	302	25,01	20,00	26,00	3,7	4,55
	1800	7,80	0,41	309	30,25	22,31	32,61	3,14	3,86
	2000	7,61	0,40	305	26,18	31,03	32,11	3,79	5,24
	2150	7,86	0,39	331	26,2	29,64	30,51	3,34	6,33
50%cBN-5%Al- **15% Si_3N_4** – 30%TaN	1600	6,80	0,40	271	21,00	23,86	25,24	2,92	5,91
	1800	6,95	0,43	262	30,77	29,84	30,37	2,32	3,84
	2000	7,07	0,40	277	26,98	26,06	28,75	2,83	3,94
	2150	7,00	0,39	268	25,87	25,72	30,13	2,55	4,08
50% cBN-5%Al – **5% $Mg_2B_2O_5$** – 40%TaN	1600	6,92	0,38	287	22,11	20,99	22,50	2,52	2,68
	1800	7,16	0,42	340	22,97	27,99	29,53	3,36	3,89
	2000	7,19	0,38	341	32,34	34,13	33,90	3,13	2,93
	2150	7,25	0,40	344	27,95	30,06	31,16	3,37	3,39
50% cBN-5%Al – **10%$Mg_2B_2O_5$** –35%TaN	1600	6,96	0,38	311		25,5	30,01	3,65	2,5
	1800	7,21	0,41	322		27,58	30,49	2,61	2,84
	2000	7,08	0,41	317		24,13	28,12	2,92	2,59
	2150	7,26	0,41	325		26,33	29,33	2,69	2,49
50% cBN – 5%Al – **15% $Mg_2B_2O_5$** –30%TaN	1600	6,57	0,39	279		21,30	23,50	3,06	2,69
	1800	6,69	0,42	299		26,20	29,90	3,00	2,34
	2000	6,63	0,40	297		20,98	24,85	2,92	3,15
	2150	6,54	0,43	284		24,61	26,62	2,82	2,16

The complexity of whiskers compaction is due to the extremely developed morphology of whiskers – on the one hand (see Fig. 1) and extremely high strength of whiskers – on the other (for instance, the strength of Si_3N_4 whiskers is 11000 MPa, which is significantly higher than the strength of Si_3N_4 in bulk form (344 MPa)) [12].

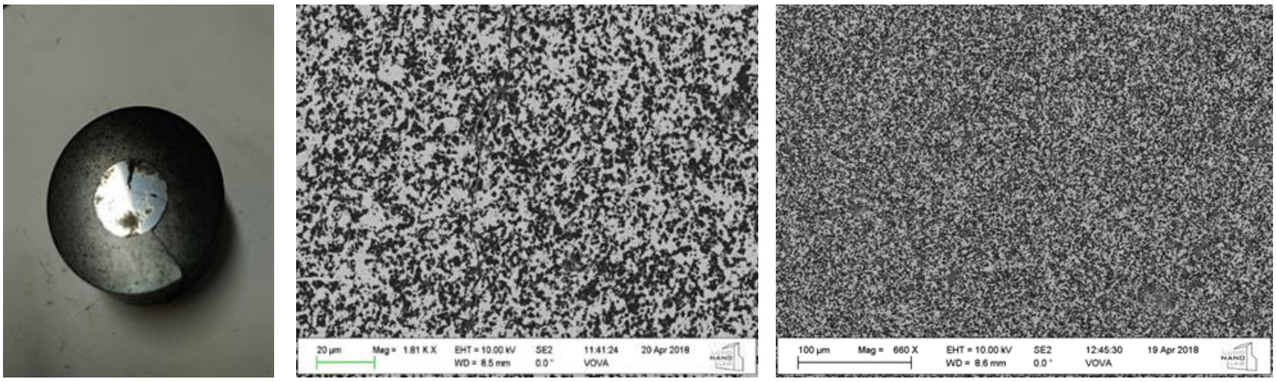


a *b*

Fig. 4. The dependence of the Young's modulus of cBN-based sintered materials, reinforced 0; 5; 10 and 15 vol. %, (4, 3, 2, and 1, respectively) whiskers of Si_3N_4 (a) and $Mg_2B_2O_5$ (b) versus sintering temperature

