



Sintering behaviors of fine-grained cBN–10 wt.% Al_{3.21}Si_{0.47} system under high pressure

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ABSTRACT

Sintering behaviors of fine-grained cBN–10 wt.% Al_{3.21}Si_{0.47} system under high pressure of 5.0 GPa and temperatures up to 1600 °C were investigated. By analyzing the phase components of sintered samples through x-ray diffraction (XRD), and detecting the in-situ electrical resistance signal during high pressure and high temperature (HPHT) sintering, we suggested the reactions between cBN and Al_{3.21}Si_{0.47} happened at about 600 °C. Between 600 °C and 900 °C, AlN, AlB₂ and Si formed, while above 1000 °C, the content of AlN, AlB₂ and Si phases increased with AlB₂ disappearing. Scanning electron microscope (SEM), Vickers hardness tests as well as cutting performance tests showed that the well-sintered samples in cBN–10 wt.% Al_{3.21}Si_{0.47} with homogeneous microstructure and best mechanical performance could be obtained at the sintering P–T condition of 5.0 GPa, 1400 °C for 10–15 min.

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1. Introduction

Cubic boron nitride (cBN) has superior properties, such as high hardness, excellent thermal conductivity and high thermal stability. In addition, it does not react with ferrous materials as a result of its high chemical inertness. Because of those unique properties, cBN is widely used for cutting hardened steel, cast iron, ferrous powder metal and heat resisting alloy [1–3].

The cBN single crystal is difficult to grow up and it is extremely brittle due to anisotropy, while the properties of polycrystalline cubic boron nitride (PcBN) are relatively homogeneous and the cost is low, showing excellent mechanical properties with high hardness and wear resistance suitable for cutting tools [4–6]. As a result, PcBN is widely applied in industrial production. The influence of the grain size of the cBN on the performance of PcBN tool is very important [6–8]. Amanda Mckie shows that the hardness of the PcBN materials increases with decreasing cBN grain size [9]. Kevin Chou and Chris J. Evans indicate that the greater wear resistance and lower wear rates are acquired for PcBN tools with the smaller grain size [10]. According to Hall–Petch relationship [11], the material yield stress and hardness are inversely proportional to the square root of the grain size. So within a certain range, the smaller the grain size, the greater the yield stress and hardness of the material. Grain refinement can improve the material toughness and ductility, which make up the deficiency of the material with high hardness and poor toughness. What's more, the smaller the grain size of cBN, the better is the surface quality of the sintered bodies as well as the impact resistance

and wear resistance, which are conducive to machine the high surface quality of the workpiece [8,10]. So, the cBN grain size of 1–2 μm is used as the starting material in this study.

Fine-grained sintered bodies of cBN can be produced at high pressure and high temperature (HPHT) by the direct transformation from the hexagonal boron nitride (hBN) or sintering of fine-grained cBN grits with or without additives. The former method involves a complicated transformation reaction from hBN to cBN. Moreover, this method requires higher pressure (>7 GPa) and higher temperature (>2000 °C) which make the cost increase for the industrial application [12,13]. The latter method without additives requires the relatively high sintering conditions [14]. Hence in this investigation, sintering of the fine-grained cBN grits with additives is adopted. Conventional binders used for cBN sintering have mainly been metals of the groups IV, V and VI of the periodic table or their compounds. Metallic elements like Ni, Co, Ti, Al and materials which have the catalytic activity for hBN to cBN conversion such as AlN and Si are used as well [15–18]. Adding the appropriate additives to the cBN grits decreases the sintering conditions, including the pressure and temperature [19]. In this study, Al_{3.21}Si_{0.47} is used as the binder in the sintering process, since it has the low melting point and can react with cBN under a wide temperature range, making cBN particles that are bonded by the reaction production to be denser and the wear performance of the sintered bodies improved. However, little information about the fine-grained cBN with Al_{3.21}Si_{0.47} has been reported at present yet. Thus the aim of this study is to extend our current understanding on the sintering behaviors of fine grained cBN with Al_{3.21}Si_{0.47} alloy as well as the properties of the sintered products.

