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Mechanical properties of nano-polycrystalline cBN synthesized by direct conversion sintering under HPHT

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ABSTRACT

Using hBN and pBN as starting materials, various types of binderless polycrystalline cBN (BL-PcBN) were synthesized in the pressure range of 8–20 GPa and temperature range of 1300–2400 °C, and their mechanical properties were evaluated. In the synthesis pressure range of 10 GPa and higher, the hardness of BL-PcBN showed a correlation not with the synthesis pressure, but with the synthesis temperature. Binderless polycrystalline cBN synthesized at about 2200 °C exhibited the highest mechanical properties, for both starting materials. Specifically, BL-PcBN(h) (100–300 nm grain size) synthesized from hBN at 10 GPa and 2200 °C showed a hardness of 45 GPa, transverse rupture strength of 1.6 GPa. In contrast, BL-PcBN(p) synthesized from pBN at the same temperature had finer grain size (50–100 nm) and exhibited the same level of hardness but lower strength properties (transverse rupture strength of approx. 1.3 GPa) than BL-PcBN(h). Consequently, the material that exhibited the best mechanical properties was BL-PcBN(h) synthesized at 10 GPa and 2200 °C. A prototype micro ball end mill made of this material was examined in a mirror-like (polished-like) finishing test using high-strength hardened steel. This ball end mill achieved a fine finishing surface with a surface roughness (Ra) of 20 nm or better. The test revealed the high potential of this material for use as a high-precision cutting tool for high strength ferrous materials.

1. Introduction

Cubic boron nitride (cBN), the second hardest material after diamond, is highly thermally and chemically stable and is, compared with diamond, less reactive with ferrous materials. It is therefore used for cutting tools for ferrous materials. The sintered cBN used today for the cutting tools is manufactured by sintering micrometer cBN particles under high-pressure and high-temperature conditions of 5–6 GPa and 1300–1400 °C using a metal or ceramic material as the binder. When sintering, cBN particle size, binder components and their amounts are adjusted for specific applications since these parameters affect cutting performance such as wear and chipping resistance. Meanwhile, one method of obtaining polycrystalline single-phase cBN without using a binder is by directly converting and simultaneously sintering from hexagonal boron nitride (hBN) or CVD-based pyrolytic hBN (pBN) into cBN under ultra-high pressure and temperature [1–11]. The combination of optimized starting material and synthesis conditions enable the synthesis of binderless polycrystalline cBN (BL-PcBN) in which micrograins are directly and strongly bound together. In previous studies, using hBN as a starting material, we synthesized BL-PcBN with a grain size of 0.1–0.5 μm through direct conversion sintering at 7.7 GPa and 2300 °C and showed that this material has higher hardness, heat resistance and thermal conductivity than conventional cBN as well as excellent cutting performance in high-speed milling of ferrous materials [7,8]. With finer grains, this BL-PcBN material would produce a sharper and stronger edge on cutting tools for high-precision machining of hard ferrous materials, which has been difficult conventionally. In recent years, there has been a growing need for precision cutting of molds made of high-strength hardened steel specifically for the manufacture of electric/electronic and optical components; hence, there is a growing demand for BL-PcBN with a finer and denser structure and increased strength as described above. To meet this need, we strived to find a material that has a finer grain size and offers increased hardness, strength and toughness. In this study, we prepared various pieces of prototype BL-PcBN under an extended range of synthesis conditions of 8–20 GPa and 1300–2300 °C and evaluated their microstructures and mechanical properties. Furthermore, we constructed a micro ball end mill made of BL-PcBN with excellent mechanical properties revealed by the evaluation results, conducted a precision micromachining test on high-strength hardened steel, and examined the potential of the BL-PcBN material as a high precision cutting tool for ferrous metals.

2. Experiments

High-purity isotropic hBN and pyrolytic BN (pBN) compacts were used as starting materials. The hBN compact was formed through molding process and high-level purification from highly crystalline

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hBN particles of size 1–3 μm. The oxygen impurity level and density of the hBN compact were 0.01% (detection limit, measured by an inert gas fusion-infrared absorptiometry) or less and 1.8 g/cm^3, respectively. Regarding pBN compacts, three types (pBN-1, pBN-2 and pBN-3) prepared through CVD-based vapor-phase synthesis were used. They all had an impurity level of 0.01% or less and density of 1.9–2.1 g/cm^3. Their crystallinity levels (degrees of graphitization) were in the order of pBN-1 > pBN-2 > pBN-3. Fig. 1 shows the X-ray diffraction patterns of these starting materials.

These starting materials were directly converted and sintered to cBN through 20-min exposure to a pressure of 8–20 GPa and a temperature of 1300–2400 °C using a Kawai-type multi-anvil apparatus to produce various BL-PcBN specimens. The specimen sizes were 6–11 mm in diameter and 8–11 mm in thickness.

The crystalline phase of each prepared BL-PcBN specimen was identified by X-ray diffraction measurements, and their structures were observed by a high resolution (HR) SEM. The specimens were evaluated in terms of their basic properties of hardness and transverse rupture strength (TRS), which are important for cutting tool applications. Specimen hardness was measured using a micro hardness tester with a Knoop indenter, applying a load of 4.9 N for 15 s. Hardness was determined as the average of five measurements. The Vickers indenter was also tried to be used for the hardness testing, but the results were not adopted, since a trial test proved that it did not lead to accurate assessments due to a high level of elastic recovery and cracks of some specimens. TRS was measured by the three-point bending test method with specimens processed into a size of 6 × 3 × 1 mm with the span of 4 mm.