Young's modulus was determined using an Olympus 38DL Plus ultrasonic thickness gauge using. Young's modulus decreases with the introduction of both whiskers types, which is associated with both the low density of whiskers and the increase in the effect of the structure of the material due to the developed morphology of whiskers. In addition, it is known that the defectiveness of the material increases with increasing sintering temperature due to a significant difference between the coefficients of thermal expansion of the whiskers and the matrix (CTE of Si_3N_4 = 3,0–3,9·10^{-6}/K, CTE of cBN = 3,7·10^{-6} /K [13] CTE of TaN = 4,9·10^{-6} /C CTE of Al = 22,2–23,1·10^{-6}/K CTE of $Mg_2B_2O_5$ whiskers = 18·10^{-6}/K and 2·10^{-6}/K along axial and radial directions, respectively [14]), which leads to the emergence of microstresses and could also cause a drop in Young’s modulus when whiskers are introduced).

The Young's modulus of a sample that was not reinforced with whiskers and sintered at the maximum sintering temperature (2150 °C) was not measured, as this sample was broken. The surface of the broken sample contained cracks over the entire surface (Fig. 5, *a*).



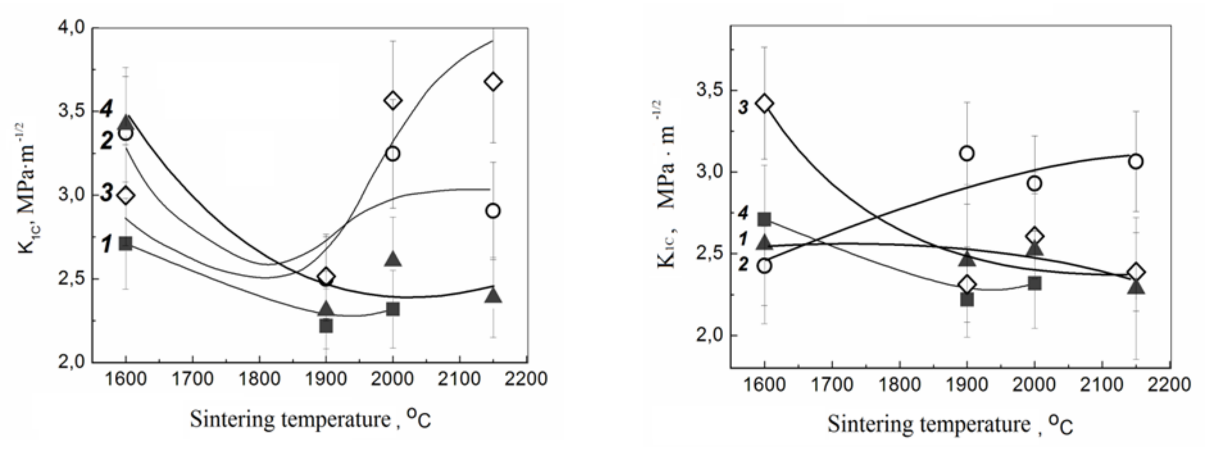
a *b* *c*

Fig. 5. Appearance of a broken sample, that was not reinforced with whiskers (a), sintered at 2150 °C; microstructure of samples, unreinforced (b) and reinforced (c) by 5 vol. % of Si_3N_4whiskers, sintered at 1600 and 2150 °C, respectively

On the microstructure of samples sintered without the addition of whiskers, cracks were observed over the entire surface even at the minimum sintering temperature (Fig. 5, *b*), while on the surface of the samples reinforced with Si_3N_4 whiskers, such cracks were not observed even at the maximum sintering temperature (Fig. 5, *c*), which indicates the feasibility of reinforcement.

The results of measurement of hardness and crack resistance. Relationship with structure and phase composition

Microhardness was measured using the Vickers indentation pyramid with a 9,8 N load. It increased both with an increase in sintering temperature and with the introduction of Si_3N_4 whiskers (*a*) and $Mg_2B_2O_5$ whiskers (*b*) into the composition of the base material (Table). The fracture toughness of materials was measured at indentation loads of 24,5 and 49 N. Material indentation at different loads allows to evaluate the behavior of the material depending on the loading conditions and the dynamics of behavior in real conditions [15]. As can be seen from the graphs below (Fig. 6, Fig. 7), the fracture toughness most of all specimens increased with increasing indentation load. According to [16], this unusual behavior of materials can be associated with the stress-strain state of the samples, as well as with the brittle destruction of microvolumes, which is characteristic of ceramic materials obtained by the method of HPHT sintering.



