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In-situ compaction and sintering of Al_2O_3-B_4C composites by using a High-Frequency Induction System

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Abstract

Alumina powders containing varying ratios of B_4C were mixed in a V-type mixer for 30 min with 35 cycle/min to obtain a homogeneous mixture. Six different chemical compositions were prepared and sintered at 1600 °C for 20 min using an Ultra-High-Frequency Induction Heated System. The samples were cooled by air. The density, surface roughness, hardness and microstructure of samples were investigated. The results showed that when the amount of B_4C increased, the values of hardness increased and the values of surface roughness decreased.

K e y w o r d s: alumina, boron carbide, sintering, induction, composite

1. Introduction

Comparing powder metallurgy (PM) with other conventional manufacturing technologies, machining process is not necessary requirement, as standard parts with complex geometry in a wide composition range can be manufactured serially. This production method has many advantages, such as low investment costs, the equipment used is flexible, high productivity, and it is easy to produce materials with different properties [1–3].

Alumina oxide is a chemical composition made up of two units of aluminium and three units of oxygen with the chemical formula Al_2O_3. Aluminium oxide is one of the most used materials in the industry because of its superior hardness, chemical inertness and electrical/thermal insulation properties [4]. Alumina is used in ceramic, glass, refractory industry, grinder material production, primary aluminium production, transparent armour production and ceramic cutting production [5–7].

Boron carbide (chemical formula B_4C) is generally used in tank armour and engine sabotage powders in industrial applications [8]. It is a highly durable product. The melting temperature, boiling point and Young's modulus of B_4C are 2450 and 3500 °C and 450–470 GPa, respectively [9–11].

Sintering is a heating process applied to a powder compact to strengthen and bond it. The temperature used for sintering is below the melting temperature of the major constituent of the Powder Metallurgy (PM) material [12, 13]. Spark Plasma Sintering (SPS) is similar to microwave sintering, inducing fast sintering due to its own thermal environment. Generally, this method is performed on Al_2O_3, SiC and ZrO_2 [14, 15]. In the induction sintering system, compacts are heated very quickly. The High-Frequency Induction Heated Sintering (HFIHS) process is similar to SPS. This method is usually applied to ceramic materials [16].

Al_2O_3-14wt.%B_4C composites with 0-1wt.%C addition were sintered in an Ar atmosphere at 1550–1650 °C by Huang et al. The densification behaviour and microstructure of the Al_2O_3-B_4C composites were investigated [17].

Al_2O_3-TiC-B_4C powders were mixed in different proportions by Sun et al. A low-pressure hot pressing process was applied to all mixtures, then the mechanical properties of all the compacts were investigated. The Vickers hardness, fracture toughness and flexural strength of the composite with the addition of 4.7wt.%Al_2O_3 and 10wt.%TiC, were 24.8 GPa,

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Table 1. Chemical composition of Al_2O_3 and B_4C powders (wt.%)

Material	Al_2O_3	N_2O (max)	B	C	Fe_2O_3 (max)	SiO_2 (max)	Free B	CaO (max)	Free C	MgO (max)	TiO_2 (max)
Al_2O_3	99.73	0.14	–	–	0.03	0.01	–	0.02	–	0.01	0.02
B_4C	–	–	78	20.13	0.17	–	0.1	–	0.92	–	–

Table 2. The codes and chemical compositions of specimens

Code	Chemical compositions of specimens
B0	0 wt.% Boron carbide + 100 wt.% Pure alumina
B1	1 wt.% Boron carbide + 99 wt.% Pure alumina
B5	5 wt.% Boron carbide + 95 wt.% Pure alumina
B10	10 wt.% Boron carbide + 90 wt.% Pure alumina
B30	30 wt.% Boron carbide + 70 wt.% Pure alumina
B50	50 wt.% Boron carbide + 50 wt.% Pure alumina

Table 3. Processing parameters of a high-frequency induction heated compacts

Parameters	Applied value for induction sintering
Maximum temperature	1600 ℃
Power capacity	2.5 kW
Frequency	900 kHz
Pre-sintering time	Unavailable
Duration	30 s
Heating rate	$\sim 20\,℃\,s^{-1}$
Cooling rate	Naturally
Environment	Atmosphere

4.8 MPa $m^{1/2}$ and 445 MPa, respectively [18].

Al_2O_3-B_4C powders were sintered successfully with no pressure. The mechanical properties of the Al_2O_3-B_4C compacts were investigated. The results showed that when the amount of B4C increased, the density of the composites decreased.

