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Characterization of wurtzitic boron nitride compacts

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Characterization of sintered polycrystalline wurtzitic boron nitride compacts was carried out regarding the different crystalline phases that are formed at high temperature and high pressure, composition, particle size distribution of BN and binder and hardness. Wurtzitic boron nitride, cubic boron nitride, TiC/TiN solid solution, TiB and TiB_2 were the crystalline phases that have been observed in sintered wurtzitic boron nitride compacts. The particle size distribution of BN and the binder was found to be comparable (1 to 5 μm), with about 80% of the particles lying between 2 and 3 μm. Weight percentages of different elements present in these compacts were determined. The average Knoop hardness values under 500 g load were measured, and the variation of hardness as a function of position on the specimen surface was studied.

1. Introduction

Boron nitride is a man-made material which crystallizes in hexagonal, cubic and wurtzitic structures. Cubic and wurtzitic boron nitride can be synthesized at high pressures [1–4]. Both these materials have hard sinterable properties and can be sintered into a compacted disc at high pressure and high temperature with the use of suitable binders. Sintered polycrystalline compacts of cubic boron nitride (C-BN) and wurtzitic boron nitride (W-BN) find extensive applications in the machining and shaping of hardened ferrous materials.

C-BN compacts have been synthesized by a number of scientists at high temperature and high pressure [5–7]. W-BN compacts have been synthesized by Saito and Sawaoka [8]. However, the compositional details of these polycrystalline sintered compacts are still less well known. Recently the detailed compositional analysis of C-BN compacts has been reported [9]. The aim of the present investigation is to carry out compositional analysis in polycrystalline sintered compacts of W-BN. Powder X-ray diffraction has been used to study the various crystalline phases that are formed at high temperature and high pressure. Scanning electron microscopy (SEM) has been used to study the microstructure and particle size distribution of boron nitride and the binder. Energy-dispersive X-ray (EDX) analysis has been carried out for determination of the concentration of different elements present. Knoop hardness measurements were also made on these compacts. Results of these investigations are reported in this paper.

2. Experimental details

2.1. Samples

W-BN compacts that have been used in the present investigation were obtained from two different sources. These samples are hereby labelled as Type A and Type B.

2.2. Powder X-ray diffraction

Powder X-ray diffraction is a standard technique used for phase analysis in a polycrystalline solid. This technique has been used to identify different crystalline phases formed at high temperature and high pressure in W-BN compacts. Cu$K\alpha$ with a divergence of 1° was used as the exploring beam to record the X-ray diffractograms. The diffractograms were recorded in reflection geometry with a scan speed of $1° \text{min}^{-1}$.

2.3. Scanning electron microscopy

SEM was used to study the microstructure and particle size distribution of boron nitride and the binder in W-BN compacts. A Jeol JSM-35 CF was used for this purpose. All the SEM photographs were recorded in the secondary electron emission mode with 0° tilt.

2.4. EDX analysis

Elemental analysis in sintered polycrystalline W-BN compacts was carried out using EDX analysis. This was done on an ISI Super-III SEM (Cambridge, UK). An electron beam with 30 kV and 0° tilt with respect to the specimen was used to excite the characteristic X-ray spectra of the constituent elements. The distance between specimen and detector was 30 mm. A working distance of 16 mm was used. The spot size was kept constant at its maximum in order to increase the emitted X-ray signal. A counting interval of 200 sec was used for counting the emitted X-ray photons from the constituent elements in the W-BN compacts. These experimental parameters were fixed constant for the unknown specimen as well as for the standard reference elements involved. The X-ray spectra were analysed using a Tracor Northern TN 2700 spectrum analyser.

2.5. Hardness measurement

Hardness measurements of W-BN compacts were made on a Zwick hardness tester (Zwick GmbH, Ulm,

