

Cubic boron nitride based composites for cutting applications

P. Klimczyk^{a,*}, P. Figiel^a, I. Petrusha^b, A. Olszyna^c

^a The Institute of Advanced Manufacturing Technology,
ul. Wrocławska 37a, 30-011 Kraków, Poland

^b V. Bakul Institute for Superhard Materials NASU,
Avtozavodskaya 2 St, 04074 Kiev, Ukraine

^c Faculty of Materials Science and Engineering, Warsaw University of Technology,
ul. Wołoska 141, 02-507 Warszawa, Poland

* Corresponding author: E-mail address: piotr.klimczyk@ios.krakow.pl

Received 06.11.2010; published in revised form 01.02.2011

Manufacturing and processing

ABSTRACT

Purpose: The aim of our work was to obtain durable fined-grained cBN-Si₃N₄ composite with high values of hardness and fracture toughness, which can be successfully used as a cutting tool. Little quantity of the Si₃N₄ nanopowder fills up the porous between cBN grains thus result in resistance to crack propagation by means so called “crack deflection” mechanism.

Design/methodology/approach: Two variants of the cBN-Si₃N₄ composites („I” – with micropowder cBN and „II” – with mixture of micro- and nanopowdes cBN) contained 3% of nanodispersed Si₃N₄ powder, have been sintered at High Pressure – High Temperature (HPHT) conditions. Basic physical-mechanical properties, phase composition and microstructure of sintered materials have been investigated.

Findings: The comparison of the mechanical properties of cBN-Si₃N₄ (I) and cBN-Si₃N₄ (II) composites showed that the addition of 10% cBN nanopowder to mixture caused small increase in hardness from 4750 up to 4855 HV₁₀ and decrease in Young’s modulus from 842 to 812 GPa. Fracture toughness of both type of composites is on the same level above 10 MPa·m^{1/2}.

Research limitations/implications: High hardness of cBN-Si₃N₄ composites present a technical challenge in shaping of them. Commercial application of presented materials, e.g. cutting tools production, needs to develop a high efficient cutting, lapping and grinding techniques.

Practical implications: The material obtained could be successfully applied for different cutting applications due to its favourable combination of hardness and fracture toughness.

Originality/value: Commercial superhard materials, so called “high content cBN composites”, have usually 10-20 vol% of binding phase, often in the form of Ti or/and Al compounds. In the presented work only 3% of Si₃N₄ phase was used as a sintering aid. The high content of cBN phase allows to keep Young’s modulus and hardness values close to the theoretical ones for pure PcBN.

Keywords: Tool materials; cBN composites; HPHT

Reference to this paper should be given in the following way:

P. Klimczyk, P. Figiel, I. Petrusha, A. Olszyna, Cubic boron nitride based composites for cutting applications, Journal of Achievements in Materials and Manufacturing Engineering 44/2 (2011) 198-204.

1. Introduction

The boron nitride in the cubic form (cBN) is the second hardest material next to the diamond and possess many excellent physical, chemical and mechanical properties such as a thermal stability up to 1200°C, high resistance to chemical attack, high hardness (~6000 HV) and Young modulus (~900 GPa) values [1, 2]. Cutting tools made from materials based on polycrystalline cubic boron nitride (PcBN) are widely used for machining of hardened steels, cast iron and others due to its unique combination of properties [3]. Especially high hardness, low friction, high corrosion resistance and low chemical affinity with iron-group metals are most important for cutting tools [4, 5]. Depending on the machining parameters, the lifetime of cubic boron nitride tools can be many times longer in comparison with ceramic or tungsten carbide tools. Moreover, PcBN based tools give a possibility of using turning instead of grinding for the finish machining of hardened steel and the possibility of turning without coolants.

At normal pressure cBN is thermodynamically metastable and cannot be heated over ~1200°C because of transition to hexagonal, graphite-like phase [6]. Therefore, sintering of PcBN involves the High Pressure – High Temperature conditions (HPHT) [6-9]. Additions of some metals (e.g. Al) or their compounds to the cBN as a binding phase allow to decrease sintering parameters owing to the formation of a liquid phase during sintering process [10, 11]. In this way the commercial materials: Amorbite (product of the Element Six) and Kiboryt (product of ISM, Ukraine) are produced.