Moreover, a specimen having good mechanical properties was selected from the BL-PcBN materials and was used to prepare a micro ball end mill with a 0.5-mm edge radius. Using this as a cutting tool on a precision machining center, 5 × 5 mm mirror finish surfaces were produced on high-strength hardened steel ELMAX (HRC = 60) inclined at an angle of 45° under wet condition. The cutting conditions were: n = 60,000 rpm, Vf = 200 mm/min, Ap (cutting depth) = 5 μm, and Pf = 3 μm. The surface roughness of the machined surface was evaluated with a non-contact optical scanning white light interferometer (ZYGO). After cutting, edge wear was observed by SEM and damage mechanisms were examined.

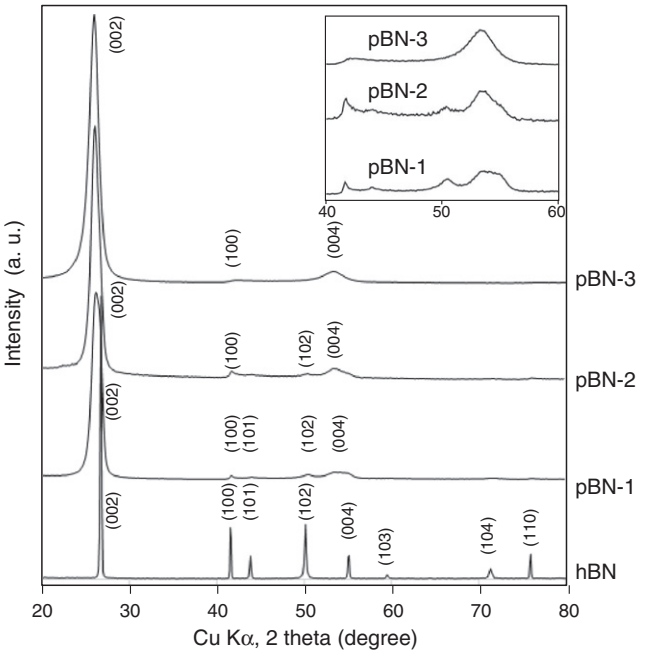

Fig. 1. X-ray diffraction patterns of starting materials.

3. Results and discussion

3.1. Synthesis conditions and microcrystalline structures

In Fig. 2 showing the pressure and temperature conditions, the line (1) represents points where starting materials hBN and pBN begin converting to cBN or wBN, the line (2) represents points where polycrystalline cBN and wBN are produced with no residual hBN, and the line (3) represents points where the starting materials are completely (100%) converted to cBN [11]. Fig. 3 shows typical X-ray diffraction patterns of prepared BL-PcBN specimens from hBN at various conditions. The results shows hBN and pBN begin converting to cBN under almost the same conditions. The conditions under which they were fully converted to cBN were also almost the same. Observation of these structures reveals that the size of constituent grains is correlated with the synthesis temperature rather than with the pressure. From hBN, polycrystalline bodies made of fine grains 300 nm or less in average grain size are produced at 2100 °C or below and those made of fine grains 100 nm or less in average grain size are produced at 1900 °C or below. The grain size increases with increasing temperature. Beyond 2300 °C, grain growth is accelerated and there are many grains exceeding 1 μm in size. Also, a lamellar structure occurs, which is similar in shape to the lamellar structure of nano-polycrystalline diamond (NPD) [12] directly converted from graphite. It is believed that both lamellar structures are formed through similar processes (hBN → wBN → cBN diffusionless phase transformation). It should also be noted that at a synthesis temperature of 1900 °C or below, residual wBN occurs. The amount of residual wBN increases with increasing pressure and decreasing temperature. Under conditions of 20 GPa and 1300 °C, the proportion of wBN was 100% [11]. In contrast, polycrystalline bodies made of finer grains (50–100 nm) are produced from pBN. The slightly lower level of wBN formation than in the case of hBN and the absence of noticeable lamellar structures suggest that diffusive phase transformation is dominant in the conversion process of starting material pBN. Nonetheless, from pBN, polycrystalline bodies containing approximately 50% wBN are formed in the low-temperature range under an ultrahigh pressure of 20 GPa [11].

3.2. Hardness properties

In the following sections, BL-PcBN(h) and BL-PcBN(p) denote BL-PcBN produced from hBN and pBN, respectively. Figs. 4 and 5 show the hardness measurement results respectively for BL-PcBN(h) and

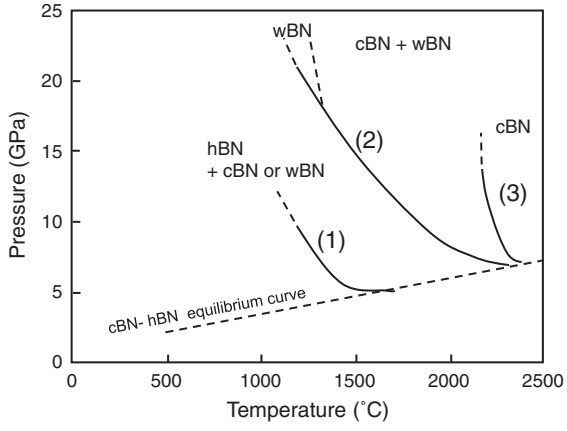

Fig. 2. Experimental results of high-pressure and high-temperature tests of direct conversion from hBN and pBN to cBN or wBN. Line (1): starting points where hBN and pBN begin converting to cBN or wBN, line (2): starting points where polycrystalline cBN are produced with no residual hBN, line (3): starting points where the starting materials completely (100%) convert to cBN.