



In situ synthesis of cBN–Ti₃AlC₂ composites by high-pressure and high-temperature technology

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ABSTRACT

In this paper, titanium aluminum carbide (Ti₃AlC₂) was studied as the binder of polycrystalline cubic boron nitride (PCBN) to overcome the weaknesses of traditional metal binders and ceramic binders. The composites of cubic boron nitride (cBN) and Ti₃AlC₂ were in situ synthesized from the mixtures of Ti, Al, TiC and cBN powders at high temperature (1050 °C–1250 °C) and under high pressure (4.5 GPa) (HTHP). The Ti₃AlC₂ proportion in as-prepared composites increased as the processing temperature increased from 1050 °C to 1150 °C. At 1200 °C, part of Ti₃AlC₂ decomposed to form new TiC. At 1250 °C, cBN transformed to hBN and reacted with other materials. The composites processed at 1200 °C had the optimal mechanical properties, including friction coefficient, hardness and mass loss of worn Si₃N₄ ball. From the microstructure of as-prepared composites, agglomeration of cBN particles existed; however, the cBN particles, in general, were evenly distributed in the matrix of Ti₃AlC₂. The cBN particles strongly bonded with the matrix with a clear interface.

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1. Introduction

Polycrystalline cubic boron nitrides (PCBN) are superhard materials with excellent properties and wide applications [1–3]. Generally, these superhard materials are prepared by high-pressure and high-temperature (HTHP) technology [4]. PCBNs consist of cubic boron nitride (cBN) particles and binder. The binder is a key factor to control the performance of PCBNs.

Usually, there are two kinds of binders. One is metal [4–7] and the other is ceramic (vitrified) [7–10]. Metal binders, such as Co or Al [4,6,7], possess good room-temperature mechanical properties, but are easy to be soft and oxidized at high temperature caused by cutting process. Ceramic binders, such as TiC or TiN [7,10], have excellent high-temperature mechanical properties and oxidation resistance, but their toughness is bad and these materials are easily broken at room temperature. To overcome the weaknesses of traditional metal binders or ceramic binders, someone chose the mixture of TiC and Al as the binder of PCBN [11,12]. If a material combines the advantageous properties of ceramics and metals, it can be used as a binder to make remarkable superhard materials. Ti₃AlC₂ is a ternary carbide with these unique properties [13–16]. Thus, it is a promising candidate as a binder to make PCBNs with high performance. Ti₃AlC₂ was first found in the ternary system of Ti–Al–C in 1994 [17]. The bulk of Ti₃AlC₂ was first made by hot isostatic press in 2000 [15]. It has similar crystal structure with another Ti–Al–C compound, Ti₂AlC, and the two compounds

always coexist. Ti₃AlC₂ has a same crystal structure with an intensively researched carbide Ti₃SiC₂ [18] except that Al is replaced by Si.

Although there is no report on Ti₃AlC₂ as the binder of PCBN, the compounds with similar crystal structure (Ti₂AlC or Ti₃SiC₂) were used as the binder by accident or in purpose. In the research of Ueda et al. [19], the mixture of Al and TiC/TiN was used as the binder to make PCBN composites by HTHP. Ti₂AlC, Ti₂AlN and Ti₂AlCN phases were accidentally found in bonding phases. Benko et al. [20] made cBN–Ti₃SiC₂ composites by high-pressure hot pressing. The starting materials, hexagonal boron nitride (hBN) particles and Ti₃SiC₂ powders, were mechanically mixed in ethanol and sintered under 7 GPa at 1750 °C to make cBN–Ti₃SiC₂ composites. Based on these previous studies, it is possible for Ti₃AlC₂ to be a good PCBN binder and it is significant in this kind of research.

Therefore, for the purpose of making novel superhard materials, in this paper, we made the attempt to in situ synthesized cBN–Ti₃AlC₂ composites by HTHP. The composition, microstructure and mechanical properties of as-prepared composites were studied.