The aim of our work was to obtain high durable fined-grained cBN composite with high values of Young's modulus, hardness and fracture toughness, which can be successfully used as a cutting tool. As known, there are two approaches to obtain cBN composites with "high" and "low" content of superhard phase [12, 13]. The cBN content varies depending on application; "low content cBN material" with ~50 vol% cBN can be used for turning case hardened steels and ball bearing steels, while "high content cBN material" with ~80–90 vol% cBN can be used in high speed machining of cast iron [5]. We used the second one which is concluded in addition a little quantity of the Si_3N_4 nanopowder (up to 3–5%) to cBN. In this case Si_3N_4 phase fills up the pores between cBN grains thus result in resistance to crack propagation by means so called "crack deflection" mechanism (Fig. 1) [14, 15].

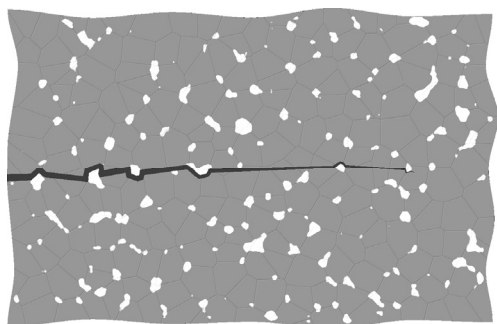


Fig. 1. Crack deflection in cBN– Si_3N_4 composite: cBN matrix – grey, Si_3N_4 phase – white, crack – black

The high content of cBN phase allows to keep Young's modulus and hardness values close to the theoretical ones for pure PcBN.

2. Description of the experiment

2.1. HPHT sintering

The powders of cubic boron nitride (Element Six Micron+ABN, 3–5 μm and 0–250 nm) and alpha Si_3N_4 (Goodfellow, 100–800 nm) were used as starting materials. The two variants of the mixtures were prepared. In the both variants $\alpha\text{-Si}_3\text{N}_4$ (100–800 nm) was applied, but in the first case (indicated as I) only cBN 3–5 μm was used whereas in the second one (indicated as II) cBN 3–5 μm with 10% cBN 0–250 nm was used.

Homogeneous mixtures were preliminary consolidated into pallets of the diameter of 15 mm and the height of 5 mm under the pressure of 200 MPa.

The samples were obtained at high pressure of 7.7 GPa in the temperature range of 1880–2240 °C using a Bridgman-type toroid high-pressure apparatus (Fig. 2). In this type of anvils the pressure distribution in the sample (Fig. 2 and Fig. 3) takes place as a result of compression and extrusion of the so-called "gasket" material. A high pressure gasket was made of a block lithographic stone. The compacted powder was placed in graphite heater in the central part of the cell and the necessary pressure and temperature were applied, after which the temperature was decreased to room temperature and the pressure to atmospheric one (Fig. 4).

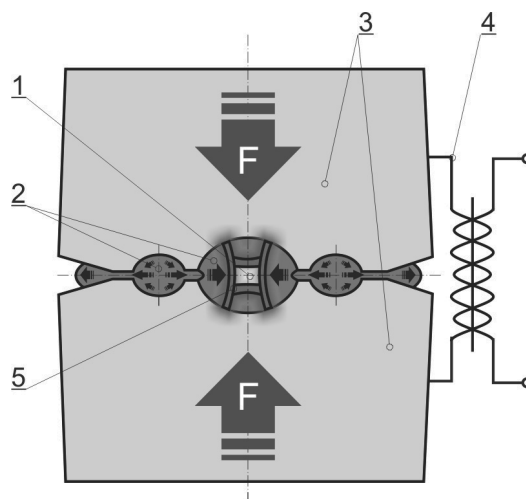


Fig. 2. Sintering process in a Bridgman-type HPHT system. Quasi-hydrostatic compression of the preliminary consolidated powders (sample - 1) is achieved as a result of plastic deformation of the gasket material (2) between anvils (3); electrical heating is provided by a high-power transformer (4) and graphite resistive heater (5)

