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Sintering and characterization of Al_2O_3–B_4C composites

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The effect of B_4C on the densification, microstructure and mechanical properties of pressureless sintered Al_2O_3–B_4C composites have been studied. Sintering was performed without sintering additives with varying B_4C content from 0–40 vol%. Up to 20 vol% B_4C, more than 97% theoretical density was always obtained when sintered at 1850 °C for 60 min. On increasing the sintering time from 30–120 min, there was no change in density. The result of X-ray diffraction analysis showed that no reaction occurred between Al_2O_3 and B_4C. The grain growth of Al_2O_3 was inhibited by B_4C particles pinned at the grain boundary and the grain-boundary drag effect. The critical amount of B_4C to drag the grain boundary migration effectively was believed to occur at 10 vol% B_4C sintered at 1850 °C for 60 min. The maximum three-point flexural strength was found to be 550 MPa for the specimen containing 20 vol% B_4C, and the maximum microhardness was 2100 kg mm^{-2} for 30 vol% B_4C specimen.

1. Introduction

Aluminium oxide (Al_2O_3) ceramics are strong, hard and consequently have many possible applications. Nevertheless one of the shortcomings of this material is its brittle characteristics. In order to obtain higher strength, hardness and fracture toughness, two composite materials have been developed by dispersing a second phase [1–4] or embedding whisker [5], respectively, in an Al_2O_3 matrix. The composites with titanium carbide (TiC) are some of the most renowned wear-resistant materials and in particular have many applications as cutting tools [4]. Table I shows the kind of ceramic cutting tools [6].

Boron carbide (B_4C), however, has similar material properties as TiC. As shown in Table II [7], B_4C has more promising properties, such as low theoretical density, good wear resistance, and high hot hardness. Therefore, composites with B_4C instead of TiC would be more suitable for cutting tools and wear-resistant materials. However, many of the important properties, especially the sintering behaviour and mechanical properties of these composties, have not yet been reported.

In the present study, the sintering behaviour of the Al_2O_3–B_4C composites and the role of B_4C on densification and grain growth were investigated. The mechanical properties of the composites were also obtained and evaluated.

2. Experimental procedure

The starting powders used in the experiments were Al_2O_3 (AKP-50, Sumitomo Chem. Co., Japan) with mean particle size 0.2 μm and 99.99% purity and B_4C (Hermann C. Starck, Berlin) with 1.0 μm average particle diameter. Al_2O_3 and 0–40 vol % B_4C were mixed for 2 h in a centrifugal mill. After drying for 48 h, the mixtures were then ground to pass a 60-mesh sieve. Specimens were pressed into rods of 10 mm diameter × 4 mm length under 75 MPa and were cold isostatically pressed (CIP) under 150 MPa. The compacted specimens were dried at 50 °C for 24 h. These compacted specimens were sintered at 1750–1850 °C for 0–120 min in an argon flowing atmosphere. The temperature in the furnace was measured with an optical pyrometer.

The composites identified were analysed by X-ray diffraction (XRD). Their relative densities were calculated from the theoretical density and the apparent density which was measured by the Archimedean method. The microstructure was examined by optical microscopy and scanning electron microscopy (SEM), and the average grain size was obtained by the linear intercept method [8]. Flexural strength was measured for sintered bodies by the three-point bending test, and micro-Vickers hardness was determined at 500 g load for at least seven points on the polished surface [9]. Fracture toughness was measured by the indentation method [10] at 1 kg load for at least seven points.

3. Results and discussion

3.1. Sintering behaviour

Fig. 1 shows the X-ray diffraction patterns of Al_2O_3–30 vol % B_4C. Here, the diffraction lines of Al_2O_3 and B_4C are observed. Although not given in the figure, similar diffraction lines were observed for the composites with different B_4C content. Such

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TABLE I Room-temperature properties of tool materials

Tool material	Strength (MPa)	Hardness, R_A	Toughness (MPa $m^{1/2}$)
Al_2O_3	500– 700	93–94	3.5– 4.5
Al_2O_3–ZrO_2	700– 900	93–94	5.0– 8.0
Al_2O_3–TiC	600– 850	94–95	3.5– 4.5
Al_2O_3–SiC_w	550– 750	94–95	4.5– 8.0
Si_3N_4	700–1050	92–94	6.0– 8.5
Sialon	700– 900	93–95	4.5– 6.0
WC–Co alloys	1250–2100	91–93	10.0–13.5

