

Получение, структура, свойства

UDC 661.657.5:621.9.025.1

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Composites of the cBN–Si₃N₄ system reinforced by SiCw for turning tools

The cBN–Si₃N₄–SiCw composites with different SiCw contents up to 20 vol % have been produced at high pressure of 8.0 GPa and high temperature of 2500 K. It has been defined that the Young modulus of the composites were within 740–846 GPa, the Vickers hardness and fracture toughness values were 37.5–42.0 GPa and 11.4–12.9 MPa·m^{1/2}, respectively. An important feature of the composite microstructure is the breaking of SiCw as a result of HPHT action. It has been shown that at the addition of 10 vol % SiCw to the structure of a cBN–Si₃N₄ composite the interrupted turning of hardened steel results in the flank wear reduction up to 20%.

Keywords: cBN, SiC whiskers, composite, HPHT-sintering, interrupted turning.

INTRODUCTION

The machining of the very hard steels and cemented carbides are inseparably linked with the development of new technologies for cutting composite materials, which must possess high stress-strain characteristics. Considerable impact loads arise in cutting edge of the tools at interrupted turning of the steel parts with the hardness above HRC 60. It leads to a significant increase of the tool flank wear and decrease of its efficient life. One of the means of the improvement of such tools is the development of cutting composite materials reinforced by SiC whiskers (SiCw). The reason for the use of SiCw into the composite materials structures is the research of tensile mechanical whiskers properties [1]. SiC whiskers 5 mm in length have been shown to possess an average tensile strength of 8.40 GPa and an average elastic modulus of 581 GPa. This indicates their significant potential as reinforcement elements in the development of high-strength ceramic composites for cutting tools. Ever since in the world there are active studies with the aim to improve the sintered ceramics cutting properties. In [2] thermome-

chanical properties of a SiC whiskers/ Al_2O_3 matrix composite were investigated as a function of the whisker surface quality. The paper [3] presented the results of the investigation of surface damage produced by whisker-reinforced ceramic cutting tools in the finish turning of Inconel 718 alloy. Whisker-reinforced aluminum oxide inserts are used successfully in the machining of various alloys. The microstructure and mechanical properties of Al_2O_3 containing 25 wt % SiCw were examined for cutting tools applications in [4]. It was shown that ceramics with participation of SiCw in Al_2O_3 matrix enhanced the mechanical properties significantly, which enabled the cutting of Inconel 718 super-alloy. The Al_2O_3 ceramics reinforced by SiCw was developed with the objective to improve its fracture toughness and crack healing ability [5]. The used SiC whiskers have diameters between 0.8 and 1.0 μm and lengths from 30 to 100 μm . The SiC whiskers were located at grain boundaries between Al_2O_3 grains and were preferentially oriented within the plane perpendicular to the pressing axis. In other studies mechanical properties of $\text{Al}_2\text{O}_3/\text{Ti}_3\text{SiC}_2$ multilayer ceramics were improved by adding SiCw into Al_2O_3 layers [6]. The optimal mechanical properties were obtained when the content of SiC whiskers was 20 wt %. The distribution, morphology, and reinforcing behaviors of the SiCw in the reaction bonded SiC-composite were presented in [7]. It was shown that whisker pullout, whisker bridging, and crack deflection are main toughening mechanisms. Paper [8] confirmed that introduction of SiC whiskers improved the densification and mechanical properties of the SiC-based ceramics. Thermal shock behavior of ultrahigh-temperature SiC-whisker-reinforced boride ceramics was studied in [9]. Ultrahigh-temperature SiCw-reinforced ZrB_2 ceramics was fabricated by the hot pressure sintering. The flexural strength of these composites attains 753 MPa. The ZrB_2 matrix with SiCw possesses a good thermal shock resistance. No cracks or other forms of damages in the specimens were induced during the quenching.

A hetero-modulus composite material containing* 90–97 % cBN and 3–10 % Si_3N_4 is presented in [10]. This material shows a high performance at interrupted turning of the very hard steels.