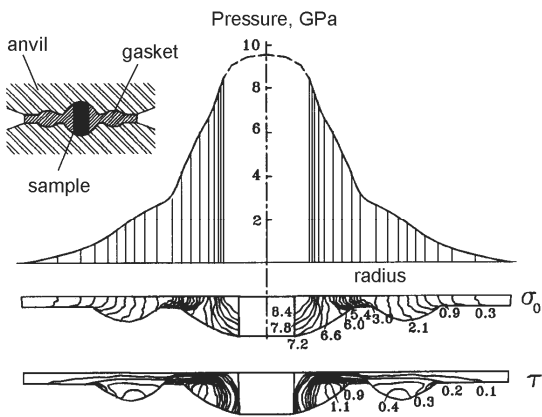


Fig. 3. Pressure distribution in the sample [16]

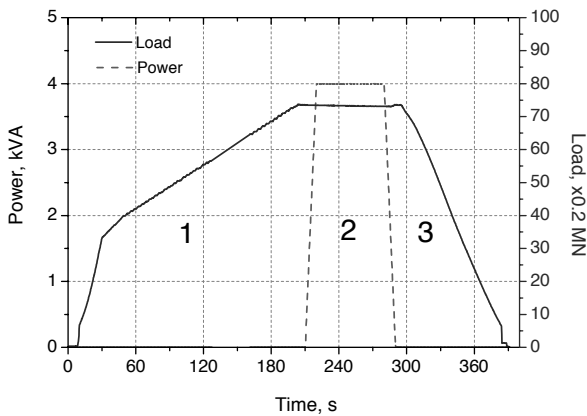


Fig. 4. Three stages of an exemplary process of sintering: 1 – loading, 2 – sintering, 3 – unloading

The sintered cBN-Si₃N₄ compacts (Fig. 5) were subsequently ground to remove remains of graphite after the technological process of sintering and to obtain the required quality and surface parallelism for physical and mechanical studies. Samples intended for microscopic investigations and for some mechanical tests were additionally polished using diamond slurries.

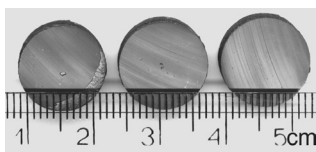


Fig. 5. HPHT sintered samples after face grinding processes

Additionally, commercial material with “high content” (82%) of cBN phase: BZN7000s (General Electric) was used in the mechanical studies as a reference sample.

2.2. Investigation methods

Density of the sintered samples was measured by the hydrostatic method. The uncertainty of the measurements was below 0.02 g/cm³, which gave a relative error value below 0.5 %.

Young’s modulus of the cBN-Si₃N₄ composites obtained at different temperatures was measured basing on the velocity of the ultrasonic waves transition through the sample using the ultrasonic flaw detector Panametrics Epoch III. Ultrasonic methods is non-destructive and belong now to the main techniques used for the determination of the elasticity constants of the materials. The elasticity modulus values are characteristic parameters for many technical materials. In the case of ceramics the elasticity modulus can be considered as the main parameter which determines their properties as well as suitability for a given applications.

The calculations are carried out according to the following formula:

$$E = \rho \cdot C_T^2 \frac{3C_L^2 - 4C_T^2}{C_L^2 - C_T^2} \quad (1)$$

where: E – Young’s modulus, ρ – density of the material, C_L – velocity of the longitudinal wave, C_T – velocity of the transversal wave.

Uncertainty of measurements is below 0.5%. The velocities of transversal and longitudinal waves were determined as the ratio of the sample thickness and the relevant transition time. Considering that the error in the thickness measurements was ± 0.01 mm and in the time-of-flight measurements ± 1 ns, the resulting error in the ultrasonic velocity was about 1%. Consequently, the accuracy of Young’s modulus calculated from equation (1) could be estimated to be 2%.