TABLE II Comparison of several properties of B_4C with TiC

	B_4C	TiC
Specific gravity (g cm^{-3})	2.52	4.93
Strength to density ratio	15.9	7.2
Knoop hardness, H_{K100} (kN mm^{-2})	29–31	21–22
Tensile strength (kN mm^{-2})	0.4	0.35
Compressive strength (kN mm^{-2})	2.8	3.0
Elasticity modulus (kN mm^{-2})	460	400
Specific electric resistance (Ω cm at 25 °C)	0.1–10	0.1

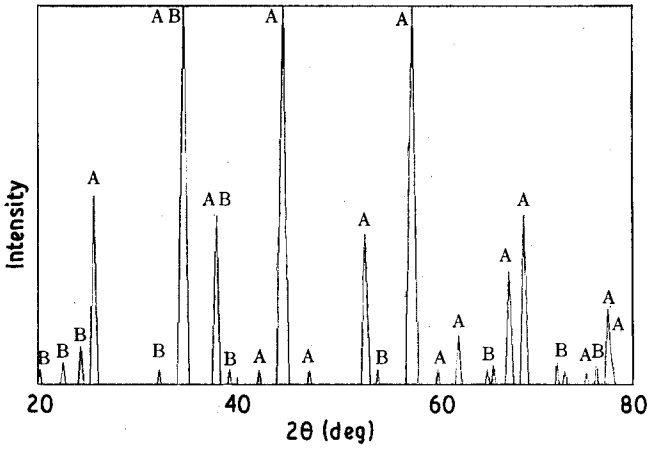

Figure 1 XRD pattern of the Al_2O_3–30 vol % B_4C. A, Al_2O_3; B, B_4C.

identical patterns indicate that Al_2O_3 and B_4C are the only host phases existing in the composites in sintering. XRD studies for the specimens show that no reaction occurs between Al_2O_3 and B_4C [11].

Fig. 2 shows the relation between relative density and B_4C content of composites which are sintered at 1850 °C for 60 min. The relative density increased with increasing B_4C addition from 94% (without B_4C) to about 97% (5–20 vol % B_4C). The change in relative density indicates that B_4C works as a sintering aid and promotes densification. However, excessive content of B_4C (40 vol % added) causes a decrease in relative density. It is believed that such a decrease in density results from the fact that densification is affected by the poor sinterability of B_4C.

Fig. 3 shows scanning electron micrographs of the polished and etched surface of the specimens which were sintered at 1850 °C for 60 min. Fig. 3a shows the etched surface of monolithic Al_2O_3. The most important feature of the microstructure is the existence of many large pores at grain boundaries or triple junc-

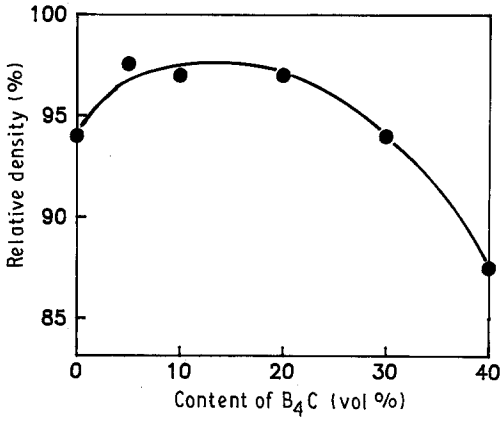

Figure 2 Effect of B_4C content on the sintered density of Al_2O_3–B_4C ceramics sintered at 1850 °C for 60 min.

tions. This indicates that the densification of Al_2O_3 was very poor and grain growth was very rapid at this temperature. The existence of these pores causes a decrease in density in the composites because the removal of such isolated pores at grain boundaries and triple junctions are generally difficult. The grain growth was so significant that the largest grain was about 40 μm in size.

