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Sintering of Cubic Boron Nitride with Added Aluminum at High Pressure and High Temperatures

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Specimens sintered at high pressure and high temperature were characterized by optical microscopy, X-Ray diffraction and TEM measurement. The crystalline phases of the reacted products and microstructural features were discussed with Knoop hardness measurements of the mirror polished surfaces.

1.INTRODUCTION

Cubic BN (cBN) of zinc-blende structure has excellent hardness, high thermal conductivity and electrical insulation. The cBN is very difficult to sinter. It was reported that the cobalt infiltration within cubic BN and WC played an important role of sintering of cBN [1]. Recently, similar experimental results were presented by the authors [2]. In the metal/ceramic systems, wetting of liquid metal is a controlling parameter for sintering. Chemical reactions between liquids and solid surfaces can be induced good wetting condition [3]. The wetting is related to the interfacial free energy between metal and ceramic.

This paper describes the bonding process of cubic BN and aluminum, and discuss the sintering mechanism.

2.EXPERIMENTAL

2.1.Sample preparation

The particle size of the cubic BN was between 1.0 and 3.0 μm(SHOWA DENKO SBN-F JAPAN) and of the aluminum was between 1.0 and 4.0 μm(RARE METALLIC CO., LTD. JAPAN). The mixtures containing cBN from 50 to 90 mol% were prepared with 5 mol% increments. The samples were prepared by the same procedure as ref.2.

2.2.Polishing and Analysis

Samples for TEM/STEM studies were prepared by the usual polishing, dimple-grinding, and ion-milling technique. The TEM experiments were done with a microscope(Model:H-9000, HITACHI, JAPAN) at 300 Kv accelerating voltage, and an analytical STEM(Model: H-9110, HITACHI, JAPAN). The other analytical instruments were described in an elsewhere[2].

3.RESULTS AND DISCUSSION

3.1 Micro Knoop Hardness

Indented Knoop microhardness of the polished surfaces were plotted in Fig.1. The hardness values were the average of the different 8 points on the surface and the deviation of the values was within ±80 kg/mm^2. The two

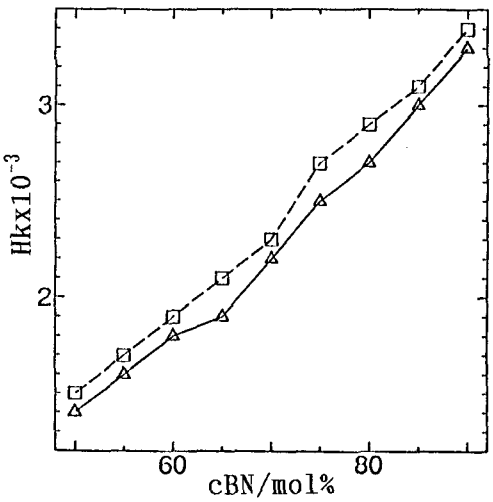

Fig.1.Knoop microhardness of the sintered specimens in cBN-Al system.

curves in the figure correspond to 1200°C sintering(solid line) and 1400°C sintering(dotted line). These two lines almost linearly increased with increasing cBN content. The hardness values of sintering at 5.75 GPa, 1400°C for 20 min was higher than of sintering at 5.75 GPa, 1200°C for 20 min from 50 mol% cBN to 90 mol% cBN composition. The highest hardness values was 3340 kg/mm^2 as sintering at 5.75 GPa, 1400°C for 20 min for 90 mol% cBN composition.

Indentation marks of the samples of sintering at 5.75 GPa, 1200°C and 1400°C for 20 min from 50 mol% cBN to 80 mol% cBN compositions all appeared no microcracks and depression except 90 mol% cBN composition. It indicated that the part toughness was improved than the cBN-WC system [2]. The typical photographs of the indentation mark were shown in Fig.2.

3.2.XRD Analysis

XRD results of the samples were tabulated on table.1. AlN(hexagonal), AlB_2(hexagonal) and α-AlB_{12}(tetragonal) were detected with cBN. No aluminum diffraction was observed. The similar results were obtained sintering for both 1200°C and 1400°C. According to the XRD results, following reactions were suggested:

$$2BN + 3Al = AlB_2 + 2AlN \quad (1)$$
$$12BN + 13Al = \alpha\text{-}AlB_{12} + 12AlN \quad (2)$$

From 50 mol% cBN to 60 mol% cBN compositions, the second reaction occurred after first step (1) of reaction end, While from 80 mol% cBN to 90 mol% cBN compositions, only the second reaction was took place, because of low aluminum composition.