a *b*

Fig. 6. The dependence of the fracture toughness of cBN-based materials, measured at 24.5 N indentation load, that reinforced with 0; 5; 10 and 15 vol. %, (1; 2; 3 and 4, respectively) Si_3N_4 (a) and $Mg_2B_2O_5$ (b) whiskers versus sintering temperature

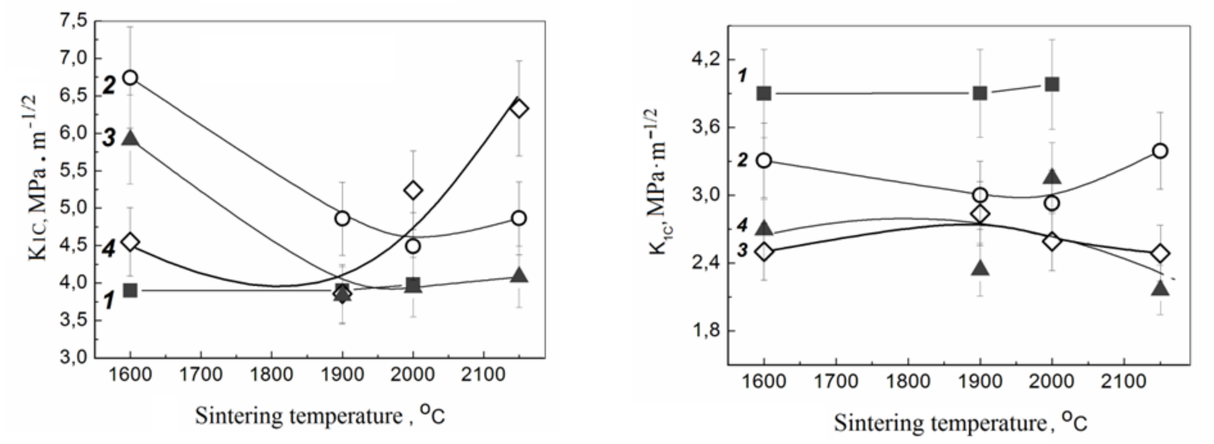


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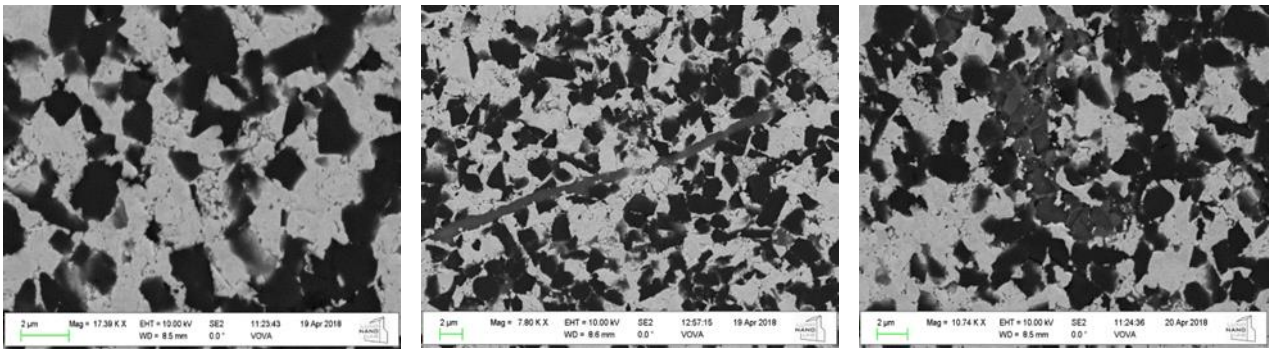
Fig. 7. The dependence of the fracture toughness of cBN-based materials, measured at 49,0 N indentation load, that reinforced with 0; 5; 10 and 15 vol. %, (1; 2; 3, and 4, respectively) Si_3N_4 (a) and $Mg_2B_2O_5$ (b) whiskers versus sintering temperature

However, from the point of view of determining the "fracture toughness " as resistance to the initiation of cracks, the results of measuring the fracture toughness under less load (24,5 N) are more correct.

