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Influence of sintering temperature on structure and mechanical properties of polycrystalline cubic boron nitride composites

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Abstract

Polycrystalline cubic boron nitride (PcBN) cutting tool materials were prepared by mixing cBN (85 wt.%) and TiN-Ti-Co (15 wt.%) micropowders under high temperature and ultra-high pressure. X-ray diffraction and scanning electron microscopy were employed to analyse the phase composition and microstructure of the composites. The effects of sintering temperature on the mechanical properties and cutting performance of the PcBN ceramics were also investigated. The results indicated that at the ultra-high pressure of 6 GPa and temperatures ranging from 1400 to 1700 °C, the PcBN composites consist of BN, TiN, TiB_2 and Co phases. The PcBN sintered at 1700 °C exhibited the most favourable comprehensive mechanical properties, including a flexural strength of 909 MPa and microhardness of 36.8 GPa. In addition, the cutting tool fabricated from the PcBN composite sintered at 1700 °C showed superior cutting performances in testing with 40Cr mechanical parts.

Keywords: *boron nitride, composites, ultra-high pressure sintering, mechanical properties, cutting tools*

I. Introduction

Polycrystalline cubic boron nitride (PcBN) is a superhard material formed from carefully selected cubic boron nitride particles and binder particles sintered under high temperature and pressure. PcBN with its numerous advantages is often considered the preferred tool for machining very hard and challenging materials [1–4]. The performance of PcBN is affected by the size and content of cubic boron nitride (cBN) grains and the type of binder used. The size and content of cBN grains primarily affect the hardness and wear resistance of PcBN. Inclusion of a binding agent optimizes sintering conditions and enhances the interfacial bonding of cBN, thereby improving the overall performance of PcBN.

The quantity and type of binder significantly influence the application areas of PcBN composites. Currently, PcBN composites are primarily categorized into two groups: high content PcBN composites (H-PcBN) and low content PcBN composites (L-PcBN). H-PcBN composites typically contain >80 vol.% cBN and are primarily employed in roughing and interrupted machining due to their high hardness and fracture toughness. L-PcBN composites generally contain between 45 and 65 vol.% cBN and are primarily utilized in continuous machining due to their high chemical stability. Some metals such as Ti, Al, Co, W and Cr are often used as binding agents [1,5–7], since their low melting point enables formation of liquid phase at the synthesis temperature and thus promote cBN densification. However, the mixtures of some metals, metal nitrides and/or metal carbides are also commonly used and very efficient binders [5,8–23].

In this study, TiN-Ti-Co were used as binders in preparation of PcBN composites. Titanium was selected since it not only increases bonding strength, but also reacts with cubic boron nitride (cBN) to form compounds such as titanium diboride (TiB_2) and titanium nitride (TiN). This reaction improves the properties of PcBN, including red-hardness, wear resistance, thermal stability and fracture toughness. Titanium nitride, with its high melting point, good electrical conductivity and high hardness, is considered as one of the best binders suitable for fabrication of cBN-based systems. Cobalt is

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a commonly used binder that can maintain strong mechanical properties at high temperatures. Co and cBN have a strong affinity and can be tightly bonded together, thus enhancing the overall toughness of PcBN [1,24].

PcBN composites were prepared from microsized precursor powders under high temperature and ultra-high pressure. The influence of sintering conditions on microstructure and mechanical properties were analysed in depth. Finally, high content PcBN cutting tool materials with excellent performance were successfully developed.

II. Experimental

2.1. Sample preparation

The cBN-TiN-Ti-Co composites were prepared using high temperature and high pressure sintering. The following raw materials were used: cBN (average particle size 4–5 μm, purity 99.9%, Funike Superhard Materials Co. Ltd, China), Ti (particle size 3 μm, purity 99.9%, Shanghai Aladdin Biochemical Technology Co. Ltd, China), Co (particle size 500 nm, purity 99.9%, Wuxi Edma Technology Co. Ltd, China) and TiN (particle size 1.5 μm, purity 99.9%, Wuxi Edma Technology Co. Ltd, China). The precursor powders were mixed in a planetary ball mill. The mixture contained 85 wt.% of cBN and 15 wt.% of TiN-Ti-Co (having the constituent weight ratio 8:5:2) powders (Table 1). After grinding and mixing in a planetary ball mill, the mixture was vacuum dried. The mixed powder was put into a cylindrical metallic zirconium cups with a diameter of 35 mm and then pre-compressed in a cold press forming machine at 30 Pa. Finally, the samples were loaded into moulds and sintered at high temperature and ultra-high pressure using a six-sided top press. The sintering conditions are given in Table 1.