On the other hand, when compared to the monolithic Al_2O_3, fewer pores at grain boundaries and smaller grain size of Al_2O_3 were observed in the composites with 5–20 vol % B_4C content (Fig. 3b–d). It is believed that B_4C slowed down the rapid growth of Al_2O_3 and may have contributed to the promotion of densification and reducing grain size of Al_2O_3.

The average grain size of Al_2O_3 plotted against B_4C content (Fig. 4). The graph shows the rapidly decreasing grain size of Al_2O_3 with the increasing B_4C content. It is recognized that B_4C effectively inhibited grain growth of Al_2O_3. The critical amount of B_4C to drag the grain boundary migration effectively was believed to occur at 10 vol % B_4C. Fig. 5 shows that the B_4C particles were located at the grain boundaries of Al_2O_3 and that they had the grain-boundary pinning effect. Owing to the B_4C particles, the movement of the grain boundaries of Al_2O_3 was slowed down, so that no pores were trapped in the grains.

Densification of the specimen appears to be enhanced by the effect of B_4C, which is regarded as a grain-growth inhibitor. The densification rate of the composite which contained 5–20 vol % B_4C was high, as shown in Fig. 6. The relative density, which was initially about 55% theoretical density for green compact (measured by dimension), reached 96% at

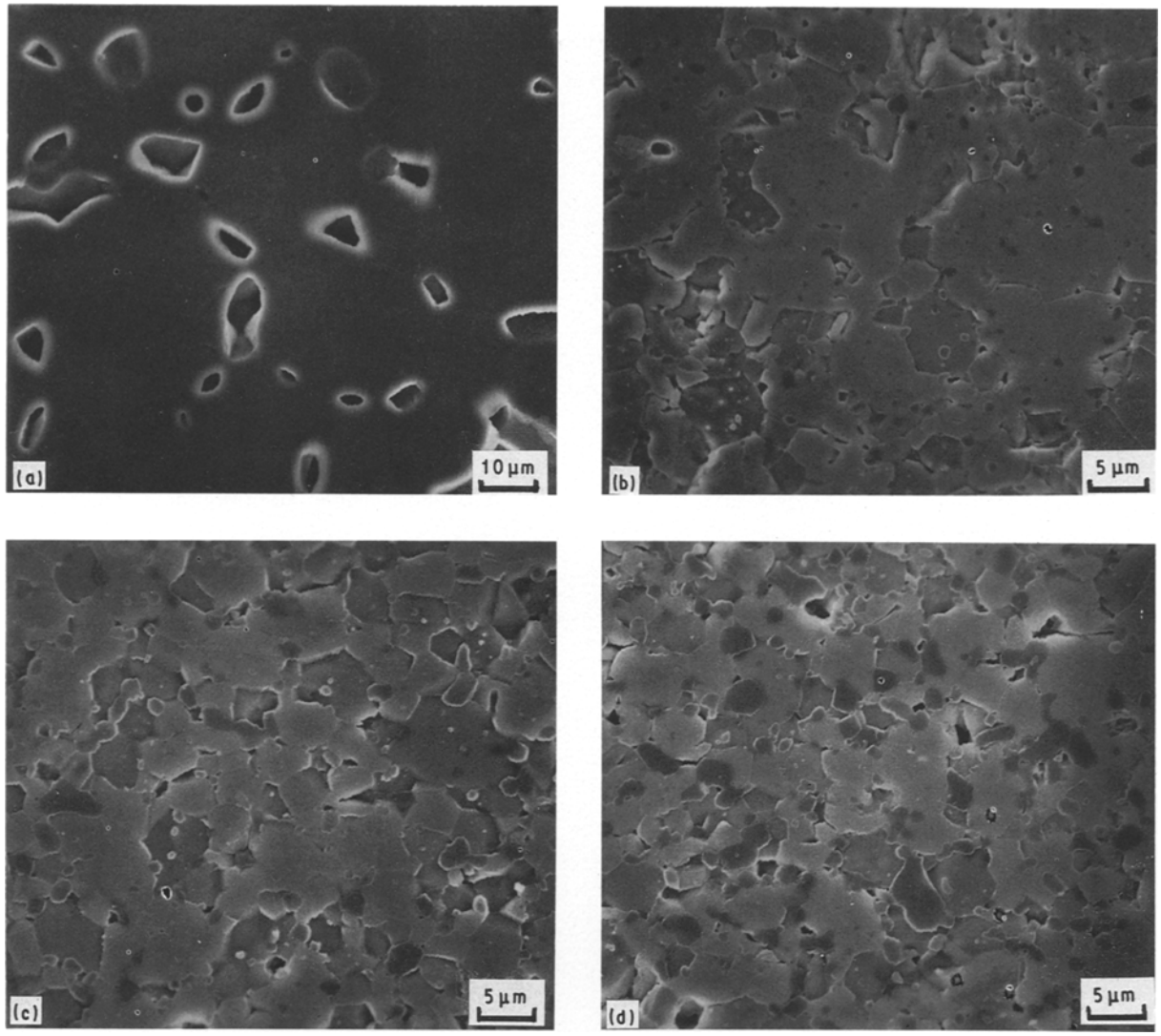

Figure 3 Scanning electron micrographs of Al_2O_3–B_4C ceramics sintered at 1850 °C for 60 min with (a) 0, (b) 5, (c) 10 and (d) 20 vol % B_4C.

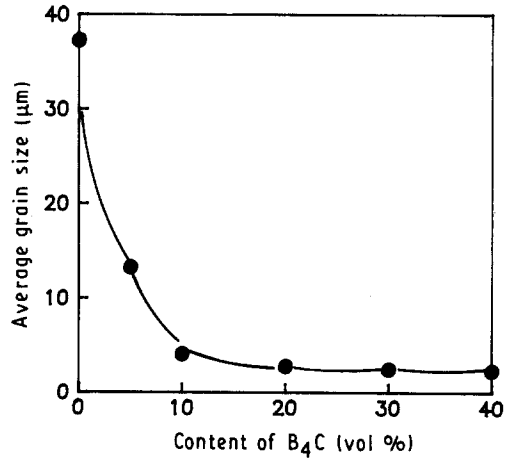

Figure 4 Effect of B_4C content on the grain size of Al_2O_3–B_4C ceramics sintered at 1850 °C for 60 min.

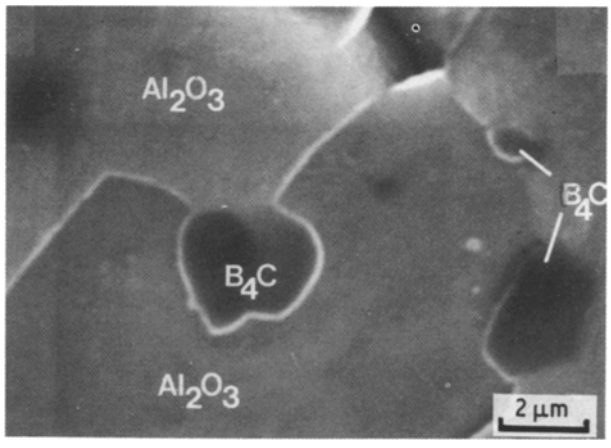

Figure 5 Microstructure of an Al_2O_3–B_4C specimen.

1850 °C after 30 min and the subsequent sintering of up to 120 min produced no change in sintered density. Therefore, when the sintering temperature was 1850 °C, the optimum value of relative density of these composites was obtained by sintering for 60 min.

3.2. Characterization

Fig. 7 shows the flexural strength for the specimens which were sintered at 1850 °C for 60 min. It was found that the maximum value was 550 MPa at 20 vol % B_4C and is caused by the decrease in poros-

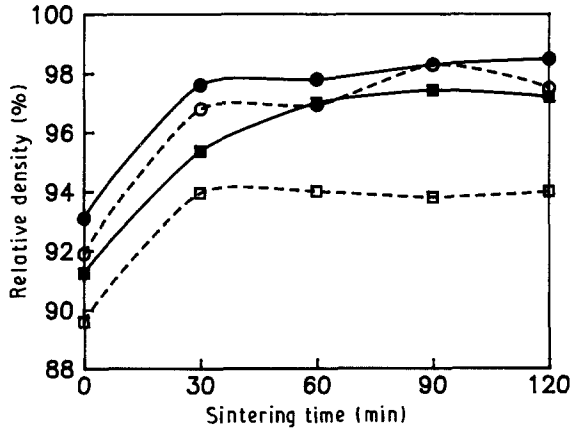

Figure 6 Variation of sintered densities with sintering time of Al_2O_3–B_4C ceramics at 1850 °C. (●) 5, (○) 10, (■) 20 and (□) 30 vol % B_4C.