In most cases, the fracture toughness of specimens reinforced with $Mg_2B_2O_5$ whiskers decreases with increasing sintering temperature, and for specimens reinforced with Si_3N_4 whiskers it increases with a load of 24,5 N and is almost independent of the sintering temperature with a higher load (49 N) (Fig.7, *b*). First of all, this may be due to the decomposition of $Mg_2B_2O_5$ whiskers with an increase in sintering temperature, which is confirmed by the results of X-ray phase analysis decoding, as well as by studying the microstructure of the materials obtained (Fig. 8).



a *b* *c*

Fig. 8. Microstructure of samples, unreinforced (a) and reinforced by 5 vol. % of Si_3N_4 (b) and $Mg_2B_2O_5$ (c) whiskers, sintered 2150 °C

On the other hand, it is known from literature [17] that a brittle oxide layer on the surface of whiskers weakens their connection with the matrix, which leads to the implementation of a mechanism for whiskers pullout and increases energy costs when the material is distributed in the matrix due to friction forces at the whisker-matrix interface. Analysis of the microstructures of the samples with the addition of Si_3N_4 whiskers showed a slight interaction of the Si_3N_4 whiskers with the matrix material at a sintering temperature of 2150 °C (Fig. 8, *b*). As for the samples reinforced with $Mg_2B_2O_5$ whiskers, that sintered at temperature of 2150 C (Fig. 8, *c*). It is already observed that the whiskers of $Mg_2B_2O_5$ recrystallized, which leads to a loss of reinforcement efficiency, a sharp decreasing of fracture toughness, measured on the 49 N load.

The results of X-ray diffraction analysis showed the presence of tantalum boride (TaB), indicating the reaction of the matrix material (cBN) and binder (TaN). (Fig. 9).

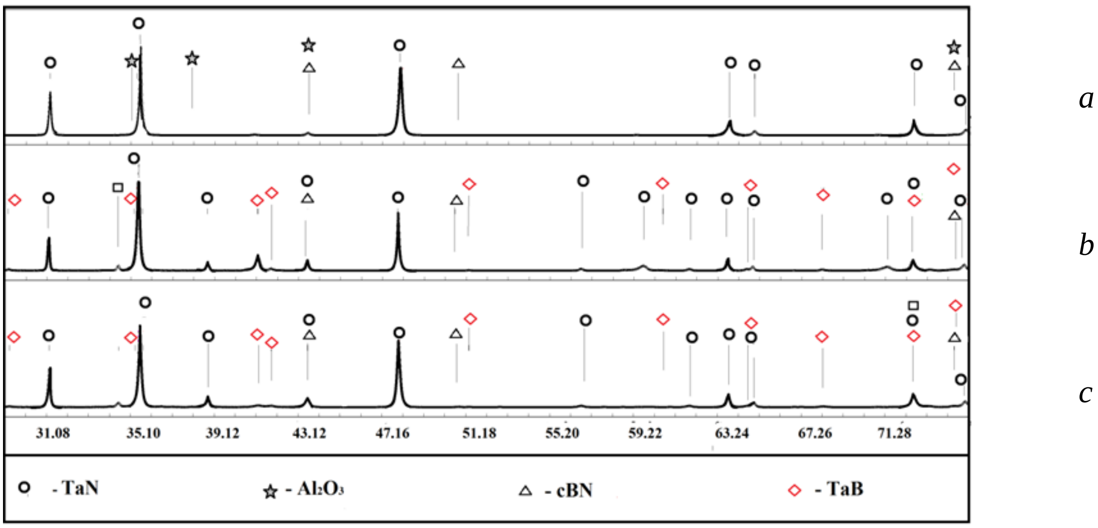
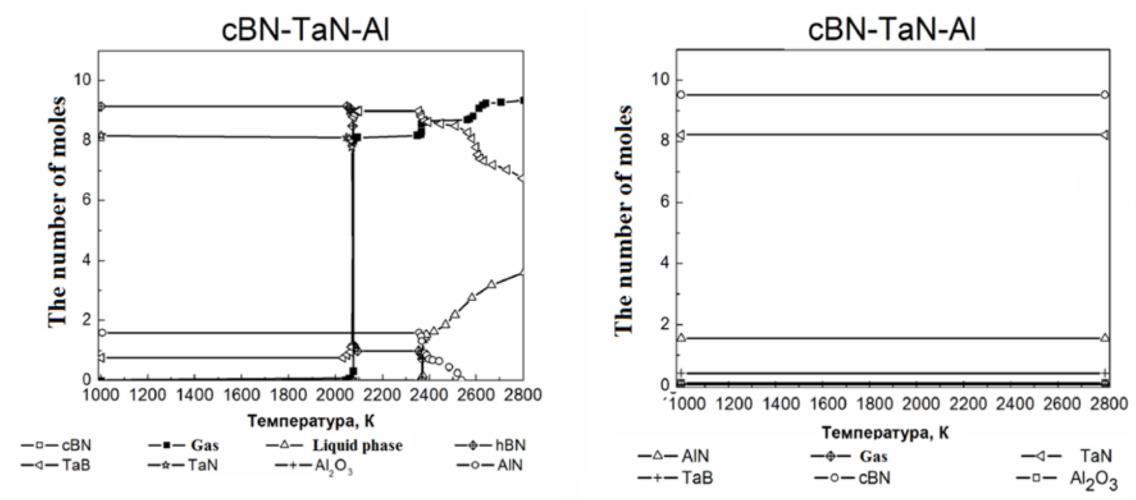