Table 1. Experimental formula and process parameters

Sample	Raw materials		Synthesis temperature	Pressure/ time
	cBN	TiN-Ti-Co		
T1	85%	15%	1400 °C	6 GPa/ 8 min
T2			1500 °C	
T3			1600 °C	
T4			1700 °C	

2.2. Testing of PcBN samples

The PcBN samples were first ground and polished, followed by the relevant performance tests. The phase compositions were examined by using X-ray diffractometry (PANalytical X'Pert PRO, The Netherlands). The cross-section microstructure and densification of the PcBN samples were characterized by using scanning electron microscopy (Gemini SEM 300, ZEISS, Germany).

The Archimedes method (GB/T3810.3-2006) was used to determine the porosity of the PcBN samples by the following formula:

$$P = \frac{M_3 - M_1}{M_3 - M_2} \cdot 100 \tag{1}$$

The sample was put into anhydrous ethanol for ultrasonic cleaning, dried and weighted (M_1). Then the test sample was put into anhydrous ethanol and kept under the vacuum until no bubbles were produced on the sample surface to obtain the mass of the sample in alcohol (M_2). Finally, the sample was removed, the surface alcohol was wiped with a towel and the mass of the sample in air (M_3) was weighed.

Mechanical properties (Vickers hardness and flexural strength) and cutting performances of the samples were analysed. The Vickers hardness was tested using HVS-50 hardness tester (Dongguan Lonroy, Equipment, Co. Ltd, China) and calculated by the formula:

$$Hv = 1.854\frac{P}{d^2} \tag{2}$$

where P is the applied load and d is the average of the two diagonals lengths of the Vickers hardness indentation. Load of 98 N and a pressure holding time of 15 s were used in the testing procedure.

The flexural strength was measured on a universal mechanical testing machine (AGS-X5KN, Shimadzu, Japan) using the three-point flexural method. The width L was set to 10 mm, the loading speed was 0.5 mm/min and the formula for calculating the flexural strength was as follows:

$$F = \frac{3P \cdot L}{B \cdot H^2} \tag{3}$$

where P is the applied load, L is the span length (equal to 10 mm), B and H are the width and thickness of the sample.

The DNMA150404 type cutting tools were fabricated from the PcBN materials and used for cutting test involving workpiece made of the 40Cr mechanical parts with a hardness of 52–58 HRC.

III. Results and discussion

3.1. Phase composition

XRD analyses of the PcBN samples sintered at different temperatures (Fig. 1) confirm that all PcBN samples are composed of BN, TiB_2, TiN and Co. Among them, BN, TiN and Co are added in the raw material and they maintain their initial state during the sintering process. While TiB_2 is the new phase generated after sintering, the XRD pattern does not detect the monomorphic titanium metal, which indicates that the monomorphic titanium reacts chemically with cBN to generate the new TiB_2 and TiN phases. The corresponding reaction is as follows:

$$2\,BN + 3\,Ti \longrightarrow 2\,TiN + TiB_2 \tag{4}$$

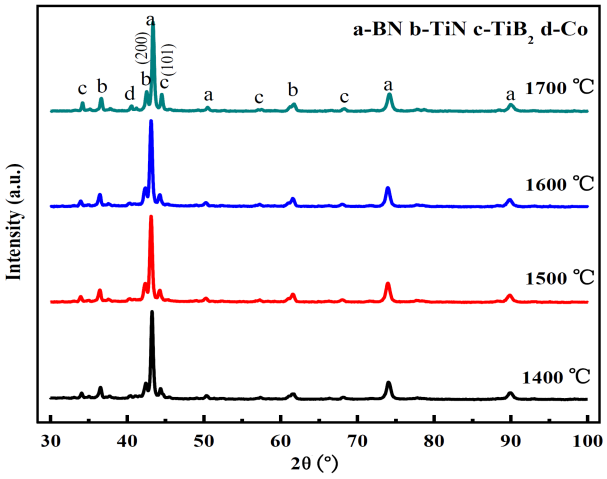

Figure 1. XRD of the PcBN samples at different sintering temperatures

The XRD patterns reveal that the diffraction peaks of TiN from the (200) crystalline plane and TiB_2 from the (101) crystalline plane increase as the sintering temperature rises. It was already indicated [21–23] that the high wear resistance, excellent thermal stability and high hardness of TiB_2 and TiN can contribute to the enhancement of the toughness and strength of the PcBN composites. The XRD patterns indicate that only one diffraction peak of Co is detected due to the small quantity of added Co. The addition of Co can significantly improve the electrical conductivity of the PcBN samples, thus facilitating the subsequent wire-cutting process.