This paper studied the reaction mechanisms and related the chemical as well as mechanical properties of the cBN composites. In order to illustrate the reaction mechanisms, x-ray diffraction (XRD)

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analysis, combining in-situ electrical resistance measurements at different P–T conditions was conducted. Scanning electron microscope (SEM) with energy dispersive spectrometer (EDS) analysis, Vickers hardness and cutting performance tests was also applied to study the sintered samples which were prepared under various conditions.

2. Experimental procedures

2.1. Sample preparation

The cBN powder (grain size: 1–2 μm , Zhengzhouzhongnanjieta Co., Ltd, China) and $\text{Al}_{3.21}\text{Si}_{0.47}$ powder (grain size: 80 nm, purity: 99.9%, Shanghai Chao Wei Nanotechnology Co., Ltd, China) as the starting materials were mixed according to the mass ratio of 9:1. Powders were manually mixed in ethyl alcohol using an agate mortar and pestled for 4 h. In order to remove the ethanol and vapors adsorbed on the powder surface, the mixtures were treated in vacuum of 3.0×10^{-3} Pa and temperature of 500 $^{\circ}\text{C}$ for 1 h.

Afterwards, about 1 g mixture powders were put into zirconium capsule (11 mm in diameter, 5 mm in thickness) and sintered at 5.0 GPa, high temperature at 500 $^{\circ}\text{C}$ –1600 $^{\circ}\text{C}$ for different heat treatment times of 5–20 min, using DS6 \times 8MN cubic press [20]. The sample assembly for the HPHT sintering experiment can be seen elsewhere (see Fig. 1 in [17]). The cell temperature was directly measured with Pt6%Rh–Pt30%Rh thermocouples, and pressure was calibrated with a method of aluminum melting-point [21]. In our experiments, samples were first compressed to a required pressure and then heated to the desired temperature with a heating rate of about 200 $^{\circ}\text{C}/\text{min}$. Because the heating rate was relatively great, there was a certain gradient of temperature in the sample chamber in our experiments. However, the temperature difference between the center part of the sample and the edge part was estimated to be less than 5 $^{\circ}\text{C}$. After keeping for a desired treatment time, the samples were quenched to room temperature with cooling rate of about 70 $^{\circ}\text{C}/\text{min}$ and then decompressed to ambient pressure with 12 min. The well shaped specimens were ground into a wafer of about 9.5 mm in diameter and 4 mm in thickness. Then the samples were polished to a smooth mirror surface by a polishing machine with 10 μm and 1.5 μm diamond pastes.

2.2. Sample characterization

The phase composition of the sintered samples was investigated by the XRD analysis (DX-2500, Dandong, China) and in-situ electrical resistance measurements at different P–T conditions were conducted to monitor the HPHT sintering behavior. SEM (S-4800, Hitachi, Japan) equipped with EDS (IE250, Oxford, England) was carried out to analyze the microstructure. In in-situ electrical resistance measurements, the sample assembly we used can be seen elsewhere [see Fig. 5 in [19]]. A constant current of 10 mA was provided by a constant current source in the measurement circuit. If the electrical resistance of the sample changed, then the voltage drop of the sample would also change. Thus by detecting the voltage signal of the sample, we could get the in-situ electrical resistance changes of our samples during HPHT sintering. The cBN–10 wt.% $\text{Al}_{3.21}\text{Si}_{0.47}$ mixture was used as the starting materials. Because of the non-conduction of the starting materials at room pressure and temperature, a constant resistance of 10 Ω was connected in parallel with the sample to show the tendency of the sample resistance changes obviously. Heating power with a constant increasing rate was controlled by a programmed power controller. The temperature was measured by a Pt6%Rh–Pt30%Rh thermocouple which was put into the sample zone. Thus the temperature and voltage signal across the sample could be monitored simultaneously and separately recorded by a multi-channel data recorder, and we could get the relations between resistance changes of the sample and temperature.

In order to determine the mechanical properties of the sintered samples, Vickers hardness and cutting performance tests were designed to measure. Vickers hardness of the polished samples was tested by a Vickers hardness tester (FV-700B, Future-Tech, Japan) with 29.4 N of applied load and 15 s dwelling time. Moreover, the sintered bodies were polished to a cutting-tool shape with 0.2 mm \times 20 $^{\circ}$ chamfer for cutting tests so as to evaluate the cutting performance. The tests were conducted on a numerically controlled lathe (SK50P/750, Baoji, China). Tool holders were CRSNR2525M09. The tool wear was gauged by a stereomicroscope (XTL-3400, Shanghai, China). The hardened steel with the hardness at about 62 HRC was used as the working piece. The testing parameters were as follows: 120 m/min of cutting speed, depth of cut of 0.15 mm, feed of 0.15 mm/rev. No lubricant or coolant was used during turning.