2. Experimental procedure

Starting powders of Ti (325 mesh, 99.3 wt.% pure, Jinzhou Haotian Titanium Co., China), Al (200 mesh, 99.99 wt.% pure, Henan Yuanyang Aluminum Industry Co., China), C (graphite, 325 mesh, 99 wt.% pure, Henan Huaxiang Carbon Powder Technology Co., China), TiC (200 mesh, 99.8 wt.% pure, Aladdin Reagent Co., China) and cBN particles (20–30 μm, XingyangshiNew Source Chemical Co., China) were employed in this research. At the beginning, four recipes with different mole ratio (Ti/1.2Al/2TiC, Ti/1.2Al/TiC, 2Ti/1.2Al/C, 3Ti/1.2Al/2 C) were

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applied to synthesize Ti_3AlC_2 matrix. Then, according to experimental results, the mole ratio was adjusted to get the best recipe. The starting materials were weighed and mixed by an airport star mixing machine for 4 h. Thereafter the mixtures were mixed with cBN particles in the volume ratio of 1:1. The mixtures were then compacted in a steel mold into compacts with diameter of 12 mm and height of 3 mm. cBN- Ti_3AlC_2 samples were prepared in a cubic anvil high-pressure apparatus (SPD 6×1200, Zhengzhou Abrasive Grinding Research Institute, China) under 4.5 GPa at 1050–1250 °C. It took ~3 min to increase temperature and pressure. All samples were processed at different temperatures for 10 min. The heating electrical power was cut off and the pressure was released in ~15 min. The processing procedure is shown in Fig. 1.

X-ray diffraction (XRD, Brukeraxs Co., Germany) with $\text{Cu K}\alpha$ radiation at 40 kV and 40 mA was applied to check phase compositions. Collected samples were examined by scanning electron microscopy (SEM, JSM-6390LV, JEOL, Japan) equipped with energy dispersive spectroscopy (EDS, INCA-ENERAGY 250, Oxford, UK) to reveal the microstructure and chemical composition. Hardness of the materials was measured under a load of 60 kg with the dwell time of 10 s using a Brinell hardness tester (BH, 500MRA, Walter Bert Measuring Instrument Co., China). The hardness tests were performed at least 20 times and the mean value and its changing range were given; the test deviation was no more than 10%.

The friction tests were carried out on a room-temperature tribometer with a pin-on-disk configuration (CFT-I Material Surface Performance Comprehensive Test Instrument, Zhongke Kaihua Instrument Equipment Co., China). The specimens were fixed in a square disk made of WC-based cermet with a size of 19×18.7 mm and a surface roughness of approximately 0.1 mm. The pin was Si_3N_4 ball with the size of Φ 4 mm. Both the pin and disk were cleaned ultrasonically in an acetone bath and allowed to dry prior to testing. The test was carried out under the condition as follows: trip for 5 mm in the dry friction condition reciprocating sliding, a velocity of 360 revolutions per minute, a load of 20 N, and a sliding time of 30 min at room temperature. Friction coefficients were recorded continuously by computer during the test. Abrasion losses were estimated by measuring the mass loss of Si_3N_4 balls. After friction test, samples were checked again by SEM to reveal the microstructure of worn surface.

3. Results

Fig. 2 shows XRD patterns of samples made from mixture powders of $\text{Ti}/1.2\text{Al}/2\text{TiC}/\text{cBN}$, $\text{Ti}/1.2\text{Al}/\text{TiC}/\text{cBN}$, $2\text{Ti}/1.2\text{Al}/\text{C}/\text{cBN}$ and $3\text{Ti}/1.2\text{Al}/2\text{C}/\text{cBN}$. The processing pressure and temperature were 4.5 GPa and

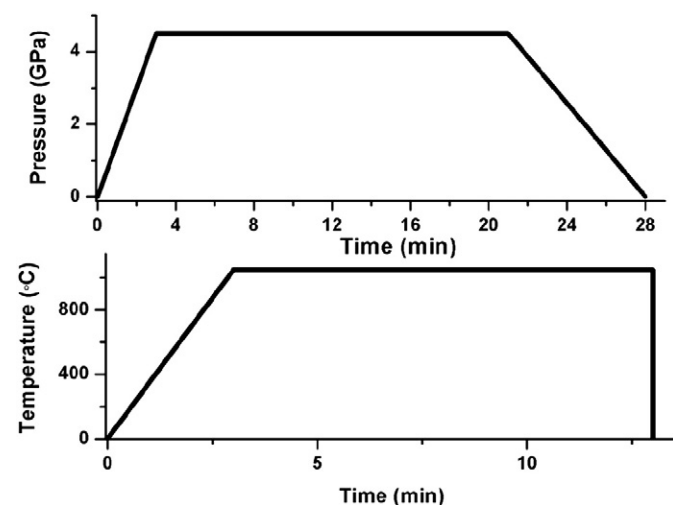


Fig. 1. Experimental HTHP procedure curve.