3.2. Microstructure analyses

SEM analysis of the cross sections of the PcBN samples sintered at various temperatures, as shown in Fig. 2, reveals that the distribution of cBN grains and binders appears uniform. There are smooth and stepped surfaces of the cBN grains, which are tightly bound to a uniformly distributed binder. Figures 2a and 2b illustrate that under lower temperature sintering conditions there is a small amount of liquid phase, resulting in challenges for inter-particle diffusion due to their high melting points and limited fusibility. This results in poor sintering performance of the samples, characterized by a relatively porous internal structure (as shown by ellipses in Fig. 2). Simultaneously, the bonding between the cBN grains and the binders is weak. As the synthesis temperature increases, particle diffusion is enhanced, leading to an increase in the liquid phase. The liquid phase can penetrate between the cBN grains more effectively, expelling air pores. In conclusion, the reaction between the binder and cBN under high temperature and ultra-high pressure can achieve densification of PcBN material, improve its internal structure and enhance its performance.

Figure 3 illustrates a gradual decrease in porosity of the PcBN samples with increasing sintering temperature. At 1700 °C, the sample exhibits the lowest porosity (only 0.98%). Conversely, the sample synthesized at 1400 °C shows the highest porosity, i.e. 2.2%. Evidently, elevating the sintering temperature promotes the sintering of composites and enhances densification. The original powder contains titanium metal, and it can be observed by XRD that titanium metal completely re-

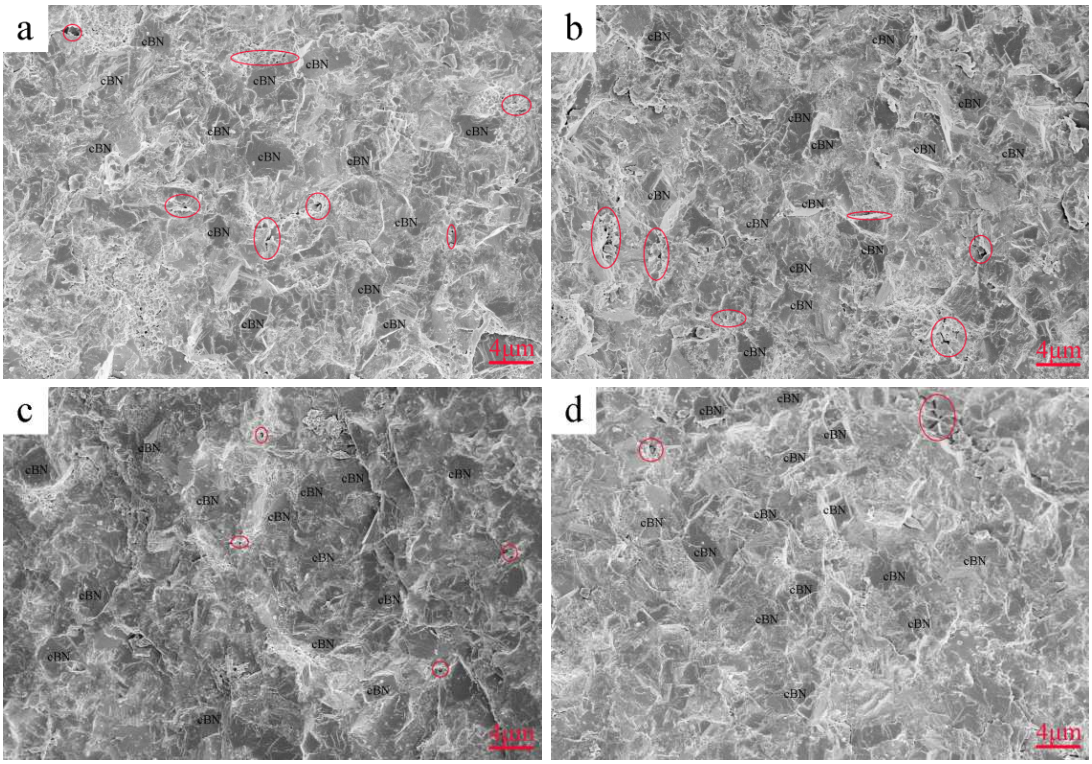

Figure 2. SEM of the PcBN samples sintered at different temperatures: a) 1400 °C, b) 1500 °C, c) 1600 °C and d) 1700 °C