3. Results and discussion

3.1. Investigate sintering mechanisms by XRD and in-situ electrical resistance measurement

According to the XRD patterns of the samples sintered at 5.0 GPa, and different temperatures, with heating times of 15 min in Fig. 1, the sintered bodies are composed of different phases at different temperatures. At 5.0 GPa, 500 $^{\circ}\text{C}$ for 15 min, the major phases detected by XRD were cBN and $\text{Al}_{3.21}\text{Si}_{0.47}$ [Fig. 1(b)]. When temperature increased to 600 $^{\circ}\text{C}$, phase components changed to be cBN, AlN, AlB_2 , Si and $\text{Al}_{3.21}\text{Si}_{0.47}$. When the temperature was set from 700 $^{\circ}\text{C}$ to 900 $^{\circ}\text{C}$, the major phases of the samples were still cBN, AlN, AlB_2 , Si and $\text{Al}_{3.21}\text{Si}_{0.47}$, but the amount of $\text{Al}_{3.21}\text{Si}_{0.47}$ decreased and AlN, AlB_2 and Si increased [Fig. 1(c)–(f)]. When we continue increasing the temperature from 1000 $^{\circ}\text{C}$ to 1600 $^{\circ}\text{C}$, AlB_2 and $\text{Al}_{3.21}\text{Si}_{0.47}$ disappeared, and cBN, AlN and Si were the final phases [Fig. 1(g)–(i)].

We deduce the possible reaction mechanisms between $\text{Al}_{3.21}\text{Si}_{0.47}$ and cBN according to XRD data. At 500 $^{\circ}\text{C}$, the major components of the samples detected by the XRD were cBN and $\text{Al}_{3.21}\text{Si}_{0.47}$ which revealed $\text{Al}_{3.21}\text{Si}_{0.47}$ and cBN did not react at such P–T condition. Until temperature increased up to 600 $^{\circ}\text{C}$, reactions happened and AlN, AlB_2 and Si formed. We speculate the possible chemical reaction at 600 $^{\circ}\text{C}$ and temperatures up to 900 $^{\circ}\text{C}$ may proceed as follows:

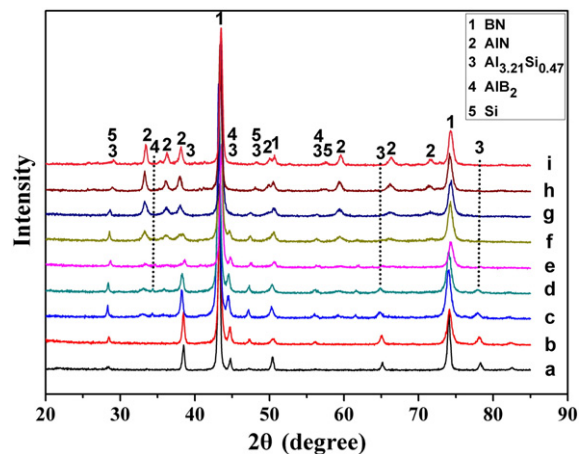
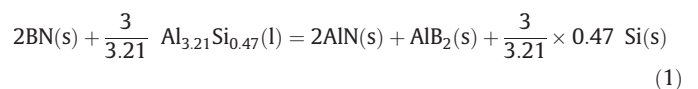


Fig. 1. X-ray diffraction patterns of starting materials (a) and the sintered samples from the cBN–10 wt.% $\text{Al}_{3.21}\text{Si}_{0.47}$ mixture at: (b) 500 $^{\circ}\text{C}$; (c) 600 $^{\circ}\text{C}$; (d) 700 $^{\circ}\text{C}$; (e) 800 $^{\circ}\text{C}$; (f) 900 $^{\circ}\text{C}$; (g) 1000 $^{\circ}\text{C}$; (h) 1400 $^{\circ}\text{C}$ and (i) 1600 $^{\circ}\text{C}$ under 5.0 GPa for 15 min.

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