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Short Communication

BN Sintered with Al: Microstructure and Hardness

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Abstract: Boron nitride (BN) was sintered with Al by high pressure hot pressing while preserving the molar ratio of BN:Al 9:1. Ready made samples were additionally annealed at 950°C under pressure, at 3×10^{-3} Pa for 1 h. The structure of sintered material before and after annealing was studied using transmission electron microscope. From the microscopic observations it could be concluded that the samples prepared both before and after annealing exhibit a compact structure. At the BN–Al interface immediately after pressing, small crystallites of Al can be observed. Additionally, after annealing, a layer of columnar AlN grains of 0·04 μm thickness are visible. In the area between AlN and BN phases polycrystalline AlB_{10} and AlB_{12} phases can be seen.

Hardness of the samples increases twice (~20 GPa/HK1) after annealing. Thermal treatment also results in an increase in mechanical strength of the sintered BN–Al system. © 1996 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Polycrystalline materials produced from cubic boron nitride are used widely in machine building because of their unique properties, such as high resistance to oxidation, good thermal conductivity and chemical inertness to iron and iron alloys. These materials are synthesized in special reactors by high-pressure and high-temperature treatment. Metals of groups IV, V and VI of the periodic table and/or other metallic elements, such as aluminium, cobalt and nickel, are added to activate sintering.[1] Chemical reactions between them and boron nitride occur, resulting in the formation of some new phases. Prediction of the final products of the reactions taking place during sintering, as well as elucidation of their mechanisms, is of crucial importance in the selection of the appropriate binding phase. Among metals, aluminium is the most frequently used as a binding material for the sintering of cubic boron nitride with other elements.

From chemical equilibria calculations in the BN–Al system it follows that aluminium reacts with BN in wide temperature and pressure ranges forming either one phase (AlB_{12}) or two two-component phases: AlB_2 and AlN, AlB_{12} and AlN.[2]

The type and number of new phases formed depends strongly on pressure and temperature. Theoretical calculations were corroborated by experimental studies. However, experiments do not give information concerning the new phase localizations with respect to BN grains.

In the present work the microstructure of BN sintered with Al was studied. Hardness of the sintered material before and after thermal annealing was also studied using the Knoop method.

2 EXPERIMENTAL PROCEDURE

Boron nitride (ABN-300, de Beers, 3–5 μm grain size) and aluminium (Polish product, 3–5 μm grain size) were mechanically mixed in ethyl alcohol and then pressed into pellets of 6 mm diameter under a pressure of 1×10^7 Pa and at a temperature of 1750°C. In the samples the molar ratio of BN:Al 9:1 was kept constant. Additionally the samples were thermally treated in vacuum of 3×10^{-3} Pa for 1 h.

Thin foils for transmission electron microscope were prepared by dimpling on a Gatan 656 Dimpler and ion milled with a Gatan 600 Duo Mill under standard conditions.

The microstructure observations were performed using Philips CM20 TWIN (200 kV) transmission electron microscope.

In hardness measurements the Knoop diamond identer loaded with 9·81 N was used.