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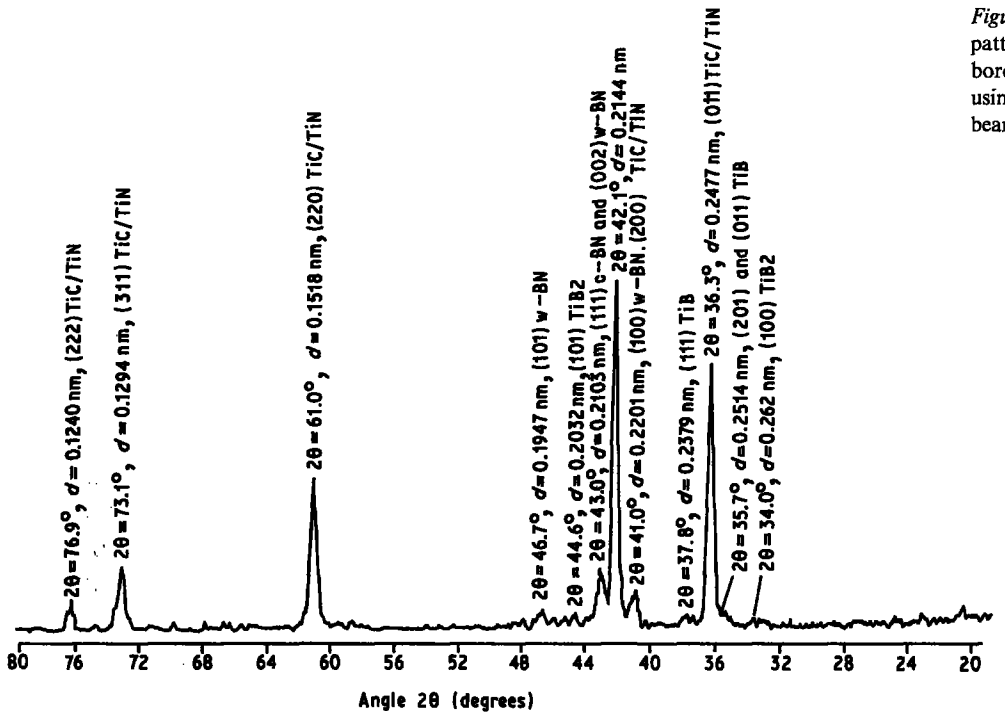

Figure 1 Powder X-ray diffraction pattern obtained for wurtzitic boron nitride compact of Type A using Cu$K\alpha$ as the exploring beam. 1 Å = 0.1 nm.

FRG) under 500 g load. Knoop hardness values were determined from these measurements. Average hardness values were determined from measurements made at several points of the specimen surface. Measurements were made at least three times at different points.

3. Results and discussion

3.1. X-ray characterization

Fig. 1 shows a typical powder X-ray diffraction pattern obtained for a W-BN compact of Type A. It is seen that there is no diffraction line due to hexagonal boron nitride, which if present would have given a diffraction line at $2\theta = 26.7°$. The diffraction maxima observed at $2\theta = 41.0°$ ($d = 0.2201$ nm) and $2\theta = 46.7°$ ($d = 0.1947$ nm) are due to (1 0 0) and (1 0 1) reflections of W-BN, respectively. Here d is the interplaner spacing and θ the Bragg angle. The diffraction line observed at $2\theta = 43.0°$ ($d = 0.2103$ nm) is due to the (1 1 1) reflection of C-BN and the (0 0 2) reflection of W-BN. The d value for (1 1 1) of C-BN is 0.2114 nm and that for (0 0 2) of W-BN is 0.2088 nm. The difference in d values for interplaner spacings of these planes, Δd, is 0.0026 nm. For Cu$K\alpha$ the difference between the Bragg angles of these reflections, $\Delta\theta$, corresponds to 0.2797°. It is not possible to resolve the two lines whose Bragg angles are separated by an angle as small as this with the present experimental conditions (divergence of exploring beam = 1°).

The observed diffraction maxima (Fig. 1) at $2\theta = 36.3°$ ($d = 0.2477$ nm), 42.1° ($d = 0.2144$ nm), 61.0° ($d = 0.1518$ nm), 73.1° ($d = 0.1294$ nm) and 76.9° ($d = 0.1240$ nm) are due to (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) reflections of TiC/TiN solid solution, respectively. The diffraction maxima observed at $2\theta = 34°$ ($d = 0.262$ nm) and 44.6° ($d = 0.2032$ nm) are due to TiB_2. Also, diffraction peaks observed at $2\theta = 35.7°$ ($d = 0.2514$ nm) and 37.8° ($d = 0.2379$ nm) suggest the presence of a TiB phase in the sintered W-BN compact. These reflections correspond to (2 0 1) and (0 1 1), and (1 1 1) diffracting planes of TiB. However, the strongest diffraction line (1 0 2) of TiB at $2\theta = 42.2°$ ($d = 0.2140$ nm) overlaps with the strongest reflection of TiC/TiN. From this analysis it can be concluded that a W-BN compact of Type A consists of crystalline phases of W-BN, C-BN, TiC/TiN solid solution, TiB and TiB_2.

