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Development of matrix microstructure in polycrystalline cubic boron nitride ceramics

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ABSTRACT

The development of matrix microstructure in polycrystalline cubic boron nitride cutting tool materials has been investigated by X-ray diffraction and scanning and transmission electron microscopy combined with energy dispersive X-ray spectrometry. The materials had a matrix based on Ti(C,O,N) and Al and were fabricated from powder mixtures milled with either WC-Co cemented carbide or (Ti,W)(C,N)-Co cermet milling media. The introduction of WC-Co debris resulted in the formation of an Al- and W-rich liquid phase during high pressure high temperature sintering. Crystalline intergranular phases rich in W partitioned from this liquid. Debris from the cermet milling media resulted in the formation of intergranular Co₂B; the W was retained in the (Ti,W)(C,N) structure during sintering. Al-rich reaction zones on cBN grain surfaces developed locally during high pressure high temperature sintering.

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1. Introduction

Cutting tool materials made of polycrystalline cubic boron nitride (PCBN) combine high hardness with adequate fracture toughness, high chemical and thermal stability and high thermal conductivity. PCBN ceramics are composite materials containing hard cubic boron nitride (cBN) grains embedded in a multiphase matrix. Titanium carbonitride with a varying C/N ratio, Ti(C_xN_{1-x}), is generally a major constituent of the matrix in materials fabricated with a cBN content of around 50 vol% [1–3].

The powder metallurgical process used in the fabrication of dense PCBN materials involves high temperature (around 1500 °C) and high pressure (around 5 GPa) sintering [2,4,5]. Aluminium or aluminium compounds are usually added to the starting powder mixture in order to promote the densification. The PCBN starting powder mixture will also contain debris from the milling media. The debris will, together with the additives, affect the chemical composition of the PCBN matrix and the development of the material's microstructure. It has been shown that the debris and additives will be involved in the formation of reaction zones between the cBN grains and the matrix phases [6,7]. Angseryd et al. [6] identified an Al₂O₃ network around and between the cBN and Ti(C,N) matrix grains. This network was suggested to be the result

of a reaction between the added Al and the surface oxides on the starting powder particles. AlN was frequently observed adjacent to the Al₂O₃ network [6]. It is believed that the morphology and distribution of the reaction products will have a strong influence on the properties of the sintered PCBN material [6,8].

Different techniques have been adopted in order to control the development of the PCBN microstructure and to improve the homogeneity of the material. Umer et al. coated the cBN starting powder particles with nanocrystalline TiN [9]. This resulted in a homogeneous microstructure containing an intergranular network rich in Ti and Al. The material had a higher hardness and an improved transverse rupture strength when compared with a PCBN material of the same composition powder processed in the traditional way. Maweja et al. [10] used mechanical alloying of the Ti(C,N)-W-Al binder raw materials. This resulted in the dissolution of W in Ti(C,N) and the formation of a homogeneous matrix during sintering. The PCBN materials processed in this way showed improved mechanical properties.

This paper is concerned with the development of the matrix microstructure in PCBN ceramic materials. The investigated PCBN materials had a matrix based on Ti(C,O,N), and were fabricated from powder mixtures milled with either WC-Co cemented carbide or (Ti,W)(C,N)-Co cermet milling media. The purpose of the investigation was to identify relationships between the phase assemblage of the sintered material, the additives and the amount and composition of the milling debris introduced during the preparation of the starting powder mixture. Partially densified materials were

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Table 1
The chemical composition of the milling bodies.

Milling body	W (at%)	Co (at%)	Ti (at%)	C (at%)	N (at%)
Cemented carbide	45.29	9.57	–	45.14	–
Cermet*	3.84	11.44	40.58	30.75	13.39

*W/(W + Ti) atomic ratio = 8.6×10^{-2} .

Table 2
The milling times used in the preparation of the starting powder mixtures.

Material	Pre-milling time	Milling time
A	30 min	15 min
B	30 min	37 min
C	30 min	60 min
D	8 h	2 h 40 min
E	8 h	6 h 25 min
F	8 h	10 h 20 min

A – C: WC-Co cemented carbide milling media; D–F: cermet milling media.

included in the study in order to create an understanding of the reactions taking place during densification.

