Influence of binder systems on sintering characteristics, microstructures, and mechanical properties of PcBN composites fabricated by SPS

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Abstract: Cubic boron nitride (cBN) with high hardness, thermal conductivity, wear resistance, and chemical inertness has become the most promising abrasive and machining material. Due to the difficulty of fabricating pure cBN body, generally, some binders are incorporated among cBN particles to prepare polycrystalline cubic boron nitride (PcBN). Hence, the binders play a critical factor to the performances of PcBN composites. In this study, the PcBN composites with three binder systems containing ceramic and metal phases were fabricated by spark plasma sintering (SPS) from 1400 to 1700 °C. The sintering behaviors and mechanical properties of the composites were investigated. Results show that the effect of binder formulas on mechanical properties mainly related to the compactness, mechanical performances, and thermal expansion coefficient of binder phases, which affect the carrying capacity of the composites and the bonding strength between binder phases and cBN particles. The PcBN composite with SiAlON phase as binder presented optimal flexural strength (465 ± 29 MPa) and fracture toughness (5.62 ± 0.37 MPa·m^{1/2}), attributing to the synergistic effect similar to transgranular and intergranular fractures. Meanwhile, the excellent mechanical properties can be maintained a comparable level when the temperature even rises to 800 °C. Due to the weak bonding strength and high porosity, the PcBN composites with Al₂O₃–ZrO₂(3Y) and Al–Ti binder systems exhibited inferior mechanical properties. The possible mechanisms to explain these results were also analyzed.

Keywords: polycrystalline cubic boron nitride (PcBN); binder system; sintering characteristic; high temperature mechanical property; fracture mechanism

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1 Introduction

Cubic boron nitride (cBN) possesses high thermal conductivity, wear resistance, chemical inertness, and excellent mechanical properties and is the second hardest material next to diamond [1,2]. It has become the most promising cutting component in the abrasive and machine tool industry because of its low chemical reactivity with iron (Fe), nickel (Ni), and related alloys; thereby it can compensate the defect of diamond reacting and decomposing at high temperatures [3–5].

Nevertheless, due to the high-strength covalent bonds and low self-diffusion coefficient of B and N, it is difficult to obtain big single crystal of cBN (or pure cBN body) even under the extreme sintering condition of ultra-high temperature ($\sim 2000^\circ\text{C}$) and pressure ($\sim 7\text{ GPa}$) [6,7]. Similar to polycrystalline diamond, therefore, polycrystalline cubic boron nitride (PcBN) was proposed by combining cBN particles and appropriate amount of binder to fabricate the composites under relatively low temperatures and pressures [8]. Moreover, PcBN composites as cutting tools allow for efficient dry and finish machining for turning case hardened steels, ball bearing steel, and chilled cast iron in multiple operations like high/low cutting speeds, high/low feed rates, and continuous/intermittent cutting manner, which could obtain considerable economic benefits on account of reducing production costs [4,9].

In PcBN composites, cBN particles mainly contribute to the high hardness and wear resistance for the monolithic materials. And more mechanical properties, such as strength, toughness, and service reliability, are depended on the binder systems and formulas. Hence, the binders play the vital roles in both synthesis processes of PcBN composites and their service properties [10]. Composition of appropriate system and amount of binder not only can reduce the sintering temperature and pressure, but also improve the properties of the sintered body [8]. According to the different application fields (hardened steel, cast iron, and other workpiece materials) and processing methods, proportioning schemes of PcBN composites with different characteristics were designed due to the factors of cBN content, particle size, and binder type [10–13]. Conventionally, some ceramic phases like Al_2O_3 [4,14], SiAlON [15], Y_2O_3 stabilized ZrO_2 (YSZ) [16,17], MAX [13] phases with fine sinterability, and good carrying capacity, metallic phases like Al with low melting point, Ti with high reactivity, and W with similar thermal expansion