Fig. 2 shows a typical powder X-ray diffraction pattern obtained for a Type B specimen using Cu$K\alpha$ radiation. The observed X-ray diffraction pattern is essentially similar to that of Type A specimens, and the corresponding crystalline phases are marked in Fig. 2 with their observed diffraction maxima along with 2θ and d values. Therefore, in this case also, the crystalline phases observed are: W-BN, C-BN, TiC/TiN solid solution, TiB and TiB_2.

3.2. Microstructure and particle size analysis

Figs 3a and b show typical SEM micrographs of W-BN compacts of Type A and B, respectively. Black regions correspond to BN grains and white regions to TiC/TiN particles. It can be seen in these figures that BN particles are uniformly distributed in the TiC/TiN matrix. The following broad features were observed in these micrographs:

(i) In most regions of the specimen surface BN particles are held together by TiC/TiN particle.

(ii) In some regions clustering of BN and TiC/TiN are observed. Clusters of BN particles are bonded by clusters of TiC/TiN particles.

(iii) BN–BN bonding is also observed occasionally.

Particle size and particle-size distributions of BN and binder grains in W-BN compacts of Type A and Type B were determined from the magnified SEM photographs. Since the signal to background ratio for BN particles was very poor it was not possible to carry out image analysis through an image analyser. This

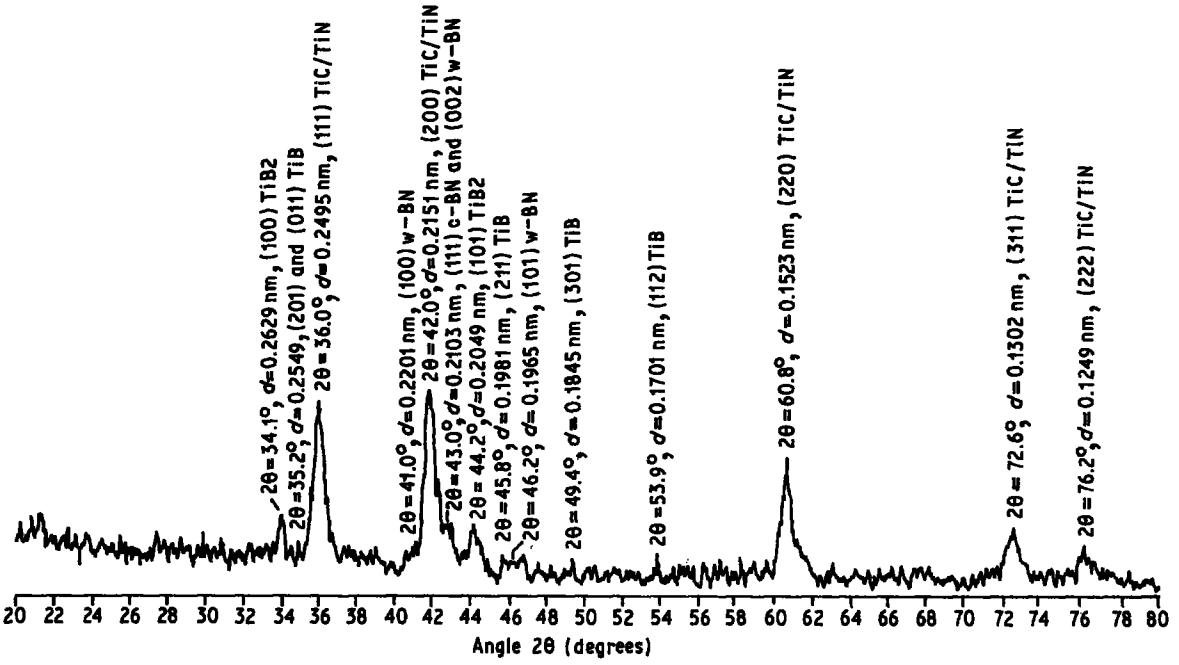

Figure 2 Powder X-ray diffraction pattern obtained for wurtzitic boron nitride compact of Type B using Cu$K\alpha$ as the exploring beam. 1 Å = 0.1 nm.

analysis was carried out manually from the magnified SEM photographs. In doing so the concept of particle size as adopted by the Federation Européene des Frabricants de Produits Abrasifs (FEPA) was used. This concept is as follows: "the diameter of the circle which completely encloses the particle is said to define the particle size".

Table I shows the results of particle size analyses made on W-BN compacts of Type A and B. This table shows the percentage distribution of BN and binder particles having a particular particle size in W-BN compacts. For W-BN compacts of Type A, the particle sizes of BN and the binder range from 1 to 5 μm. Furthermore, for both BN and the binder, the maximum number of particles have their particle size around 2 μm with 80% of particles lying between 2 and 3 μm. For W-BN compact of Type B, the particle size distribution of BN and the binder are similar to that of Type A specimen. In this case also, the maximum number of BN and binder particles have their particle sizes around 2 μm, with about 75% of the particles lying in the range of 2 to 3 μm. These results show that W-BN compacts of both types consist of BN and binder particles with similar particle size distributions (1 to 5 μm) with the majority of BN and binder particles having a size of 2 to 3 μm.