2. Experimental method

Alumina (particle size of 2.3 μm, purity of 99.4 %) was used as the starting material. Boron carbide (B_4C) (average particle diameter of 63 μm, purity of 99.6 %) was supplied by Boroptik Engineering, R&D Manufacturing and Trade Co. The chemical composition of Al_2O_3 and B_4C is shown in Table 1.

All the powders' compositions, which are shown in Table 2 were mixed in a V-type mixer for 30 min at 35 cycles/min to obtain a homogeneous mixture. The size of the Al_2O_3-B_4C samples was ø 10 × 3 mm^2 and they weighed approximately 0.5 g.

Table 4. Density values of all compositions (Error Range [ER] is between ± 1 μm)

Code	Density ($g\,cm^{-3}$)
B0	3.476
B1	3.362
B5	3.319
B10	3.271
B30	2.985
B50	2.622

The mixtures of Al_2O_3-B_4C obtained were placed in the graphite die (outside diameter 45 mm; inside diameter 20 mm; height 18 mm). The induction sintering process was carried out using a 2.5 kW power supply at a frequency of 900 kHz. All compacts were sintered at 1600 ℃ for 20 min dwell time with an applied pressure of 30 MPa. Duana et al. [19] compacted their powders using the same sintering pressure. In contrast to Çavdar [20] and Çavdar and Atik [16] who used 5 minutes dwell time for induction sintering of the metallic materials, in this work a sintering dwell time of 20 min was used due to the sintering application of the ceramic composition. The temperature of the compacts was measured by an infrared thermometer (± 5 ℃) throughout the induction sintering process. After the completion of the sintering process, all compacts were cooled by air. All the sintering parameters are given in Table 3.

The relative densities of compacts were evaluated by the Archimedes' Method. Radwag AS 220/C/2 Archimedes' Scale was used for this test. Surface roughness was measured with the Mitutoyo surf test SJ-301 Surface Test Device. After the surface roughness test, an abrasion test was conducted on the specimens. In this study, a WC ball was used and applied at a normal load of 10 N with a sliding speed of 20 cm s^{-1} for 1000 m. The weight loss of the specimens was calculated and the results are given in Table 5.

The surface hardness was evaluated using the FM 700 Micro-hardness Tester with an automatic load of 31.25 kg and a dwell time of 12 s. The values of Vickers hardness were obtained. The Metkon Forcipol IV was used for etching and polishing. Micro-structural characterisation was made on the product samples which had been polished and etched using a solution of

Table 5. Average surface roughness (μm) (ER is between ± 1 μm) and weight loss (%) (ER is between ± 0.5 %) of sintered samples

Code	*Ra*	*Ry*	*Rz*	Weight loss
B0	2.08	21.13	14.15	–2.06
B1	2.37	21.48	14.59	–1.99
B5	2.62	21.84	14.94	–1.62
B10	3.27	22.45	16.08	–0.99
B30	4.01	24.17	17.30	+0.32
B50	5.12	25.42	18.64	+0.41

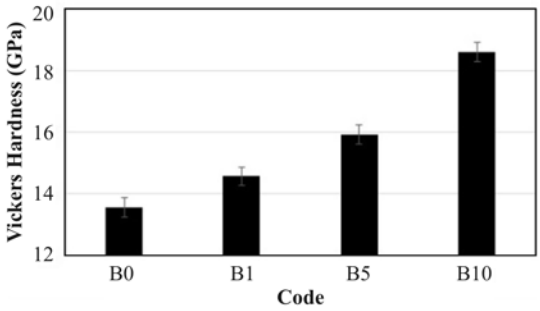

Fig. 1. The average hardness of sintered composites.

C_2H_6O (97 vol.%), HNO_3 (3 vol.%). The microstructures were observed using a MEIJO metal microscope and the images obtained are shown in Figs. 2, 3.