coefficient [8], were usually chosen as binders. Ji *et al.* [5] reported a eutectic melt of Y–Si–Al–O–N system formed from Y_2O_3 , Al_2O_3 , Si_3N_4 , and Al, which could accelerate the densification of the composites, improve mechanical properties and thermal performance. PcBN composites were reactively sintered by spark plasma sintering (SPS) through adding 14.7 wt% Al and 43 wt% Ti as binder system. During sintering processes, the generation of new phases deriving from chemical reactions promoted chemical bonding with cBN, whose relative density, bending strength, and fracture toughness reached $98.9\%\pm 0.10\%$, $390.7\pm 4.5\text{ MPa}$, and $7.6\pm 0.10\text{ MPa}\cdot\text{m}^{1/2}$, respectively [18]. In addition, considering the problem of phase transformation from cBN to hBN due to its metastability during high-temperature and long-time sintering, it is crucial to choose suitable sintering technology and process parameters. SPS, a superior sintering technology to heat powder materials via a sequence of direct current pulses [19], possesses high heating rate, and short holding time [20], which can shorten the exposure time of the cBN grains at high temperatures and suppress phase transformation from cBN to hBN [18,21].

Moreover, due to the heat generated by serious friction, high speed or high feed rate would result in local high temperature of PcBN composites during dry machining processing [22,23]. It was reported that the average cutting temperatures of the tool-chip were ranged from 400 to 800°C when machining at feed rate of 0.25 mm/rev and cutting speed from 35 – 125 m/min by PcBN composite tools [22]. With the temperature rising during processing, the mechanical performance and wear resistance of PcBN composites were frequently degraded, thereby bringing down the tool life [24,25]. However, few relative researches on evolution of mechanical properties with the increment of temperature could be found. Thus, it is of great significance of investigating the high temperature mechanical properties of PcBN composite to master its performances at actual service status and material design.

In present work, three systems of binders consisting of ceramic and metal phases were selected to fabricate PcBN composites by SPS. The sintering characteristics and phase transition behaviors, together with the compositions, microstructure, and mechanical properties of each proportioning scheme at different sintering temperatures were investigated. Moreover, aiming at high temperature service conditions, the mechanical

properties at elevated temperatures up to 800 °C were investigated based on the optimal proportioning scheme, followed by discussing the fracture behaviors of the composites.

2 Materials and methods

Commercially available cBN particles (5–10 µm; Zhengzhou Zhongnan Jete Superabatives Co., Ltd., Zhengzhou, China) was chosen as raw material. Al and Ti powder (~75 µm; Beijing Huisheng Advanced Materials Technology Co., Ltd., Beijing, China), Si₃N₄ powder (~20 nm, purity 99.9%; Beijing Huawei Ruike Chemical Co., Ltd., Beijing, China), AlN powder (~40 nm, purity 99.9%; Beijing Huawei Ruike Chemical Co., Ltd., Beijing, China), α-Al₂O₃ powder (80–100 nm, purity 99.9%; Jiangsu Lida High-Tech Special Materials Co., Ltd., Jiangsu, China), Y₂O₃ powder (~50 nm, purity 99.9%; Beijing Huawei Ruike Chemical Co., Ltd., Beijing, China), and ZrO₂(3Y) powder (10–20 nm, purity 99.9%; Jiangsu Lida High-Tech Special Materials Co., Ltd., Jiangsu, China) were applied in binder systems. The proportioning schemes of PcBN composites were designed with three different binder systems and formulas. The corresponding samples were marked as Samples S1, S2, and S3. Detail components and the proportions are listed in Table 1. The relevant raw powders were mixed by ball milling in ethanol solution for 20 h and then dried at 70 °C for 24 h. The homogeneous mixtures were dry-pressed in a steel mold for 10 min at a pressure of 300 MPa to obtain consolidated green body. Subsequently, the green body was equipped in a graphite die and sintered by SPS. Sintering temperature was measured by an infrared thermometer through graphite pressing rod with a penetrating hole and graphite block close to sample, to more accurately master the actual temperature of the sample. A graphite paper with thickness of 0.2 mm was inserted between the graphite block and graphite die to ensure fully contact to pass the uniformly current. The sintered procedures were conducted at different temperatures for 10 min in vacuum with the

heating rate of about 125 °C/min. A bidirectional pressure of 35 MPa was adopted in the axial direction of the graphite mold.