2. Experimental procedures

2.1. PCBN materials

The experimental PCBN materials had a cBN volume fraction of around 50% and were fabricated with the addition of Ti(C,O,N) and smaller amounts of Al. Two types of milling media were used in the preparation of the starting powder mixtures; WC-Co cemented carbide milling bodies and cermet milling bodies containing (Ti,W)(C,N) grains in a Co-rich binder phase. The elemental compositions of the milling bodies are shown in Table 1. It may be noted that the WC-Co cemented carbide milling bodies had a significantly higher W content than the cermet bodies, but the amount of Co was similar (around 10 at%) in the two materials (Table 1).

The Ti(C,O,N) starting powder (Treibacher; 71 an% C, 23 an% O, 6 an% N; B1 NaCl crystal structure) was pre-milled in ethanol in an attritor mill in order to reduce the particle size. After pre-milling, powders of cBN (Diamond Innovations) and Al (Ecka Granules, MEPO27) were added to the slurries. Powder mixtures prepared in this way were then attritor milled for three different times in order to prepare batches with different amounts of milling media debris (Table 2). The use of cermet milling media (Table 2, materials D–F) required longer pre-milling and milling times in order to get similar W contents as in the WC-Co milled starting powder mixtures (Table 3). The prepared starting powder mixtures had, hence, three different levels of introduced W (Table 3); a low (around 0.3 at%), an intermediate (around 0.7 at%) and a high (around 1 at%) level. The amount of introduced Co also increased with milling time. The Co levels (in at%) were, however, around 10 times higher in the cermet-milled starting powder mixtures (Table 3, materials D–F) than in the WC-Co-milled batches (Table 3, materials A–C) for each

Table 3
The elemental composition of the starting powder mixtures.

Material	Al (at%)	W (at%)	Co (at%)	Ti (at%)	C (at%)	N (at%)	O (at%)	B (at%)
A	4.31	0.33	0.10	14.72	11.36	36.05	3.47	29.67
B	4.12	0.70	0.20	15.06	11.79	36.45	3.59	28.08
C	4.14	1.14	0.24	14.12	11.53	37.12	3.50	28.21
D	3.56	0.34	1.00	15.49	11.73	34.19	3.67	30.01
E	2.90	0.62	1.77	14.46	10.71	37.69	3.26	28.58
F*	1.88	0.94	2.69	12.89	9.32	38.42	3.00	30.87

A – C: WC-Co cemented carbide milling media; D–F: cermet milling media.

* Chemical composition determined after pre-sintering.

level of introduced W. The elemental compositions of the milled starting powder mixtures presented in Table 3 were determined by chemical analysis; the Al, W, Co and Ti cation contents by X-ray fluorescence and the C, N and O anion contents by combustion, balance B.

Polyethylene glycol (PEG) was added to the slurries towards the end of the milling process. The slurries were spray dried, and the granules were pressed into disc-shaped green bodies with a diameter of around 6 cm and a thickness of around 3 mm. These were pre-sintered in vacuum at 900 °C for 15 min. High pressure (HP) high temperature (HT) sintering was carried out under a pressure of 5.5 GPa at 1400 °C for 45 min.

2.2. Microstructural characterization

The phase compositions of the spray-dried granules, the pre-sintered compacts and the HPHT sintered compacts were determined by X-ray diffractometry (XRD) using $\text{CuK}\alpha$ radiation. The microstructures of the HPHT sintered materials were characterized by analytical scanning and transmission electron microscopy (SEM, TEM). Polished sections were imaged by backscattered electrons in a field emission Leo Ultra 55 SEM operated at an accelerating voltage of 8 kV. The instrument is equipped with an Oxford Instruments X-ray detector and the IncaEnergy system for qualitative and quantitative elemental analysis by X-ray energy dispersive spectrometry (XEDS). The detailed characterization of the microstructures was carried out in a Fei Tecnai LaB₆ TEM operated at 200 kV. The Tecnai is equipped with an EDAX X-ray detector and XEDS software for elemental analysis. The PCBN specimens were sufficiently conducting under the electron beam in both SEM and TEM and did not require coating for imaging and analysis.

Thin foils for TEM were prepared by standard techniques; i.e. grinding, polishing and dimpling to a thickness of 30–40 μm followed by ion beam milling. Sputtering was initially carried out by 5 kV Ar ions at an incidence angle of 15°. The Ar ion energy and the angle of incidence were reduced during sputtering, and the final thinning was carried out by 1 kV ions under an angle of 4°.

Based on the results from the XRD and SEM/XEDS investigations of all HPHT sintered materials, the HPHT sintered materials C, E and F (Tables 2 and 3) were selected for a detailed TEM characterization.

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