3.3. Elemental analysis

Elemental analysis was carried out for W-BN compacts of Types A and B using the EDX technique. The experimental conditions for obtaining characteristic X-ray spectra of constituent elements and the standard reference elements were fixed constant and are given in Section 2.4. It may be mentioned that the determined values of the weight percentages of the elements represent absolute values. These values were determined by comparing the X-ray counts for 200 sec emitted by the elements in the given compact to the corresponding values obtained for pure elements involved. The following amounts (wt %) of elements were determined in W-BN compacts:

Type A: Ti 42.0, Al 2.0, Fe 0.6, Mo 0.4

Type B: Ti 40.0, Al 0.8, Fe 0.5, Co 0.5

3.4. Hardness

Hardness measurements were carried out under 500 g load on W-BN compacts and average Knoop hardness

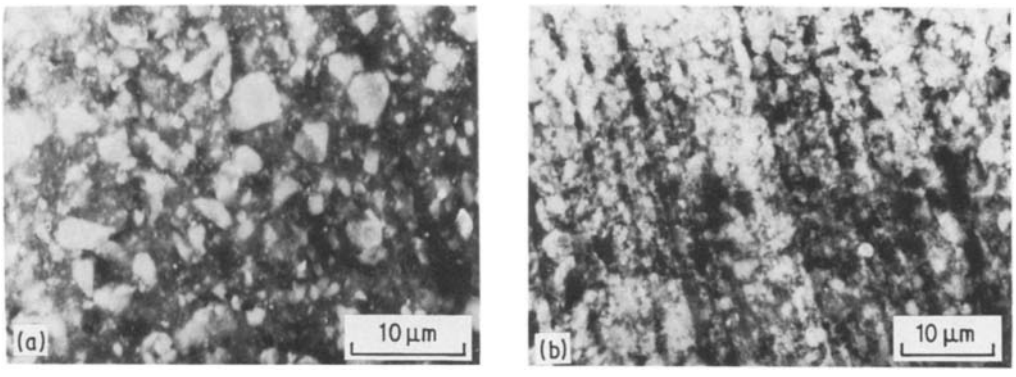

Figure 3 Typical SEM micrographs for samples of (a) Type A, (b) Type B.

TABLE I Particle size distribution of BN and binder

Sample	BN particles		Binder particles	
	% of particles	Particle size (μm)	% of particles	Particle size (μm)
Type A	10.0	1.0	8.0	1.0
	60.0	2.0	55.0	2.0
	20.0	3.0	25.0	3.0
	6.0	4.0	10.0	4.0
	4.0	5.0	2.0	5.0
Type B	15.0	1.0	13.0	1.0
	65.0	2.0	62.0	2.0
	10.0	3.0	12.0	3.0
	7.0	4.0	9.0	4.0
	3.0	5.0	4.0	5.0

values were determined from measurements made at several points of the specimen surface. At least three measurements were made at each indentation. The average values measured for W-BN compacts were: Type A 3850 kg mm^{-2}, Type B 4100 kg mm^{-2}.

A detailed hardness measurement was also made on the specimen surface to study the variation of hardness as a function of position. Knoop hardness under 500 g load was measured at a regular interval of 0.3 mm along the diameter of the W-BN disc. It was observed that values fluctuated significantly from point to point on the specimen surface. This behaviour was observed in both types of W-BN compact. The observed variation in hardness seems to be due to microstructural fluctuations caused by inhomogeneity of the material.

4. Conclusions

The crystalline phases detected in W-BN compacts of Type A and B were found to be the same, namely W-BN, C-BN, TiC/TiN, TiB, TiB_2.

Particle size distribution analysis of BN and the binder in specimens of Types A and B showed that these compacts consist of BN and binder particles with practically the same particle size range, 1 to 5 μm, with the maximum number of particles lying between 2 and 3 μm.

Quantitative EDX analysis showed the following weight percentage of elements present in W-BN compacts:

Type A: Ti 42.0, Al 2.0, Fe 0.6, Mo 0.4

Type B: Ti 40.0, Al 0.8, Fe 0.5, Co 0.5

Average knoop hardness values under 500 g were: Type A 3850 kg mm^{-2}, Type B 4100 kg mm^{-2}.

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