Density measurements of the composites were carried out using the Archimedes principle according to the standard ISO 18754:2003. The apparent porosity (π_a) and bulk density (ρ_b) were calculated from Eqs. (1) and (2):

$$\pi_a = (m_3 - m_1)/(m_3 - m_2) \times 100 \quad (1)$$

$$\rho_b = m_1/(m_3 - m_2) \times \rho_l \quad (2)$$

where m_1 , m_2 , and m_3 are the dry weight, buoyant weight, and wet weight of the samples, respectively. ρ_l is the density of immersed liquid at experimental temperature.

The Vickers hardness (HV) of specimens was determined by indentation test using a microhardness tester (MH-5-VM) on the polished surface of specimens under 500 g and keeping for 5 s. The specimens were machined into 3 mm×4 mm×25 mm and 2 mm×4 mm×25 mm for the flexural strength and fracture toughness tests, respectively. The tests were conducted on an electric universal testing machine (AGS-X-20KN) at room temperature (RT) and high temperatures (400, 600, and 800 °C). Single-edge notched beam (SENB) tests were conducted to measure the fracture toughness with a span of 16 mm and a crosshead speed of 0.05 mm/min. The flexural strengths were measured using a 3-point flexural test method with a span of 20 mm and a crosshead rate of 0.5 mm/min. Noted that the values presented are the average values of 5–8 repeats. When testing the high temperature mechanical properties, the operating equipments were placed in a furnace to create a high temperature environment, as shown in Fig. 1. At the beginning of the test, the temperature rose to target value and hold for 5 min to ensure specimen reach the corresponding temperature.

Fig. 1 Schematic diagram of high temperature operating equipment of mechanical test.

Table 1 Component mass ratios (wt%) of three composites

Sample	cBN	Si ₃ N ₄	AlN	Y ₂ O ₃	Al ₂ O ₃	ZrO ₂ (3Y)	Al	Ti
S1	50	22	14	4	10	—	—	—
S2	50	—	—	—	45	5	—	—
S3	45	—	—	—	—	—	15	40

The microstructures, morphologies of polished and fracture surfaces of the samples, as well as crack propagation patterns were characterized by the scanning electron microscope (JSM-5600LV, Japan), high resolution field emission scanning electron microscope (SU8020, Hitachi, Japan and Quanta 650 FEG, FEI, USA) equipped with backscattered electron (BSE) images, and energy dispersive spectrum analysis (EDS). The crystalline phases were identified qualitatively using X-ray diffraction (XRD) spectrum analysis (EMPYREAN, PANalytical, the Netherlands).

3 Results and discussion

3.1 Phase composition, microstructure, and density of the PcBN composites

Various systems of binders have complicate influence on the compositions of PcBN composites, and typically there are new phases generated from chemical reaction during sintering. The composites sintered at different temperatures were analyzed by XRD, and the results are shown in Fig. 2. It can be observed that several relevant new phases were detected in Samples S1 and S3, indicating chemical reaction existed during the sintering process in their composition systems. In contrast, the spectra of Sample S2 show the phases of α -Al₂O₃, cBN, and ZrO₂, the same as initial compositions of the proportioning scheme, which demonstrates that there were no chemical reactions among α -Al₂O₃, cBN, and ZrO₂ during sintering process.

As to Sample S1, β -SiAlON (Si₄Al₂O₂N₆) and O'-SiAlON (Si_{1.8}Al_{0.2}O_{1.2}N_{1.8}) phases were produced from the reaction among Si₃N₄, Al₂O₃, and AlN. The Si–N bonds of Si₃N₄ were constantly replaced by Al–O bonds of Al₂O₃ to construct β -SiAlON ceramic as a result of similar bond length between Al–O (1.75 Å)

and Si–N (1.74 Å) bonds to keep charge balance [26]. The chemical equation could be expressed as follows:

SiAlON ceramics have superior sinterability, excellent mechanical properties, and good high temperature performance including high temperature strength, oxidation resistance, and thermal shock resistance, thereby having widely application prospects as structural components and cutting tools [27,28]. Sample S3 mainly produced new reaction products of TiB₂, AlB₂, TiN, AlN, as well as minor amounts of hBN when sintered at 1500 °C. The corresponding chemical equations could be expressed as follows [18]:

The metal phases Al and Ti can promote density of composites during sintering process by filling the clearance of cBN particles, activating the surface of cBN, and reacting with cBN [18]. Meanwhile, the chemical reactions produced TiN, AlN, and TiB₂ phases with high strength, hardness, and chemical stability, which were conducive to improving the mechanical properties of composites. Nevertheless, the phase transformation from cBN to hBN could result in degradation of mechanical properties and density of the composites on account of weak bond between interlayers of hBN similar to graphite. Although the same and even higher sintered temperature was adopted in Samples S1 and S2, there was no hBN phase detected, indicating that these binder systems contribute to the inhibition of the phase transition.