This study has been performed in order to carry out HPHT-sintering of composites of the cBN– Si_3N_4 –SiCw system with different contents of SiCw. In the present paper physical and mechanical properties of cBN– Si_3N_4 –SiCw composites, microstructure peculiarities, and the testing at interrupted turning of very hard steel are presented. The final purpose of this research was to determine possibilities to improve the strength properties of cBN– Si_3N_4 composite material by reinforcing it with SiC-whiskers.

MATERIALS AND METHODS

Compacts based on cBN were created under HPHT conditions using micron powder of cBN with an average grain size of 3–6 μm produced by the Element Six company, micron powder of Si_3N_4 with an average grain size of 0.5–0.7 μm (produced by the Inc. Starck, Germany), SiC whiskers with an average diameter size of 1.5 μm and with an average length size of 18.0 μm (produced by the Inc. Starck, Germany). Powders of cBN, Si_3N_4 , and SiCw were mixed in isopropyl alcohol using Si_3N_4 ball mill of the Pulverisette 6 Model, (Fritsch) for 2 h. As a result five mixtures were prepared. Then powder mixtures were dried at room temperature for 12 h. First mixture consisted of 97 % cBN and 3 % Si_3N_4 . The remaining four mixtures contained 5, 10, 15, and 20 % of SiCw: 92 % cBN, 3 % Si_3N_4 , and 5 % SiCw; 87 % cBN, 3 % Si_3N_4 , and 10 % SiCw; 82 % cBN, 3 % Si_3N_4 , and 15 % SiCw; 77 % cBN,

*From now on the compositions of the mixtures and materials are given in vol %.

3 % Si_3N_4 , and 20 % SiCw. The SEM images of the initial SiC whiskers and micron powders mixture of 87 % cBN, 3 % Si_3N_4 , and 10 % SiCw are presented in Fig. 1.

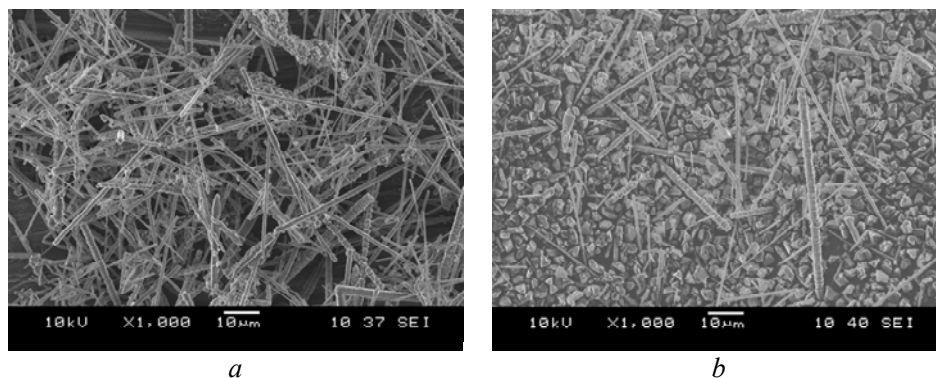


Fig. 1. SEM-images of the initial SiC whiskers (a) and micropowders mixture of 87 % cBN, 3 % Si_3N_4 , 10 % SiCw (b).

All patterns were taken of the composites HPHT-sintered using a toroid-type high-pressure apparatus with 30 mm diameter of the central cavity and 2.55 cm^3 working volume of a high pressure cell [11]. Experimental mixtures were placed in toroidal gaskets and compressed between two cemented tungsten carbide anvils, as shown in Fig. 2. Value of the pressure into the high pressure cell was determined using a well-known method to fix phase transition of bismuth, thallium, and lead selenide. The sintering temperature was measured by thermocouples and refined according to the melting temperatures of silicon and copper [12]. Samples of composite materials were produced in the thermodynamic stability region of cubic boron nitride modification at high pressure of 8.0 GPa and high temperature of 2500 K. Duration of the sintering was 1.0 min. HPHT-sintered samples were prepared in a disk form with diameter of 9.5 mm and thickness of 4.0 mm after grinding and polishing.