The hardness and the fracture toughness of samples having the highest values of density and Young’s modulus, were determined by the Vickers method [17] under a load of 98.7 N, using a Future Tech FV-700e Vickers hardness tester. The stress intensity factor K_{Ic} was calculated from the length of cracks which develop during a Vickers indentation test using equation 2:

$$\frac{K_{Ic} \varphi}{H \sqrt{a}} \left(\frac{H}{E \varphi} \right)^{\frac{2}{5}} = 0.129 \cdot \left(\frac{c}{a} \right)^{\frac{3}{2}} \quad (2)$$

where: K_{Ic} – critical stress intensity factor, φ – constrain factor, H – Vickers hardness, E – Young’s modulus, a – half of indent diagonal, c – length of crack.

The coefficients of friction for cBN-Si₃N₄ composites and commercial material BZN7000s (General Electric) in sliding contact with an LH15 bearing steel were determined in Ball-On-disc tests, using a CETR UMT-2MT universal mechanical tester (USA). In the Ball-On-Disc method, sliding contact is realized by pushing a ball specimen onto a rotating disc specimen under a constant load. Tests were carried out without lubricant. The loading mechanism applied a controlled load F_n to the ball holder (Fig. 6.) and the friction force F_t was measured continuously during the test using an extensometer.

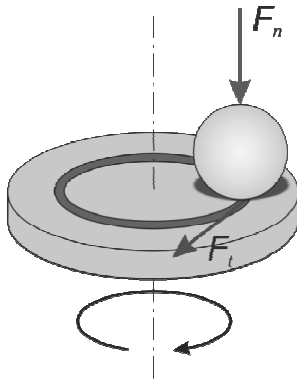


Fig. 6. Scheme of the Ball-On-Disc method

For each test, a new ball was used or the ball was rotated such that a new surface was in contact with the disc. After mounting of the ball and sample, materials were washed in ethyl alcohol and dried.

The size of the disc-shaped samples was $\sim 13.5 \times 3.8$ mm; the surface of the discs flat and parallel to within 0.02 mm; and the roughness of the test surface not more than $0.1 \mu\text{m Ra}$. The samples were ground using diamond wheels and polished using diamond slurries.

The following test conditions were established:

- ball diameter: 3.175 mm,
- applied load: 4 N,
- sliding speed: 0.1 m/s,
- diameter of the sliding circle: 2-4 mm,
- sliding distance: 100 m,
- calculated duration of the test: 1000 s.

Friction coefficient was calculated from the equation 3:

$$\mu = \frac{F_f}{F_n} \quad (3)$$

where: F_f – measured friction force, F_n – applied normal force.

The microstructures were observed by SEM (LEO 1530 GEMINI and JSM-6460LV).

The phase composition was determined using a PHILIPS 1830 X-ray diffractometer ($\text{CuK}\alpha$ radiation). A qualitative phase analysis was based on the records obtained within the angle range 2θ (20 - 120°) using steps of 0.05° and a computation time of 3 s.

3. Results

The first step of experiment was to determine the optimum sintering temperature. For this purpose, composites were sintered at increasingly higher temperatures until it reaches maximum density and Young's modulus values.

Young's modulus and density of cBN-Si₃N₄ (I) composites sintered at temperatures from 1880 to 2240°C are presented in Fig. 7.

Composites show an increase in density and Young's modulus with sintering temperature up to a maximum value. Further

increase of the sintering temperature results in cracking of the samples. This is typical behavior in the sintering of advanced ceramics by the HPHT method. The samples sintered at the temperature of 2240 °C reached the highest relative values of density ($\sim 99.9\%$) and Young's modulus (97%). All samples of cBN-Si₃N₄ (I and II) composites, intended to further testing (hardness, fracture toughness etc.), were sintered at previously determined temperature.