The morphologies of three kinds of PcBN composites are shown in Fig. 3. Different phases can be distinguished easily due to their distinct atomic number from BSE images (Figs. 3(a)–3(c)). The larger black contrasts (zones 1, 3, and 6 in Fig. 3) dispersed in PcBN composites

Fig. 2 XRD spectra of the PcBN composites sintered at different temperatures: (a) Sample S1, (b) Sample S2, and (c) Sample S3.

were mainly composed of cBN particles with large amounts of B and N elements, as shown in corresponding element weight percent. And the particle size of cBN was 5–10 μm resulted from raw microsized powders. Some element weight percents in other zones were also given in Fig. 3. For Sample S1, the gray area (zone 2) was mainly $\alpha\text{-Al}_2\text{O}_3$ and SiAlON phases (Fig. 3(a)). The gray and white areas (zones 4 and 5) were mainly formed by $\alpha\text{-Al}_2\text{O}_3$, $\text{ZrO}_2(3\text{Y})$, and a bit of cBN (Fig. 3(b)). The gray (zone 8) and white (zone 7) areas in Sample S3 were mainly composed of TiN , AlN , TiB_2 , AlB_2 , and a small quantity of BN (Fig. 3(c)). There are no obvious interfacial defects like cracks, holes, or fragments in the composites. And the polygonal cBN particles surrounded by binder phases relatively uniformly distributed in composites to construct a continuous net structure in Samples S1 and S2. Nevertheless, Sample S3 exhibited obvious agglomeration and maldistribution of cBN particles compared with that of Samples S1 and S2, and it can be ascribed to the difference of plasticity and fluidity between ceramic and metal during sintering.

It is favorable to improve sintering temperature for promoting the compactness of PcBN composites (Fig. 4). The apparent porosity of Samples S1 and S2 could reduce to 0.44% and 0.74% at the sintered temperature of 1600 and 1700 $^{\circ}\text{C}$, respectively, while that of Sample S3 just decreased to 7.68% at sintered temperature of 1600 $^{\circ}\text{C}$. Meanwhile, the maximum bulk densities of three samples were obtained at 1600–1700 $^{\circ}\text{C}$, which were 3.34, 3.60, and 3.49 g/cm^3 . Although Sample S3 presented the highest porosity, it simultaneously possessed high bulk density because of the high density of metal binder system. Low apparent porosity could produce a considerable promotion on carrying capacity of materials, thereby directly improving mechanical properties.

3.2 Influence of binder systems and formulas on mechanical properties

The mechanical performance is a crucial parameter to evaluate carrying capacity and damage resistance of ceramic materials. To elucidate the influences of binder

Fig. 3 BSE morphologies and element weight percents with different areas of polished surfaces of (a) Sample S1 sintered at 1600 $^{\circ}\text{C}$, (b) Sample S2 sintered at 1700 $^{\circ}\text{C}$, and (c) Sample S3 sintered at 1600 $^{\circ}\text{C}$.

Fig. 4 (a) Apparent porosity and (b) bulk density of three kinds of PcBN composites sintered at different temperatures.

formulas and sintered temperatures on the mechanical properties of PcBN composites, HV, flexural strength (σ), and fracture toughness (K_{IC}) of three kinds of PcBN composites sintered at different temperatures were measured, and the results are given in Figs. 5 and 6. From Figs. 5 and 6, it can be seen that the mechanical properties were generally improved with the increase of sintered temperature in general. At the same time, the binder systems and formulas also have significant influence on mechanical performances. The hardness of three kinds of PcBN composites showed contrary tendency to their apparent porosity, indicating that the variation of hardness related to their binder phases and densification. The hardness of Samples S1, S2, and S3 reached a maximum value of 15.59 ± 0.54 , 14.83 ± 0.82 , and 14.14 ± 0.65 GPa, respectively.