Fig. 9. Results of decoding X-ray phase analysis of samples sintered at 1800 ° C of various composition (a – 50% cBN-45% TaN – 5% Al; b – 50% cBN-45% TaN-5% Al - 10% Si_3N_4; c – 50% cBN-45% TaN-5% Al-10% $Mg_2B_2O_5$)

Thermodynamic calculations in the ThermoCalc program also confirmed the formation of tantalum boride in this system (Fig. 10). From the graphs below, it can be seen that an increase in pressure leads to a decrease in the number of phases that can form in the system – on the one hand, and on the other, the formed phases stabilize over the entire calculated temperature range.



a *b*

Fig. 10. Results of thermodynamic calculations in the HermoCalc program of reaction products formed in the cBN-TaN-Al at atmospheric pressure (a) and under. the action of pressure 7,7 GPA (b)

Conclusions

In this work, in order to increase the fracture toughness, we carried out reinforcement of the cutting material based on cubic boron nitride consisting of 50% cBN, 5% Al and TaN as a binder and whiskers of Si_3N_4-and $Mg_2B_2O_5$ in the amount of 5, 10, 15 vol. %

Samples were obtained with the help of HPHT sintering in the «toroid-type» apparatus. The study of the influence of sintering temperature, volume content, and the type of whiskers on the properties of the samples obtained showed that the properties of the materials obtained essentially depend on the type of whiskers introduced, their morphology and chemical composition:

1. The results of X-ray phase analysis showed that an increase in sintering temperature leads to the formation of tantalum boride, which indicates the passage of a chemical reaction between the matrix (cBN) and the binder (TaN) to form tantalum monoboride (TaB) and is confirmed by the results of thermodynamic calculations in the ThermoCalc program.

2. Compaction of samples reinforced with $Mg_2B_2O_5$ whiskers is easier than compaction of specimens reinforced of Si_3N_4whiskers, despite the more developed morphology of $Mg_2B_2O_5$ whiskers, which is connected.

3. Young's modulus decreases with the introduction of both types of whiskers, which is associated with both the low density of whiskers and the increase in the effect of the structure of the material due to the developed morphology of whiskers. In addition, it is known that the defectiveness of the material increases with increasing sintering temperature due to the difference between the coefficients of thermal expansion of the whiskers and the matrix, which leads to the emergence of microstresses and could also cause a drop in Young's modulus when microfibers are introduced into the base material.

4. The recrystallization of $Mg_2B_2O_5$whiskers takes place already at a sintering temperature of 1800 °C, while a slight chemical interaction of Si_3N_4whiskers with the matrix begins to occur only at 2150 °C.

5. Hardness increases with the introduction of any of the types of whiskers in the composition of the base material – 20–25 GPa for non-reinforced composites to 25–32 for reinforced microfibers.

6. Fracture toughness of the reinforced material with Si_3N_4 whiskersis higher than the fracture toughness of the non-reinforced material with both indentation loads (24, 5 and 49 N), which should lead to an increase in the service life of the cutting tool with the addition of Si_3N_4 whiskers compared to the non-reinforced material.