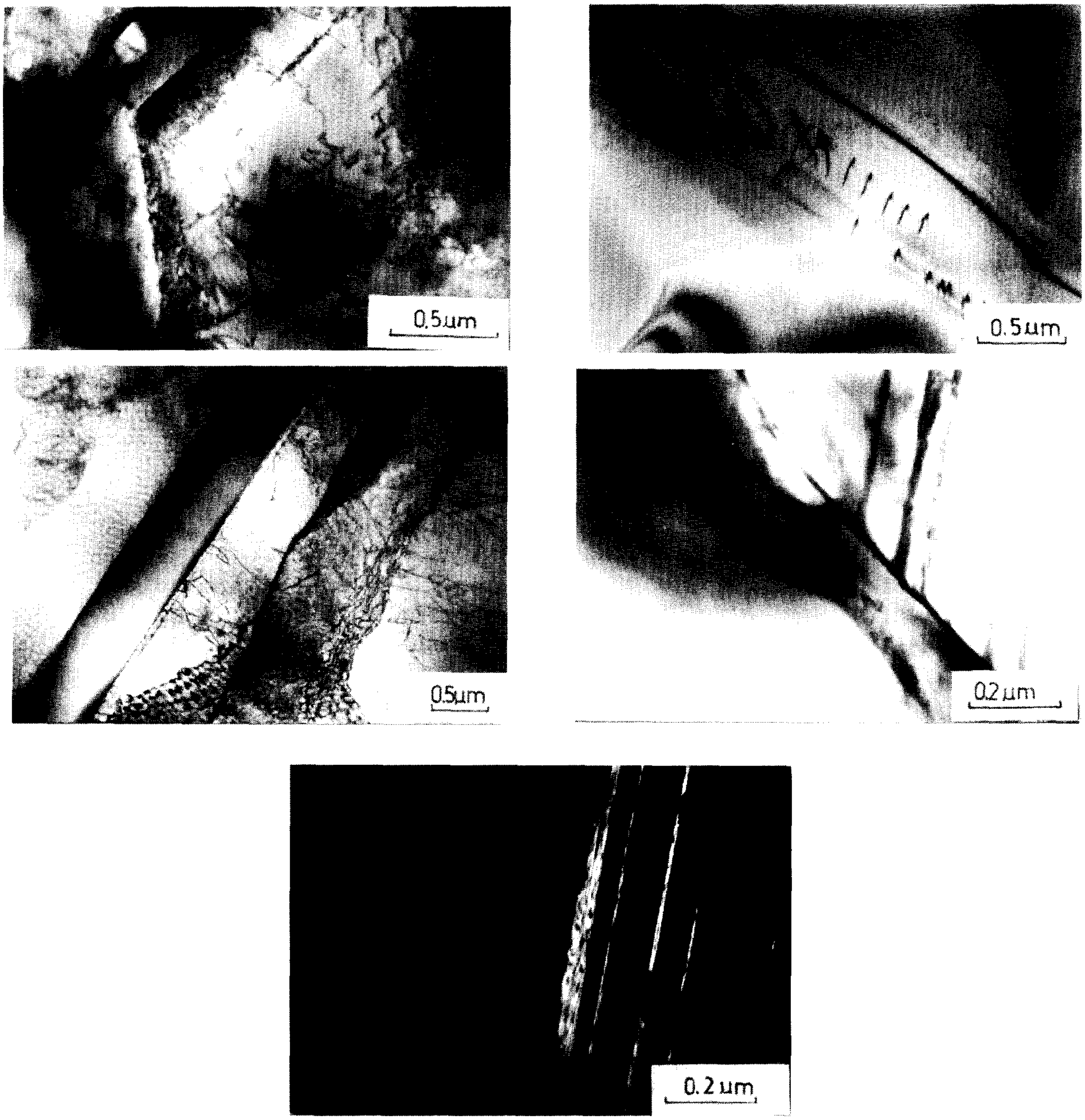

Fig. 1. Microstructure of BN sintered with Al before thermal annealing (1a), dislocations in Al (1b, foil orient.$[001]_{Al}$), dislocations in BN (1c, orient.[112]BN), microtwins in BN ($1d_1$-BF, $1d_2$-DF from additional twin spot orient. $[011]_{BN}$).

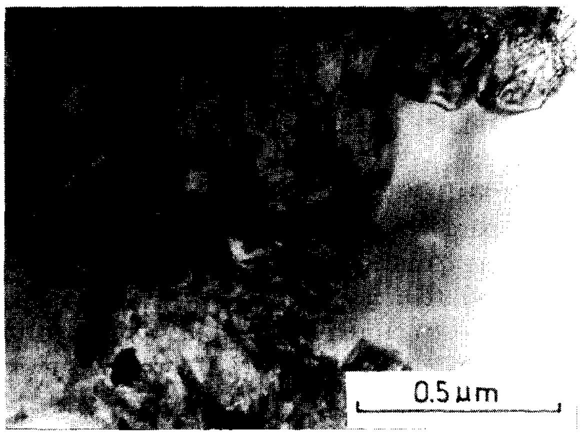

Fig. 2. Polycrystalline areas on BN surface. Polycrystals of AlB_{10}, AlB_{12} were identified.

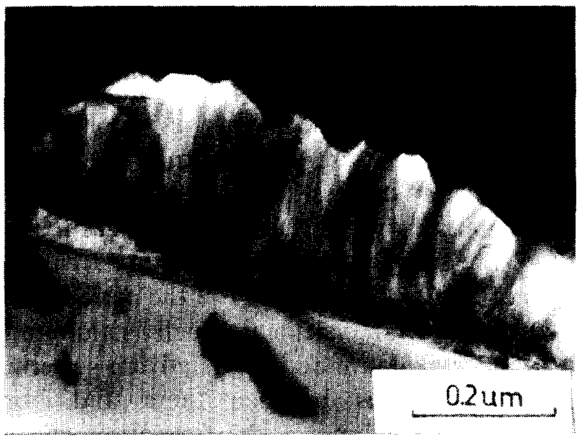

Fig. 3. Microstructure of AlN columnar grains.

3 RESULTS AND DISCUSSION

The microstructure of BN sintered with Al immediately after hot pressing is shown in Fig. 1(a). In both, BN and Al structural defects, namely dislocations in the metal (Fig. 1(b)), dislocations (Fig. 1(c)) and microtwins in boron nitride (Fig. 1(d)), can be observed. In BN grains the presence of Al was established and by the EDX method this shows that the metal diffuses into ceramics.

In the sample after thermal treatment, close to the BN surface, fine crystalline areas were observed (Fig. 2). Polycrystals of AlB_{10} and AlB_{12} were identified. At the interfaces one can observe the formation of thin layers of columnar grains of 0·04 μm thickness and similar crystallographic orientation. (Fig. 3). Microanalysis of chemical composition showed that this layer is formed mainly by aluminium nitride.

Electron diffraction obtained from selected grains agrees well with AlN lattice. More detailed analysis of the grains shape allowed to conclude that the AlN grains nucleate at the Al–BN interface and then they grow into the metal area.

Table 1. Hardness of BN sintered with Al

Sample	Hardness HK 1 [GPa]
1 Al+9 BN (before annealing)	10
1 Al+9 BN (after annealing)	20

The hardness of the samples before and after thermal treatment differs significantly. Results of these studies are collected in Table 1. It can be seen that hardness increases from 10 to 20 GPa. Thus it can be stated that thermal annealing of BN sintered with Al accompanied by formation of the new phase (AlN) results in the increase in mechanical strength of the material.

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