Fig. 5 Vickers hardness of the PcBN composites sintered at different temperatures.

Fig. 6 (a) Flexural strength and (b) fracture toughness of the PcBN composites sintered at different temperatures.

The flexural strength and fracture toughness of the PcBN composites presented a great difference. Sample S1 exhibited a significant improvement and possessed a highest flexural strength and fracture toughness with the values of 465 ± 29 MPa and 5.62 ± 0.37 MPa·m^{1/2}, respectively, when the sintering temperature rose to 1600 °C. Nevertheless, there was no significant improvement of flexural strength and fracture toughness of Sample S3, with relatively high initial value and low final value as the sintering temperatures increased from 1400 to 1600 °C. Compared with Sample S1, Sample S2 showed relative inferior carrying capacity and fracture resistance property with a slight improvement. Its flexural strength and fracture toughness just reach 145 ± 7 MPa and 3.52 ± 0.21 MPa·m^{1/2}, respectively, when the sintering temperature increases up to 1700 °C. The mechanical properties of PcBN composites can be attributed to the relative density, binding force between different phases, as well as the fracture mechanisms of samples, which will be discussed below.

The microstructures of fracture surfaces of the PcBN composites after fracture testing are shown in Fig. 7. The rough and uneven fractural morphologies could be observed directly. The features of cBN particle pullout and tear as well as brittle fracture of SiAlON phase were presented in the fracture surface of Sample S1 (Fig. 7(a)), demonstrating that the fracture pattern similar to transgranular and intergranular fractures simultaneously existed in the PcBN composites during fracture process. Meanwhile, there are no obvious cracks or pores between cBN particles and SiAlON phases, indicating good interface bond, thereby improving the strength of the composites. As for Sample S2, cBN particles pulled out with obvious particles and deep pits, indicating that the cracks could easily propagate from the weak bonding strength between cBN particles and Al₂O₃ phase (Fig. 7(b)). The weak bonding strength may be mainly due to the conspicuous difference of thermal expansion coefficient between cBN ($(1.2\text{--}3.4) \times 10^{-6} \text{ K}^{-1}$) and Al₂O₃ ($7.2 \times 10^{-6} \text{ K}^{-1}$) phases, which resulted in high residual stress and thermal stress concentration at their interface [11]. Moreover, the Al₂O₃ phase also presented typical transgranular fracture characteristics with clear grain boundaries. Thus, Sample S2 presented poor flexural strength and fracture toughness. Additionally, some pores can be observed in the fracture surface of Sample S3 (Fig. 7(c)), which was consistent with its high porosity and was the main adverse factor for the mechanical

Fig. 7 SEM morphologies of fracture surfaces of the PcBN composites: (a) sample S1 sintered at 1600 °C, (b) Sample S2 sintered at 1700 °C, and (c) Sample S3 sintered at 1600 °C.

properties. Moreover, the phase transformation of cBN would also weaken the bonding strength between cBN particles and binder phases by generating new weak phase of hBN. Hence, the binder phases played the key roles for maintaining the mechanical properties of the PcBN composites. The intergranular fracture will assume much more fracture energy compared with transgranular fracture as a result of laniating particles with strong bond, and previous research illustrated the fracture mode of a mixed fracture of intergranular and transgranular of ceramic composites resulted from a balance between the grain boundary “strengthening” and grain “weakening” exhibiting the optimal strength [29].

To further confirm the above descriptions, the crack propagation paths of the PcBN composites were further observed and the results are shown in Fig. 8, which can reflect crack propagating along phase interfaces or across cBN particles, thereby indicating the bonding strength between cBN particles and binder phases. Obviously, both cracks propagating through cBN particles and SiAlON phase existed in Sample S1 except for along with their interfaces (Figs. 8(a) and 8(d)).

It was these fracture behaviors that could efficiently improve the carrying capacity as a result of cracks through cBN particles and deflection at the interfaces. As for Sample S2, however, cracks almost completely propagated along with the interfaces between cBN particles and Al_2O_3 phase, illustrating that it was easy for cracks to propagate rapidly accompanied by the drawing of cBN particles and small quantities of microcracks. The inferior mechanical properties were attributed to the poor bonding strength that could reduce its carrying capacity. Furthermore, cracks in Sample S3 mainly propagated through the binder phases with some cBN particles pulling out other than through cBN particles, indicating the bonding strength between cBN particles and binder phases together with the performance of binder phases limiting the mechanical properties.