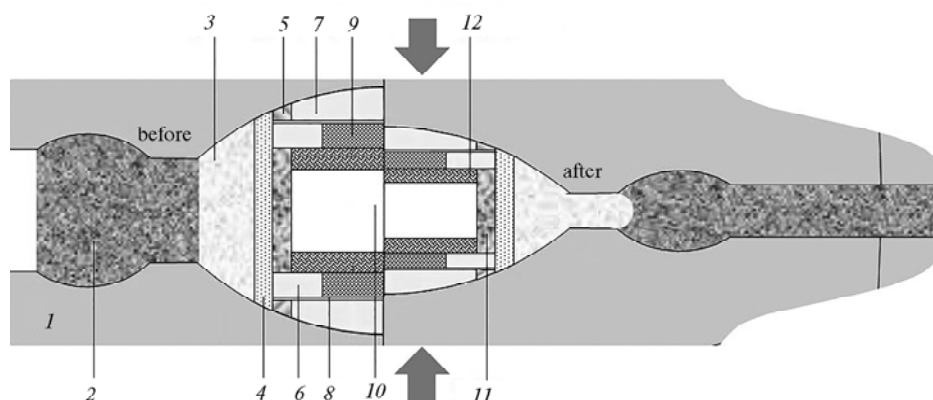


Fig. 2. Assembling of the toroid-type HP apparatus for sintering of composites before and after actions of axial compression up to 18 MN: 1 – cemented tungsten carbide anvil; 2 – toroidal ring of lithographic limestone; 3 – container of block lithographic limestone; 4 – heat-insulating ring; 5 – steel ring; 6 – heat-insulating ring of pyrophyllite; 7 – butt part of pyrophyllite; 8 – a molybdenum disk; 9 – disk of graphite- ZrO_2 ; 10 – micropowders mixture of cBN- Si_3N_4 -SiCw system for HPHT sintering of composites; 11 – a graphite heater; 12 – a graphite disk.

The elasticity moduli of the samples were measured with an accuracy of 2 % by an ultrasonic method measuring for the propagation velocity of transverse and longitudinal waves transmitted through the samples using a Panametrics Epoch III ultrasonic flaw detector [13]. The density was determined by hydrostatic weighing with an accuracy of $\pm 0.01 \text{ g/cm}^3$. Vickers hardness was measured at a load of 9.81 N using a Hardness Tester Future-Tech FV-700. The fracture toughness was calculated from the length of the cracks developed after the Vickers indentation test using the equation from [14]. The measurement errors for Vickers hardness and fracture toughness were $\pm 1.3 \text{ GPa}$ and $\pm 0.7 \text{ MPa}\cdot\text{m}^{1/2}$, respectively. Microstructure investigations of the samples were made using a JEOL JSM-6460LV scanning electron microscope with an EDS spectrometer. The phase compositions of the sintered compacts were identified by X-ray diffraction based on the ICDD and ICSD databases.

The interrupted turning tests of produced composites were carried out using a standard screw-cutting lathe [15]. The work-in-process of high carbon steel shows the best correlation with 107WCR5 (EN) steel containing up to 1.2 at % Cr, 1.6 at % W, 1.1 at % Mn and hardened to HRC 62 (conventional deviation HRC is 1.5). The diameter of the workpiece rod was 100 mm. The workpiece has six key holes with width of 10 mm, as shown in Fig. 3. Machining process parameters are listed in Table 1. Such machining conditions are characterized by high thermal and mechanical loads in cutting edge of the composite tool. All tested samples were round cutting blanks of the RNMN 09T300T type with diameters of 9.52 mm and 3.97 mm thick. After the interrupted turning tests, the flank wear of cutting blank was measured with an accuracy of $\pm 0.01 \text{ mm}$ using an optical microscope with a cross-hair micrometer.



Fig. 3. Hardened steel rod with key holes for interrupted turning tests. The arrow indicates one of the key holes.