3.3.Microstructural Analysis

The polished surfaces of the samples with different cubic BN contents were observed by optical microscopy shown in Fig.3. No porosity was found out, but many of coarse grains were visible. Figure 4 shows a few SEM photographs of the fracture surfaces of the sintered specimens. The SEM fractography sintered at 1200°C and 1400°C, and 5.75 GPa for 20 min showed primarily transgranular fracture. For 50 mol% cBN composition(see Fig.4(a) and (d)), the reacted crystals of about 12 μm in size were grown with layered texture. The cracks were observed between cubic BN and AlN as well as

Table.1.XRD data of the sintered at 1200°C and 1400°C,5.75 GPa for 20 min in cBN-Al system.

composition (cBN mol%)	50	55	60	65	70	75	80	85	90
phase present	cBN AlN AlB_2 α-AlB_{12}	cBN AlN AlB_2 α-AlB_{12}	cBN AlN AlB_2 α-AlB_{12}	cBN AlN AlB_2	cBN AlN AlB_2	cBN AlN AlB_2	cBN AlN α-AlB_{12}	cBN AlN α-AlB_{12}	cBN AlN α-AlB_{12}

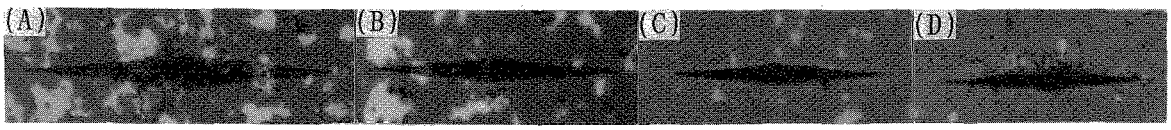

Fig.2.Surface optical micrographs of the indentation marks of the typical specimens sintered at 1400°C,5.75 GPa for 20 min. (A) 50 mol% cBN, (B) 70 mol% cBN, (C) 80 mol% cBN, (D) 90 mol% cBN.

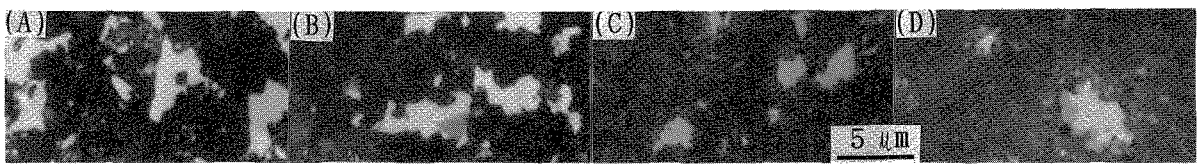

Fig.3.Optical micrographs of polished surfaces sintered at 5.75 GPa,1400°C for 20 min. (A) 50 mol% cBN, (B) 70 mol% cBN, (C) 80 mol% cBN, (D) 90 mol% cBN.