Therefore, the binder systems and formulas are critical for the mechanical properties of PcBN composites by means of influencing fracture model and crack behaviors, which were decided by performance of binder phases and the bonding strength between cBN particles and binder phases.

Fig. 8 SEM morphologies of crack propagation path after mechanical tests: (a, d) Sample S1 sintered at 1600 °C, (b, e) Sample S2 sintered at 1700 °C, and (c, f) Sample S3 sintered at 1600 °C.

3.3 Mechanical properties of the PcBN composite at elevated temperatures

Considering the local temperature rise of the composite as mentioned in Section 1, its high temperature mechanical properties are even more important for determining the actual service performance and life. The Sample S1 with superior mechanical properties at RT was selected to evaluate its high temperature mechanical performances at 400, 600, and 800 °C. The results are shown in Fig. 9. With the test temperature increasing, the fracture toughness showed a slight downward trend, demonstrating that the environment temperature had a certain effect. Even at 800 °C, however, the fracture toughness can maintain at a relatively high value ($5.13 \pm 0.40 \text{ MPa} \cdot \text{m}^{1/2}$), which was only 8.7% less than that at RT. There was no obvious attenuation of the flexural strength but a slight increase and the values were always not less than 450 MPa with elevating test temperatures. It can reach 545 ± 37 , and $467 \pm 18 \text{ MPa}$ at 600 and 800 °C, respectively. The reduction of fracture toughness was probably related to the softening of SiAlON phases at high temperatures, insufficient thermal resistance, and poor grain boundary bond, which led to the degradation of its mechanical properties at high temperatures [30–32]. On the whole, the composite can still maintain considerable mechanical properties at high temperatures without obvious reduction, illustrating that the material possessed excellent carrying capacity from RT to 800 °C.

Fig. 9 High temperature mechanical properties of Sample S1 at elevated temperatures.

The morphologies of fracture surfaces and crack propagation path after the mechanical property tests at elevated temperatures are shown in Figs. 10 and 11, respectively. Similar to the morphologies tested at RT, the characteristics of cBN particle pullout and SiAlON phase brittle failure could be observed from Figs. 10 and 11. Crack deflection, unit broken, and cBN particle pullout and tear could also be observed from crack propagation pattern (Fig. 11). When test temperature raised to 800 °C, the crack propagation path became straighter because the crack propagated predominately through SiAlON phase with few cBN particle pullout. All of above implied that the binder system has good performances and interaction with cBN particles from RT to 800 °C. Therefore, the PcBN composite adopting Sample S1 as binder system could maintain considerable high strength, toughness, and stability to resist damage from RT to 800 °C.

Fig. 10 SEM morphologies of fracture surfaces after the mechanical property tests at elevated temperatures.

Fig. 11 SEM morphologies of crack propagation path after the mechanical property tests at elevated temperatures.

4 Conclusions

In this study, the PcBN composites with different systems and formulas of binders were fabricated at 1400–1700 °C by SPS. The sintering characteristics, compositions, microstructures, and mechanical properties of composites were investigated. The following conclusions can be drawn:

1) The proportioning schemes of PcBN composites have a great influence on the sintering characteristics. The binder systems of Samples S1 and S2 can restrain the phase transition of cBN and promote the compactness of the composites, and the apparent porosity can reduce to less than 0.8%.

2) The mechanical properties of the PcBN composites are determined by binder systems and formulas. The good mechanical performances and appropriate coefficient of thermal expansion of binders influence the fracture characteristics. The flexural strength and fracture toughness of the PcBN composite with SiAlON phase as binder can reach 465 ± 29 MPa and 5.62 ± 0.37 MPa·m^{1/2}, respectively.

3) The PcBN composite with SiAlON phase as binder can maintain considerable mechanical performance from RT to 800 °C. The flexural strength and fracture toughness can reach 467 ± 18 MPa and 5.13 ± 0.40 MPa·m^{1/2}, respectively, at 800 °C, attributing to the good performance and interaction of the binder phase with cBN particles.

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