Table 1. Parameters of the machining process

Cutting speed v_c , m/min	100
Depth of cut a_p , mm	0.2
Feed rate f , mm/rev	0.1, 0.2, 0.4
Turning time T , min	10, 5.3, 2.6
Chamber land width b , mm	0.2
Angle of chamber λ , deg	20
Rake angle β , deg	-10
Relief angle α , deg	10

RESULTS AND DISCUSSION

The results of our investigation of physical and mechanical properties of HPHT-sintered composites are given in Table 2. All composites produced have high degree of compaction and low porosity (Fig. 4). The value of closed porosity is 1 %. As a consequence of increasing the SiCw content of the composite from 0 to 20 %, the composite density decreases from 3.46 to 3.41 g/cm³. The change of the SiCw concentrations in starting mixtures does not influence the consolidation of the cBN–Si₃N₄–SiCw system at the used HPHT conditions. The maximum velocity of the longitudinal ultrasonic wave of 15500 m/s was fixed at the measurement of the Young modulus of the sample comprising of 97 % cBN and 3 % Si₃N₄. It is the evidence of the low ultrasonic wave dissipation because of grains boundaries presence in the composite. The highest Young's modulus ensures the bonding between cBN grains. Addition of SiCw component into cBN–Si₃N₄ system leads to a reduction of the Young modulus. This characteristic parameter equals 846 GPa for a composition of 97cBN–3Si₃N₄ and 740 GPa for the composition of 77cBN–3Si₃N₄–20SiCw. On the other hand, the Vickers hardness of a sintered composite decreases from 42.0 to 37.5 GPa with increasing the SiCw component to 20 %. This interval of the Vickers hardness values for composites based on cBN produced at HPHT conditions is conclusive prerequisite to their application in cutting tools for turning of very hard steels [16]. The fracture toughness hasn't appreciable correlation with a change of the SiCw concentration up to 20 %. The average value of the fracture toughness is about 12.0 MPa·m^{1/2}. A high value of the fracture toughness combined with a high value of Young's modulus and Vickers hardness gives ground to suppose that the produced composites can show good results in cutting hard-to-machine materials, especially in the interrupted turning when the cutting tool edge is subjected to periodic impacts.

Table 2. Physical and mechanical properties of sintered composites

Composition, vol %			Density, g/cm ³	Relative density, %	Young's modulus, GPa	Vickers hardness, GPa	Fracture toughness, MPa·m ^{1/2}
cBN	Si ₃ N ₄	SiCw					
97	3	0	3.46±0.01	99.2	846±16	42.0±1.1	12.9±0.4
92	3	5	3.44±0.01	98.9	820±17	41.1±1.2	12.5±0.4
87	3	10	3.43±0.01	99.0	790±15	41.0±1.3	11.4±0.5
82	3	15	3.42±0.01	99.1	763±18	39.0±1.1	11.9±0.7
77	3	20	3.41±0.01	99.2	740±15	37.6±1.3	12.2±0.6

Figure 5 shows SEM images of the cBN–Si₃N₄–SiCw composites microstructures. As can be seen, SiC whiskers are homogeneously distributed in the cBN–Si₃N₄ composite. An important feature of the composite microstructure is the fact that the SiCw breaking as a result of the high-pressure action. Dark grains in Fig. 5 represent cBN, Si₃N₄ and SiCw appear at the grain boundaries. Description of HPHT sintering of the cBN–Si₃N₄ system was presented in [10]. The X-ray diffraction analysis doesn't determine the interaction between components of the cBN–Si₃N₄–SiCw composite during the HPHT sintering.

On the interrupted turning tests of the composites produced, it was defined that all cutting inserts show high performance at the hardened steel machining (Fig. 6). The results of the interrupted turning tests are presented in Fig 7. The composite inserts of 87cBN–3Si₃N₄–10SiCw have the lowest speed of wear and flank wear.

Cutting properties of the $97\text{cBN}-3\text{Si}_3\text{N}_4$ and $92\text{cBN}-3\text{Si}_3\text{N}_4-5\text{SiCw}$ composites are very similar. In all cases the composite inserts exhibited a classic wear land without chipping and spalling. So, it was established that the addition of the SiC whiskers into the $\text{cBN}-\text{Si}_3\text{N}_4$ system led to an increase of the strength property.