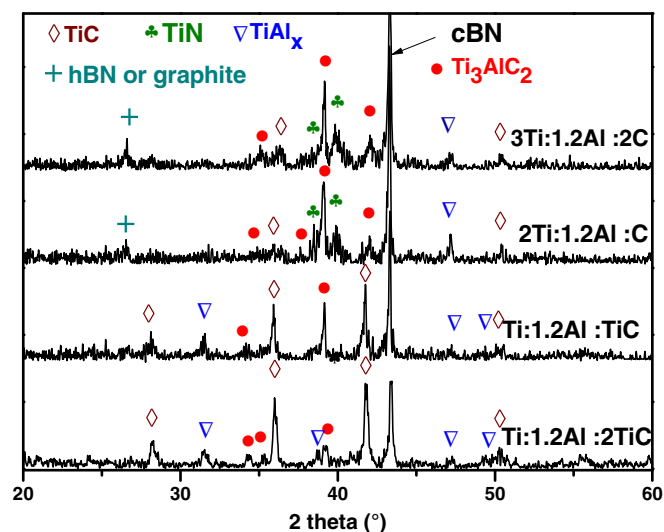


Fig. 2. XRD patterns of Ti_3AlC_2 -cBN composites from different starting materials at 1050 °C.

1050 °C, respectively. As shown in Fig. 2, cBN and Ti_3AlC_2 peaks are noticeable. This is true that Ti_3AlC_2 can be synthesized under ~4.5 GPa and cBN- Ti_3AlC_2 composites can be in situ prepared by HTHP. The bottom two samples with TiC as C source have weaker Ti_3AlC_2 peaks and stronger TiC peaks. Additionally, the top two samples with graphite as C source have stronger Ti_3AlC_2 peaks, weaker TiC peaks, some TiN peaks and a peak at 26.5°. The 26.5° peak can be attributed to either unreacted graphite or hBN formed from the phase transformation of cBN at high temperature.

According to the results of Fig. 2, the synthesis of Ti_3AlC_2 under 4.5 GPa is possible for all starting materials. However, the graphite or hBN appeared if graphite powders were chosen as C source. Compared with TiC, both graphite and hBN are undesirable in superhard materials. Therefore TiC powders were used as C source in the following experiments. However, from Fig. 2, strong TiC peaks exist at the XRD pattern of the sample from $\text{Ti}/1.2\text{Al}/2\text{TiC}/\text{cBN}$. Thus it was decided to reduce the content of TiC and increase that of Ti. XRD patterns of cBN- Ti_3AlC_2 samples synthesized from $2\text{Ti}/1.2\text{Al}/\text{TiC}/\text{cBN}$ under 4.5 GPa at different temperatures are shown in Fig. 3. Ti_3AlC_2 (and/or Ti_2AlC) was synthesized at 1050 °C for 10 min. TiAl_x and TiC were other phases in the synthesized samples. Higher processing temperature increased the content of Ti_3AlC_2 . At ~1150 °C, Ti_3AlC_2 was the main phases besides cBN in the prepared composites. Higher processing temperature to 1250 °C introduced an impurity TiN and a peak at ~26.5°. Same as Fig. 2, the 26.5° peak can be attributed to either graphite or hBN. And the peak increases with temperature. If that peak belongs to residue unreacted graphite, it should decrease rather than increase with temperature. Also, the decompose product of Ti_3AlC_2 at high temperature is TiC rather than graphite. Therefore, it is more reasonable to assign the peak to hBN. The content of hBN increases with increasing temperature because more cBN transform to hBN at high temperature with the existence of Ti_3AlC_2 and TiC.

The 26.5° peak at Fig. 2 is also can be reasonably assigned to hBN by this explanation. If graphite is used as a starting material, the exothermal reaction between Ti and C can generate a lot of heat in short time and local temperature is increased to induce the phase transformation of CBN to hBN. Therefore, it is more reasonable to attribute the 26.5° peak at Figs. 2 and 3 to hBN though we cannot rule out the possibility that it belongs to graphite.

For superhard materials based on cBN, the measurement of most mechanical properties is extremely difficult and often even impossible because of small sizes of the samples as well as their small cylindrical shape (12-mm diameter). In this paper, tribological properties and

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