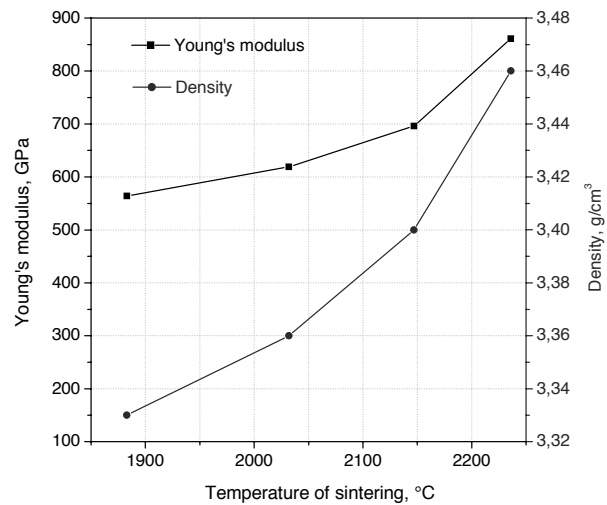


Fig. 7. Young's modulus and density of cBN-Si₃N₄ (I) composites sintered in various temperatures

Average values of density, Young's modulus, hardness, and the fracture toughness for the best samples of cBN – Si₃N₄ (I) and (II) composites sintered at 2240°C as well as for the BZN7000s commercial material (General Electric) are presented in Table 1.

Table 1.

Basic physical and mechanical properties of cBN – Si₃N₄ (I) and (II) composites sintered at 2240 °C

Properties	Materials		
	cBN – Si ₃ N ₄ (I)	cBN – Si ₃ N ₄ (II)	BZN7000s (GE)
Density, g/cm ³	3.46±0.02	3.45±0.02	3.38±0.02
Young's modulus, GPa	842±9	812±17	714±3
Vickers hardness, HV ₁₀	4750±580	4855±630	3820±83
Fracture toughness, K _{Ic} , MPa·m ^{1/2}	10.3±0.8	10.9±1.3	12.2±1.2

All materials sintered at 2240 °C have relative density close to 100%. The comparison of the mechanical properties of cBN-Si₃N₄ (I) and cBN-Si₃N₄ (II) composites showed that the addition of 10 % cBN nanopowder to mixture caused only small increase in hardness from 4750 up to 4855 HV₁₀ and small decrease in Young's modulus from 842 to 812 GPa. Fracture toughness of both I and II type of composites is on the same level above 10 MPa·m^{1/2}. All differences between mechanical properties of investigated cBN-Si₃N₄ composites are in the margin of error.

Commercial material have lower density, Young's modulus and hardness but a little higher fracture toughness in comparison with cBN – Si₃N₄ composites (Table 1).

Microstructures of sintered cBN – Si₃N₄ (I) and (II) composites are presented in Fig. 8 and Fig. 9. Cracks in the cBN – Si₃N₄ (II) composite which developed during a Vickers indentation test are visible in Fig. 10.

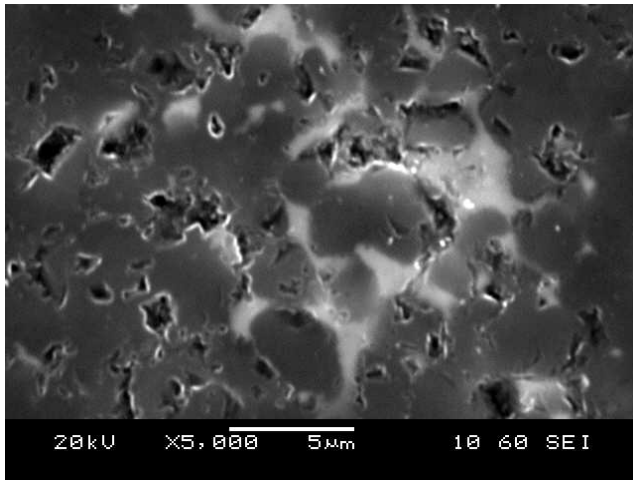


Fig. 8. Microstructure (SEM) of cBN – Si₃N₄ (I) composite (polished surface): cBN – dark areas, Si₃N₄ – light areas