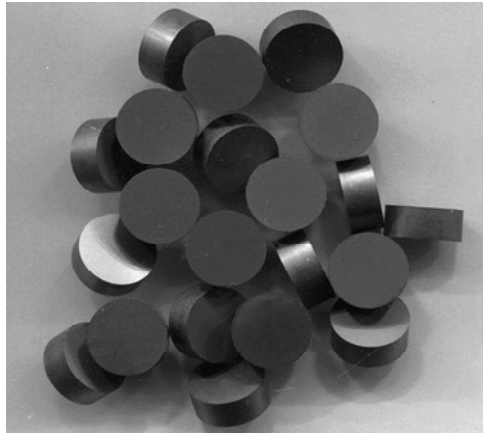


Fig. 4. Samples of a $\text{cBN}-\text{Si}_3\text{N}_4-\text{SiCw}$ composite after grinding and polishing.

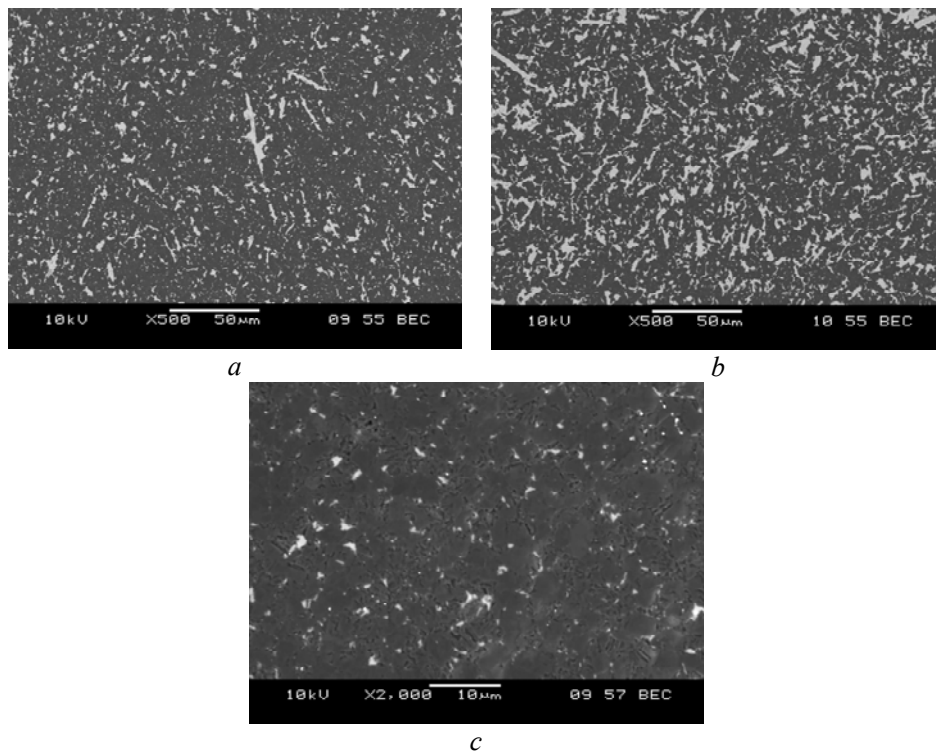


Fig. 5. Microstructures of materials having different compositions: $87\text{cBN}-3\text{Si}_3\text{N}_4-10\text{SiCw}$ (a); $77\text{cBN}-3\text{Si}_3\text{N}_4-20\text{SiCw}$ (b) (SiCw is white spots in the SEM image); $97\text{cBN}-3\text{Si}_3\text{N}_4$ (c).



Fig. 6. Interrupted turning tests of the composites produced.