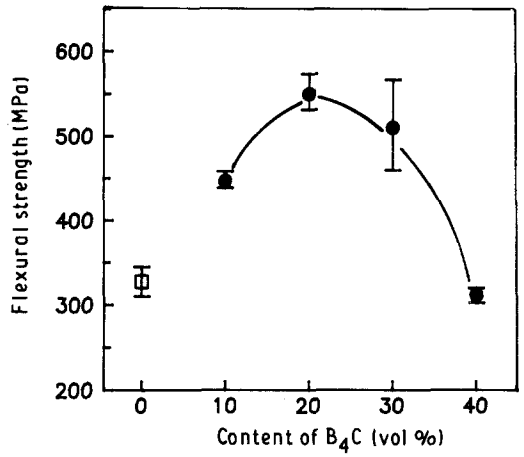

Figure 7 Effect of B_4C content on the flexural strength of Al_2O_3–B_4C ceramics sintered at 1850 °C for 60 min. (□) Al_2O_3–0.1 wt % MgO, sintered at 1575 °C for 120 min in air.

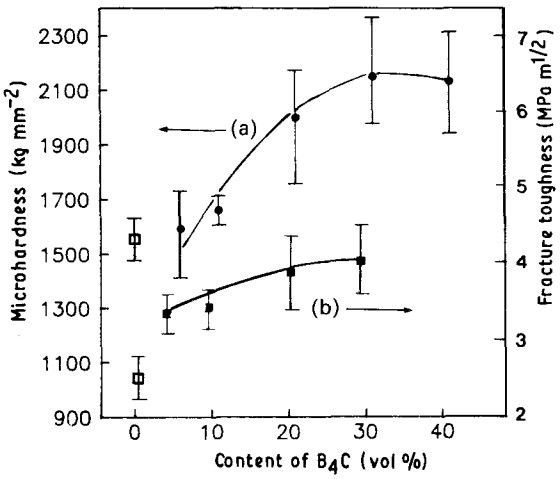

Figure 8 Effect of B_4C content on (●) the microhardness and (■) fracture toughness of Al_2O_3–B_4C ceramics sintered at 1850 °C for 60 min. (□) Al_2O_3–0.1 wt % MgO sintered at 1575 °C for 120 min in air.

ity and grain size as occurs in most ceramic materials [12, 13]. Such maximum flexural strength is higher than that of Al_2O_3–0.1 wt % MgO ceramics having similar grain size which were sintered at 1575 °C for 120 min in air. It is believed, therefore, that the addition of B_4C confers much improvement on flexural strength.

Fig. 8a shows the variation of Micro-Vickers hardness with varying B_4C content. The hardness increases with increasing B_4C content up to 30 vol %. The largest value of hardness exceeded that of Al_2O_3 (1600 kg mm^{-2}) and reached 2100 kg mm^{-2}, indicating that B_4C addition improves hardness. Fracture toughness slightly increases with increasing B_4C content up to 20 vol % (Fig. 8b), due to crack deflection around the B_4C particles [14]. Consequently, the maximum value of toughness reached was 4 MPa $m^{1/2}$.

4. Conclusion

The sintering behaviour of the Al_2O_3–B_4C composites has been studied at 1850 °C for 0–120 min in an argon flowing atmosphere. An increased densification was observed in the composites with 5–20 vol % B_4C. The role of B_4C in these composites was to inhibit rapid grain growth of Al_2O_3 at this temperature, so that B_4C behaved as a grain-boundary pinning effect.

The maximum value of flexural strength was 550 MPa at 20 vol % B_4C, caused by the low porosity and smaller grain size. The microhardness and toughness values reached were 2100 kg mm^{-2} and 4 MPa $m^{1/2}$, respectively.

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