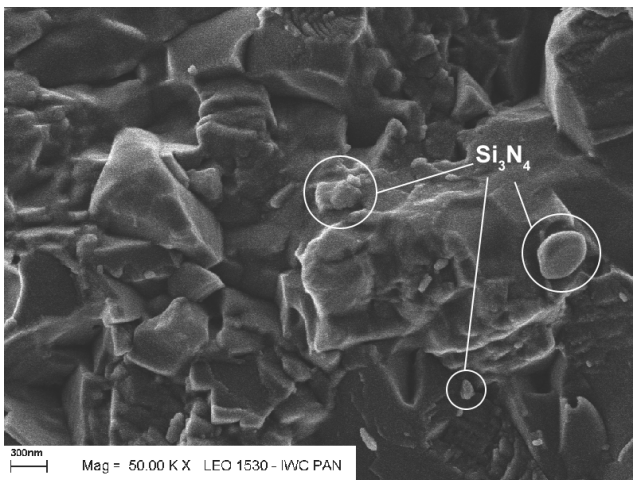


Fig. 9. Microstructure (SEM) of cBN – Si₃N₄ (II) composite (fracture)

Dispersed Si₃N₄ phase is located between cBN grains and filling up the voids and pores in both I and II type of investigated composites (Fig. 8 and Fig. 9). A phase analysis of the composite (Fig. 11) shows no new phases produced during the sintering process. The peaks observed in the diffractogram can be identified to be induced by the cBN and β-Si₃N₄ phases. Evidently α-Si₃N₄ phase is fully transformed in β-Si₃N₄ phase at given sintering conditions.

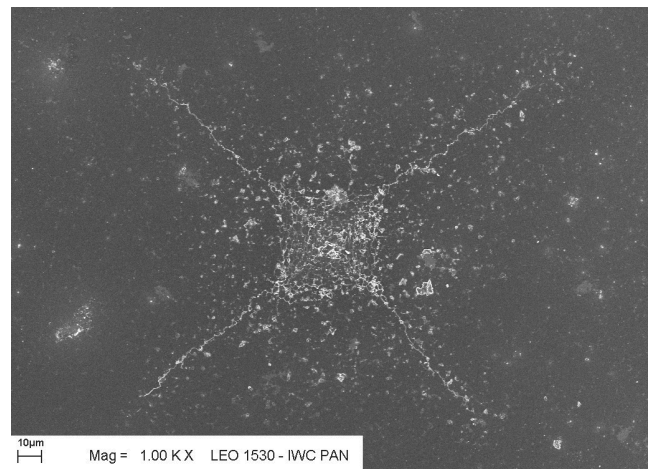


Fig. 10. Cracks in the cBN – Si₃N₄ (II) composite as result Vickers indentation (SEM)

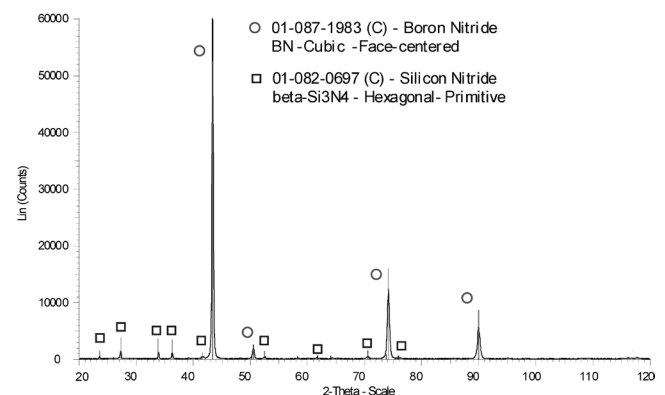


Fig. 11. X-ray diffraction pattern of cBN – Si₃N₄ (II) composite

Because of difference between thermal expansion coefficient of silicon nitride β-phase ($\sim 3.2 \cdot 10^{-6} \cdot K^{-1}$) and of cubic boron nitride ($\sim 5.6 \cdot 10^{-6} \cdot K^{-1}$) some stress is generated in the composite during cooling after sintering process. The highest stress gradient is located at the interface between relatively soft Si₃N₄ particles ($E = 320$ GPa) and stiff cBN matrix ($E = 909$ GPa). When propagating crack (Fig. 10) encounters a sharp stress gradient, it can be deflected from its original trajectory. Accordingly, length of the crack path increases and more fracture energy is absorbed. Furthermore, the change of crack direction reduces the stress intensity near crack tip. As a result, crack propagation process is inhibited which contribute to increasing of fracture toughness of composite.