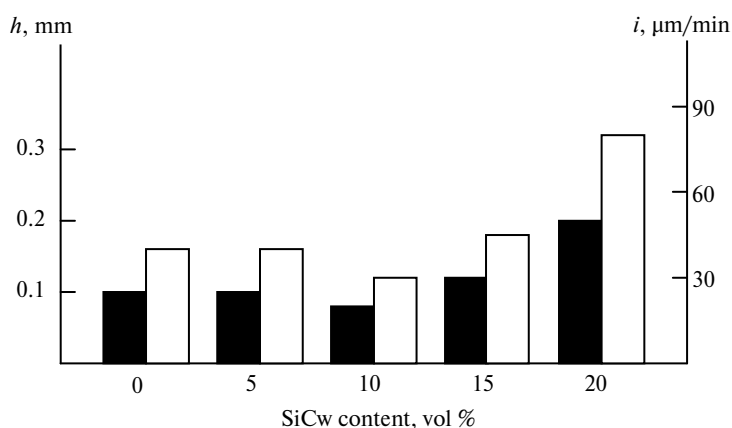


Fig. 7. Effect of the SiCw content of a composite on the flank wear (h , mm) is indicated by black bars, wear speed (i , μm) by white bars.

CONCLUSIONS

Superhard composite materials of the cBN-Si₃N₄-SiCw system have been produced at high pressure of 8.0 GPa and high temperature of 2500 K. The Vickers hardness has been measured in the 37.5–42.0 GPa range. Young's modulus and fracture toughness are in the range of 740–846 GPa and 11.4–12.9 MPa·m^{1/2}.

The interrupted turning tests show that the 10 % SiC whiskers, which has been added into the structure of the cBN-Si₃N₄ composite, leads to the reduction of the flank wear by 20 %. Therefore, there is reason to suggest that the participation of 10±2 % SiCw in a cBN-Si₃N₄ composite improves the composite cutting properties.

Although the addition of SiC whiskers into the composition of the produced cBN material allows a rather low increase of the impact resistance as compared with Al₂O₃-SiCw composites, our research shows that SiC whiskers as an reinforce-

ing component should be applied to HPHT sintered composite materials based on cBN.

The whisker-reinforced composite consisting of 87 % cBN, 3 % Si₃N₄, 10 % SiCw confirmed good results in the interrupted turning of hardened steel and can be recommended to use in cutting tools.

This work was supported by the European Commission under the FP7 Specific Programme 'Capacities' within the framework of the SINTERCER project Development of a sintering centre and know-how exchange for non-equilibrium sintering methods of advanced ceramic composite materials (REGPOT-2012-2013-1 EU FP7 Research Potential, project no. 316232).

В умовах високого тиску 8,0 ГПа і високої температури 2500 К було отримано композити cBN-Si₃N₄-SiCw з різним вмістом компоненти SiCw до 20 % (за об'ємом). Модуль Юнга знаходиться в інтервалі 740–846 ГПа. Твердість за Вікерсом і тріщиностійкість відповідно мають значення в інтервалі 37,5–42,0 ГПа і 11,4–12,9 МПа·м^{1/2}. Характерною особливістю мікроструктури композиту є наявність зруйнованих SiC-вусів, як результат впливу високого тиску. Розроблено методику тестування зразків при переривчастому точінні. Випробування зразків при переривчастому точінні високотвердих сталей показують, що добавка SiC-вусів в кількості 10 % (за об'ємом) у структурі композиту cBN-Si₃N₄-SiCw приводить до зниження зносу ріжучої кромки до 20 %.

Ключові слова: cBN, SiC-вуси, композит, спікання в умовах високих тисків і температур, переривчасте точіння.

В условиях высокого давления 8,0 ГПа и высокой температуры 2500 К были получены композиты cBN-Si₃N₄-SiCw с различным содержанием компоненты SiCw до 20 % (по объему). Модуль Юнга находится в интервале 740–846 ГПа. Твердость по Викерсу и трещиностойкость соответственно имеют значения в интервале 37,5–42,0 ГПа и 11,4–12,9 МПа·м^{1/2}. Характерной особенностью микроструктуры композита является наличие разрушенных SiC-усов, как результат воздействия высокого давления. Разработана методика тестирования образцов при прерывистом точении. Испытания образцов при прерывистом точении високотвердых сталей показывают, что добавка SiC-усов в количестве 10 % (по объему) в структуре композита cBN-Si₃N₄-SiCw приводит к снижению износа режущей кромки до 20 %.

Ключевые слова: cBN, SiC-усы, композит, спекание в условиях высоких давлений и температур, прерывистое точение.

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Received 28.07.15