7. Analysis of the fracture toughness of materials at different indentation loads showed that reinforcement with Si_3N_4 whiskers is more efficient than reinforcement with $Mg_2B_2O_5$ whiskers. The study of the microstructure of the obtained materials showed that the recrystallization of $Mg_2B_2O_5$ whiskers occurs already at a temperature of 1900 °C. This explains the drop in the fracture toughness values when the temperature rises when the indentation load is 24,5 Н, and the fact that at the 49,0 N indentation fracture toughness of the reinforced material is lower than the fracture toughness the non-reinforced (K_{1C} of non-reinforced composites at 49,0 N =2,75–4,08 MPa·m$^{-1/2}$, K_{1C} of composites reinforced with $Mg_2B_2O_5$ whiskers at 49,0 N =2,16–3,85 MPa·m$^{-1/2}$). All this indicates the effectiveness of material reinforcement with Si_3N_4 whiskers and the inefficiency of material reinforcement with $Mg_2B_2O_5$ whiskers. The study of cutting ability of sintered samples is needed.

Методом высокотемпературного спекания под высоким давлением (HPHT спекание) было получено три вида композитов на основе cBN (без микроволокон, армированные микроволокнами Si_3N_4 и армированные микроволокнами $Mg_2B_2O_5$). Для всех образцов были измерены плотность, модуль Юнга, коэффициент Пуассона и трещиностойкость. Композиты на основе cBN, армированные микроволокнами Si_3N_4, обладали лучшими механическими свойствами (твердость, трещиностойкость), чем неармированные композиты. Армирование микроволокнами $Mg_2B_2O_5$ оказалось неэффективным вследствие их низкой термохимической стабильности.
***Ключевые слова**: микроволокна, армирование, cBN, трещиностойкость*

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ВПЛИВ АРМУВАННЯ МІКРОВОЛОКНАМИ Si_3N_4 ТА $Mg_2B_2O_5$ КОМПОЗИТІВ НА ОСНОВІ cBN НА ЇХНІ ВЛАСТИВОСТІ

За допомогою методу високотемпературного спікання під високим тиском (HPHT синтезу) було отримано три види композитів на основі cBN (без мікроволокон, армовані мікроволокнами Si_3N_4 і армовані мікроволокнами $Mg_2B_2O_5$). Для усіх композитів були визначені модуль Юнга, коефіцієнт Пуасона та тріщиностійкість. Композити на основі cBN, армовані мікроволокнами Si_3N_4, мали кращі механічні характеристики (твердість, тріщиностійкість), ніж неармовані. Армування мікроволокнами $Mg_2B_2O_5$ виявилось неефективним внаслідок їх низької термохімічної стабільності.
***Ключові слова**: мікроволокна, армування, cBN, тріщиностійкість*

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ОСОБЛИВОСТІ ФОРМУВАННЯ СТРУКТУРИ КОМПОЗИТІВ СИСТЕМИ cBN–TiC–Al

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В роботі представлено результати дослідження структури і властивостей PCBN композитів, отриманих при реакційному спіканні в умовах високого тиску і температури порошків системи cBN-TiC- Al. Використовували апарати високого тиску типу «ковадло з заглибленням». Методом рентгеноструктурного аналізу в композиті ідентифіковано фази cBN, AlN, TiC і твердий розчин $Ti_xAl_{1-x}B_2$. Не було ідентифіковано як окремі фази тверді розчини азоту і кисню в кристалічній ґратці TiC, а також вуглець (графіт). Утворення твердого розчину диборидів титану і алюмінію свідчить про реакційну взаємодію на міжфазних контактах TiC-cBN і TiC-Al, а також про вплив TiC на гальмування структурних перетворень від дибориду до вищих боридів алюмінію при зміні термобаричних параметрів спікання. Відмічено, що під час спільного спікання порошків cBN і TiC твердофазна взаємодія між ними реалізується шляхом дифузії легких елементів (кисню, азоту, вуглецю). Це приводить до створення твердих розчинів на базі кристалічних ґраток карбіду титану Ti(C,O) і сфалеритного нітриду бору B(N,O), при цьому в умовах гетеровалентного заміщення утворюються структурні вакансії в підґратках титану і бору.

Ключові слова*: кубічний нітрид бору, карбід титану, надтвердий композит, кристалічна структура, тверді розчини, фазовий склад, високий тиск, рентгеноструктурний аналіз, твердість.*

Вступ

Полікристалічні надтверді матеріали на основі кубічного нітриду бору (PCBN) широко відомі у світі як інструментальні матеріали для оснащення лезового інструменту, ефективного при обробленні загартованих сталей, чавунів, спеціальних сплавів, інших важкооброблюваних