Comparison of friction coefficient for cBN – Si₃N₄ composites and BZN7000s commercial material during sliding contact with the LH15 still ball is presented in Fig. 12.

Both I and II type of cBN – Si₃N₄ composites as well as BZN7000s commercial material have relatively low coefficient of friction during sliding on LH15 steel ($\mu < 0.45$). The lowest average value of μ of all investigated materials have commercial material ($\mu = 0.31$), the highest have cBN – Si₃N₄ (II) composite ($\mu = 0.41$) while composite I is characterized by the intermediate coefficient of friction ($\mu = 0.36$).

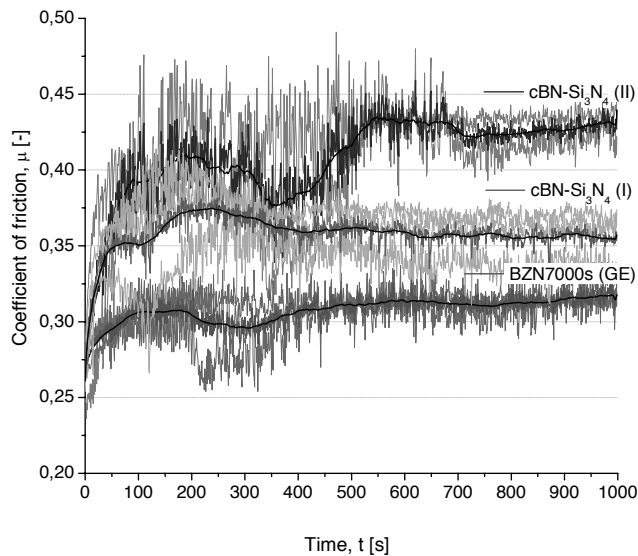


Fig. 12. Coefficient of friction of cBN – Si₃N₄ I and II composites compared with BZN7000s commercial material during sliding contact with the LH15 bearing still ball

Hard ceramic bodies – possessing high fracture toughness and low coefficients of friction – used in mechanical systems that involve high loads, velocities and temperatures, will reduce costs and be less harmful to the environment. Therefore presented materials can be promising for different challenging machining operations e.g. for high speed cutting or interrupted hard cutting.

4. Conclusions

Two variants of the cBN-3%Si₃N₄ composites, contained micro cBN (I) or mixture of micro + 10% nano cBN powders (II), have been sintered by HPHT method.

The samples sintered at the temperature of 2240 °C reached the highest relative values of density (~99.9%) and Young's modulus (97%).

The comparison of the mechanical properties of materials obtained showed that the addition of 10 % cBN nanopowder to mixture caused only small increase in hardness from 4750 up to 4855 HV₁₀ and decrease in Young's modulus from 842 to 812 GPa. Fracture toughness of both I and II type of composites is on the same level above 10 MPa·m^{1/2}. All differences between mechanical properties of investigated cBN-3%Si₃N₄ composites are in the margin of error.

Microstructure analysis indicate that the toughening of presented materials can be attributed to crack deflection mechanism caused by the influence of dispersed Si₃N₄ phase.

All investigated materials have relatively low value of friction coefficient ($\mu < 0.45$).

The material obtained is characterized by favourable combination of hardness and fracture toughness required in different machining operations, e.g. in interrupted hard cutting.

Acknowledgements

This work was supported in the Innovative Economy Programme (2007-2013) under the National Strategic Reference Framework co-financed from EU resources; Priority Axis 1 Research and development of modern technologies; Activity 1.1 Support for R&D projects for entrepreneurs carried out by scientific entities; Section 1.1.3 Development projects; Project No UDA-POIG.01.03.01-12-024/08-00.