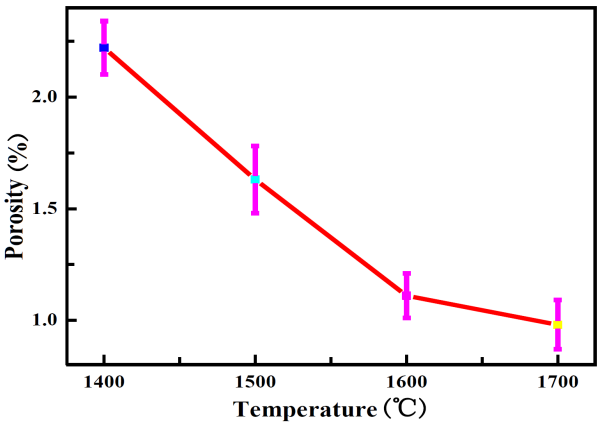

Figure 3. Porosity of PcBN samples at different sintering temperatures

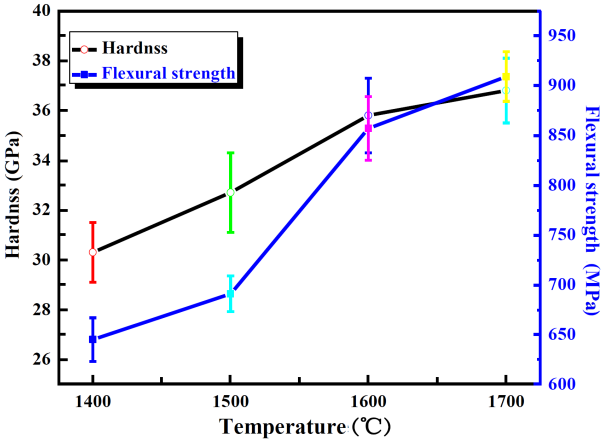

Figure 4. Mechanical properties of PcBN samples at different sintering temperatures

acted with cBN and compounds such as TiN and TiB_2 were formed when the sintering temperature reaches 1400 °C. The samples experienced increased shrinkage due to the combined effects of ultra-high pressure and high temperature. With increasing temperature, the internal bonding within the sample strengthens, enhancing the inter-grain bonding and leading to improved distribution uniformity of the PcBN phase. At high temperatures, a liquid phase fills in between the cBN grains promoting the chemical reaction between titanium and cBN to generate new phases. At the same time, liquid phase can effectively bond the cBN grains and make the internal structure of the sample more dense. Therefore, the reaction between titanium metal and cBN facilitated the formation of PcBN materials during the sintering process under ultra-high pressure and high temperature conditions. The formation of a new phase and the development of a dense structure enhanced the performance and stability of PcBN.

3.3. Hardness and flexural strength analysis

The Vickers hardness of the PcBN samples sintered at different temperatures was tested and the results are shown in Fig. 4. It can be seen that the hardness of the samples increases as the temperature increases from 1400 to 1700 °C. The hardness of ceramic materials is usually influenced by the density and grain size.

Higher density and smaller grain size are associated with higher hardness. This is because high densities provide more atomic bonding sites, while small grain sizes limit the movement of dislocations, making the material more difficult to deform and scratch. It is important to note that in addition to density and grain size, the composition and structure of ceramic material also have an effect on hardness. In this experiment, lower densities have the PcBN composites sintered at lower temperatures. Low densification results in the material containing more pores and defects, which may act as stress concentration points, leading to reduced overall hardness and strength of the material and making it more susceptible to rupture or damage under stress. High densification, on the other hand, results in the material exhibiting low porosity and being tightly bonded to each other. This usually results in increased material hardness due to reduced defects and pores, and the material can withstand higher external stresses without deformation or damage. Overall, there is minimal variation in grain size at different synthesis temperatures, so densification is regarded as the primary factor influencing the hardness of composites. The Vickers hardness of the samples increased from 30.3 to 36.8 GPa as the sintering temperature increased.