3. Results and discussion

The relative densities of all compacts were measured by using the Archimedes' method. The theoretical density of Al_2O_3 and B_4C is 3.95 and 2.51 g cm^{-3}. It was observed that density values changed between 3.476 and 2.62276 g cm^{-3} as shown in Table 4, and the percentage of density values changed from 88 to 81.2 %. Sun et al. [21] obtained a higher concentration of Al_2O_3-B_4C composite as the sintering process was carried out at a higher temperature. The results show that the relative density of the specimens rapidly decreased when the amount of B_4C increased. The melting temperature of alumina and Al_2O_3 is 2072 and 2500 ℃. Alumina was sintered better than B_4C at 1600 ℃. Thus, the highest density was obtained with pure alumina.

The surface roughness test was applied to all sintered compacts. The surface roughness values are shown in Table 5. In the same table, *Ra* is the arithmetic average of the absolute values, *Ry* means the highest micro-peak to the lowest micro-trough vertical distance within a single sample length, *Rz* is a parameter that averages the height of the five highest micro-peaks plus the depth of the five deepest micro-troughs over the evaluation length [22]. According to the results, when the amount of B_4C increased, surface roughness values increased. This is because the grain size of B_4C is bigger than alumina's and hence the porosity of the material is greater.

The wear test was applied to the samples. Before and after the wear test, their weights were measured. Percentage weight loss was calculated and is given in Table 5. As shown in the table, when the amount of B_4C increased, the percentage weight loss decreased. For the 30 and 50wt.%B_4C reinforced compacts, the weight of the specimens increased. Due to the extreme increase in hardness and brittleness of both compacts,

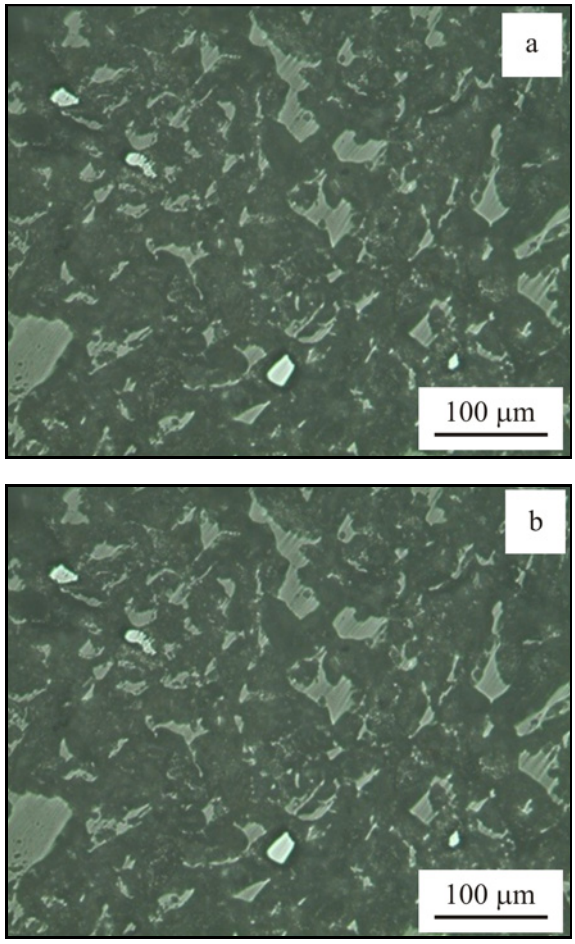

Fig. 2. (a) 1wt.%B_4C-99 wt.% Al_2O_3 and (b) 5wt.%B_4C--95wt.%Al_2O_3 compacts were taken from the centre of the surface of the microstructural images.

abrasion was seen on the ceramic balls' counter-bodies for these two compositions. These results demonstrate that the hardness of the compacts is higher than the hardness of the ceramic balls.

Vickers hardness was taken from five different points and the average values calculated are given in Fig. 1. According to results, when the amount of B_4C increased, the hardness of sintered compacts in-