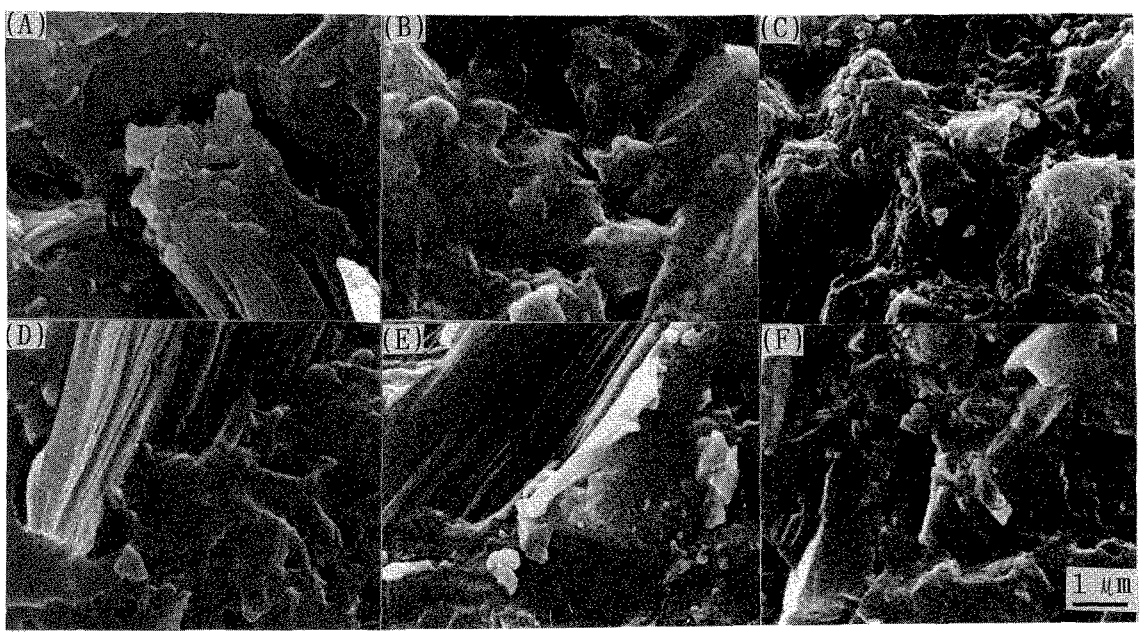

Fig.4.SEM micrographs of fracture surfaces of the typical specimens sintered at 5.75 GPa for 20 min. (A) 50 mol% cBN,1400°C; (B) 70 mol% cBN,1400°C; (C) 90 mol% cBN,1400°C;(D) 50 mol% cBN,1200°C; (E) 70 mol% cBN,1200°C; (F) 90 mol% cBN,1200°C.

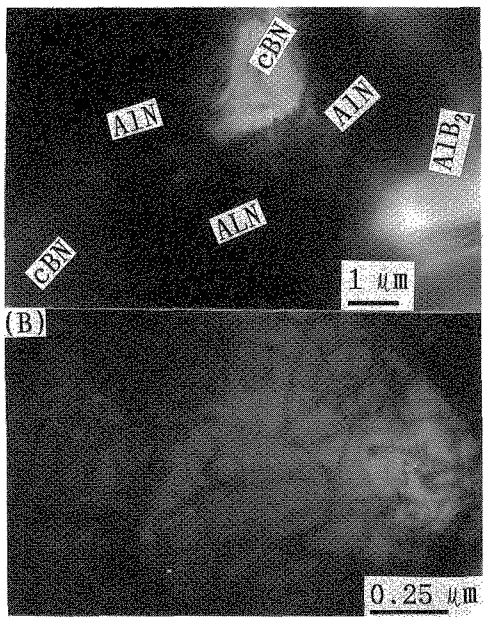

Fig.5.TEM micrograph of microstructure.(A)Bright-fieldimage of 50 mol% cBN sample sintered at 1400°C, 5.75 GPa for 20 min. (B) dislocation survey of ALN phase showing in (A).

AlN and AlB_2 or α-AlB_{12} sintered at 1200°C and 1400°C, due to the difference of the thermal expansion coefficients between these crystals. In general, the specimens sintered at 1400°C, 5.75 GPa for 20 min showed more homogeneous microstructure than those at 1200°C.

The compositional distribution of 50 mol% cBN sample sintered at 1400°C, 5.75 GPa for 20 min was investigated by diffraction patterns and EDX analysis as shown in Fig.5(A). It indicated that AlN phase exist around cubic BN grain, AlB_2 phase and or α-AlB_{12} phase exist between AlN phase and AlN phase. The diffusion of B ion was faster than N ion within aluminum liquid, and AlN phase was in intimately contacted with cBN grain.

Further dense dislocations of AlN phase around cBN grain were observed shown in Fig.5(B). All possible slip systems[4] were observed.

4.CONCLUSION

Sintering of cubic BN and aluminum were carried out at 5.75 GPa, 1200°C and 1400°C for 20 min. The reactions between cubic BN and aluminum decreased the interfacial free energy, and improved the wetting, so the hardness was high. On the other hand, the reactions between cubic BN and aluminum was fast at 5.75 GPa, both 1200°C and 1400°C. This means that the viscosity of aluminum at 1200°C was larger than at 1400°C, so that the liquid of aluminum did not cover the cubic BN grain at 1200°C, the reactions occurred rapidly, further the crystals of AlN and AlB_2 or α-AlB_{12} grew large. The difference of the thermal expansion coefficients between these crystals decreased the hardness at 1200°C, as well as the toughness in cBN-Al system.

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