The three-point flexural strength of the PcBN samples at different temperatures was also evaluated. According to Fig. 4, it can be seen that the flexural strength of the samples shows an increasing trend as the temperature increases from 1400 to 1700 °C. The PcBN composites exhibited the highest flexural strength of 909 MPa when sintered at 1700 °C. Density, grain size and the characteristics of grain boundary phases are the main factors influencing the flexural strength of the PcBN materials sintered at different temperatures under ultra-high pressure. Examination of the cross-section SEM micrographs in Fig. 2 reveals that the fracture morphology of the cBN-Ti-TiN-Co composites remained relatively consistent despite the rising synthesis temperature, with fracture modes predominantly characterized by along-grain fractures of boron nitride grains and binder particles. Thus, the enhanced densification observed in this experiment offers greater benefits for enhancing the mechanical properties. Analysis of porosity revealed that lower sintering temperatures lead to increased porosity and decreased composite density. Additionally, the SEM images indicate that the sizes of both cBN grains and the primary binders (TiB_2, TiN) exhibit minimal change with increasing synthesis temperature.

Table 2 presents a comparison of the mechanical properties of several reported PcBN composites with the findings of this study. The analysis indicates that the PcBN prepared in this study exhibits favourable mechanical properties, including high flexural strength and

Table 2. Comparison of mechanical properties of reported PcBN composites with the results of this work

Raw material composition	Sintering conditions	Hardness [GPa]	Flexural strength [MPa]	Reference
cBN-TiC-Al-Co	1500 °C/5.8 GPa	36.5	710	[3]
cBN/Ti_3AlC_2/Al	1450 °C/5.5 GPa	48.8	586.22	[8]
cBN/PSN/Al	1450 °C/5 GPa	25.2	602	[9]
cBN/AlN/ Al/Ni	1500 °C/5 GPa	44	661	[13]
cBN/Ti/Al	1300 °C/5.5 GPa	25.8	840.39	[14]
cBN/Ti_3AlC_2	1350 °C/5.5 GPa	33.8	422.45	[15]
cBN/hBN/SiCw	1450 °C/6 GPa	36.5	687.4	[16]
cBN/Si_3N_4/Al_2O_3/Al	1550 °C/5.5 GPa	49.2	610	[20]
cBN/Al/Zr	1600 °C/5.5 GPa	33.4	864.2	[21]
cBN/TiN-Ti-Co	1700 °C/6 GPa	36.8	909	This work

Table 3. Cutting test results of PcBN tools

Cutting tool	Number of parts	Cutting parameters
M-T1 (1400 °C)	72	
M-T2 (1500 °C)	136	Vc = 115 m/min
M-T3 (1600 °C)	198	Ap = 0.05 mm
M-T4 (1700 °C)	222	f = 0.1 mm/r
M-5 (Other model)	202	

microhardness in comparison to other PcBN composites. Thus, the prepared PcBN composites have promising potential for applications in cutting machining.

3.4. Cutting experiment

The DNMA150404 type cutting tools were fabricated from the PcBN materials T1, T2, T3 and T4, and labelled as M-T1, M-T2, M-T3 and M-T4, respectively (Table 3). The prepared tools were used for cutting test involving workpiece made of the 40Cr mechanical parts with a hardness of 52–58 HRC. Comparative experiments were conducted concurrently using the PcBN tools from the top international brand (labelled as M-5).

The quantity of 40Cr mechanical parts, machined under identical cutting conditions, served as the performance evaluation metric for all tested PcBN tools. Only 72 40Cr mechanical parts were machined using the M-T1 tool fabricated from the PcBN sintered at 1400 °C. This is the lowest amount among all experiments and is attributed to the low synthesis temperature resulting in inferior mechanical properties and consequently poor cutting performance. As the sintering temperature increased, the quantity of machined parts rose. Thus, 222 40Cr mechanical parts were machined using the M-T4 tool. The cutting results indicate a correlation between the cutting performance of the tool material and its mechanical properties. Superior mechanical properties lead to excellent cutting performance of the corresponding tool. In a comparative study, the first-tier brand processed 202 40Cr mechanical parts, slightly fewer than that of the M-T4. This confirmed superior performance of the cutting tool fabricated from the T4 PcBN composite sintered at 1700 °C with the tested 40Cr mechanical parts.

IV. Conclusions

The cBN-TiN-Ti-Co composites were prepared using high temperature and high pressure sintering at a pressure of 6 GPa and a temperature ranging from 1400 to 1700 °C. The prepared PcBN composites are primarily comprised of BN, TiN, TiB_2 and Co. With increasing temperature, the pores inside the samples gradually decrease, resulting in increased structural density.

The PcBN tool material prepared at the sintering temperature of 1700 °C exhibited improved performance in terms of flexural strength and hardness, with a flexural strength of 909 MPa and a microhardness of 36.8 GPa. The cutting tool fabricated from the PcBN composite sintered at 1700 °C demonstrates good cutting performance and machinability, making it suitable for processing of mechanical parts made from 40Cr and related materials.

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