References

- [1] R. Haubner, M. Wilhelm, R. Weissenbacher, B. Lux, Boron Nitrides - Properties, Synthesis and Applications, in: M. Jansen, (Ed.), High Performance Non-Oxide Ceramics, Series: Structure and Bonding Vol. 102, Springer-Verlag, Berlin Heidelberg, 2002.
- [2] L. Vel, G. Demazeau, J. Etourneau, Cubic boron nitride: synthesis, physicochemical properties and applications, Materials Science and Engineering B 10 (1991) 149-164.
- [3] S. Thamizhmanii, B. Bin Omar, S. Saparudin, S. Hasan Surface roughness analyses on hard martensitic stainless steel by turning, Journal of Achievements in Materials and Manufacturing Engineering 26/2 (2008) 139-142.
- [4] X.Z. Rong, T. Tsurumi, O. Fukunaga, T. Yano, High-pressure sintering of cBN-TiN-Al composite for cutting tool application, Diamond and Related Materials 11 (2002) 280-286.
- [5] J. Angseryd, M. Elfving, E. Olsson, H.-O. Andrén, Detailed microstructure of a cBN based cutting tool material, International Journal of Refractory Metals and Hard Materials 27 (2009) 249-255.
- [6] O. Fukunaga, The equilibrium phase boundary between hexagonal and cubic boron nitride, Diamond and Related Materials 9 (2000) 7-12.
- [7] P. Klimczyk, V.S. Urbanovich, Micro-, submicro- and nano-Si₃N₄ - SiC composites sintered by the HPHT method, Archives of Materials Science and Engineering 39/2 (2009) 92-96.
- [8] I. Sulima, P. Figiel, M. Suoeniak, M. Świątek, Sintering of TiB₂ ceramics, Archives of Materials Science and Engineering 28/11 (2007) 687-690.
- [9] I. Sulima, P. Klimczyk, P. Hyjek, The influence of the sintering conditions on the properties of the stainless steel reinforced with TiB₂ ceramics, Archives of Materials Science and Engineering 39/2 (2009) 103-106.
- [10] R. Lv, J. Liu, Y. Li, S. Li, Z. Kou, D. He, High pressure sintering of cubic boron nitride compacts with Al and AlN, Diamond and Related Materials 17 (2008) 2062-2066.
- [11] A. McKie, J. Winzer, I. Sigalas, M. Herrmann, L. Weiler, J. Rodel, N. Can, Mechanical properties of cBN-Al composite materials, Ceramics International 37 (2011) 1-8.
- [12] K. Okamura, S. Kukino, T. Fukaya, Development of SUMIBORON BN350 and BNC300 for Interrupted Cutting of Hardened Steel, Sumitomo Electric Technical Review 59 (2005) 66-70.
- [13] M. Ota, S. Kukino, S. Uesaka, T. Fukaya, Development of SUMIBORON PCBN Tool for Machining of Sintered

- Powder Metal Alloys and Cast Iron, Sumitomo Electric Technical Review 59 (2005) 60-65.
- [14] Y.-L. Chin, W.-H. Tuan, Contribution of plastic deformation of Ti_3SiC_2 to the crack deflection in the $\text{Al}_2\text{O}_3/\text{Ti}_3\text{SiC}_2$ composites, *Materials Science and Engineering: A* 528 (2011) 3270-3274.
- [15] L.A. Dobrzański, B. Dołżańska, Hardness to toughness relationship on WC-Co tool gradient materials evaluated by Palmqvist method, *Archives of Materials Science and Engineering* 43/2 (2010) 87-93.
- [16] M.I. Eremets, *High pressure Experimental Methods*, Oxford University Press, 1996.
- [17] T. Taniguchi, M. Akaishi, S. Yamaoka, Mechanical Properties of Polycrystalline Translucent Cubic Boron Nitride as Characterized by the Vickers Indentation Method, *Journal of American Ceramic Society* 79/2 (1996) 547-49.