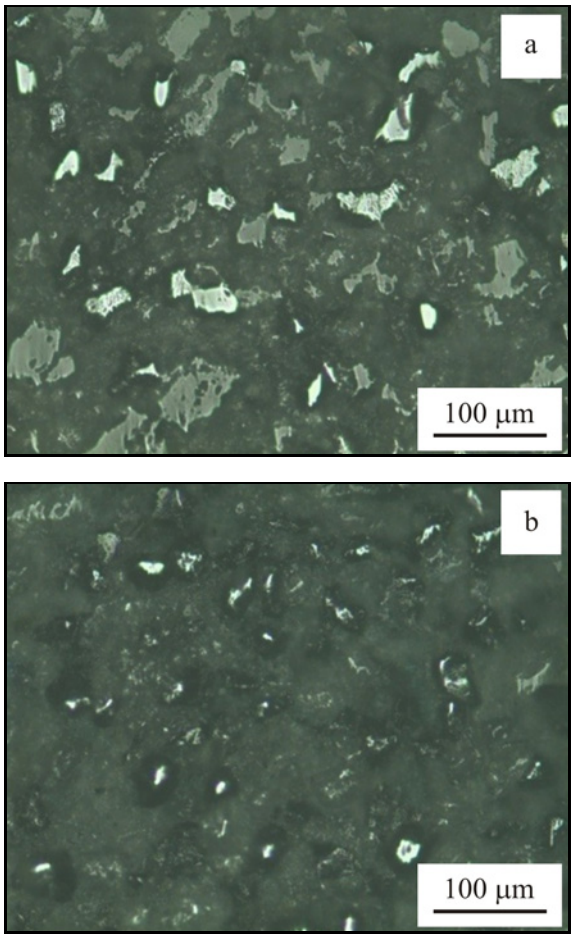

Fig. 3. (a) 10wt.%B_4C-90wt.%Al_2O_3 and (b) 30wt.%B_4C-70Al_2O_3 compacts were taken near the broken surface of microstructural images.

creased. This is because the hardness of B_4C is higher than that of the alumina. Also, when the amount of B_4C increased, the sintering temperature of alumina and boron carbide changed which showed us that we had to consider the alumina and boron carbide sintering temperature, too. The sintering temperature of alumina is less than that of B_4C. The hardness values of 70wt.%Al_2O_3-30wt.%B_4C (B30) and 50wt.%Al_2O_3-50wt.%B_4C (B50) compacts could not be measured due to the formation of weak bonds between the grains during the sintering process, and hence the compacts broke. Sun et al. [23] investigated the effects of alumina addition to boron carbide ceramics prepared by the SPS technique. They used sintering temperatures from 1700 to 1800 ℃. In contrast to this study, we sintered our compacts at 1600 ℃. Consequently, the broken B30 and B50 compacts indicate that our sintering temperature was inadequate for boron carbide reinforcements above 10 wt.%.

The microstructures of grain boundaries of sintered pellets are shown in Figs. 2, 3. It was seen that additive material of B_4C is dispersed homogeneously in the composite. It is also seen that the denser the mixture, B_4C dispersed more uniformly in Al_2O_3 30 wt.% and 50 wt.% B_4C containing Al_2O_3 compacts, shown in the images taken near the broken surface of the microscope image (Figs. 3a,b). The rate of porosity increased, and weak bonds between grains occurred during the sintering process due to increasing the amount of B_4C. For this reason, the composite had a more brittle structure.

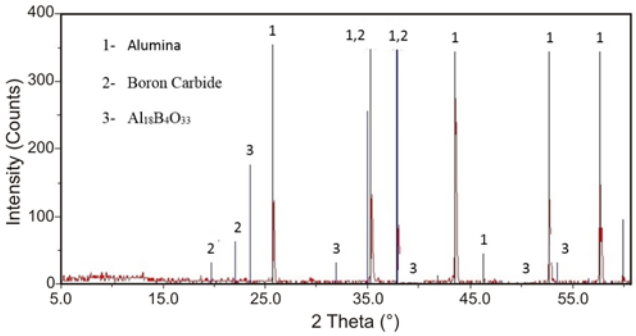

Fig. 4. XRD patterns of Al_2O_3-B_4C composite.

In-situ observation of the dark scattered images revealed that the microstructures of Al_2O_3 ceramics contained a white B_4C matrix with a dispersed grey phase as shown in Figs. 2, 3. Based on XRD analysis, the grey phase was identified as Al_2O_3-B_4C ceramics.

Figure 4 summarises the results of the XRD phase identification of ultra-high frequency induction sintered Al_2O_3-B_4C ceramic. Previous reports confirmed the formation temperature (about 1100 ℃) of $Al_{18}B_4O_{33}$ phase by reaction (Eq. (1)) [24, 25]. After the sintering process, these studies detected the same $Al_{18}B_4O_{33}$ phase in our composition. During sintering, the reaction occurred between Al_2O_3 and B_4C. This reaction is given by Eq. (1):

$$9Al_2O_3 + 2B_4O_{33} \rightarrow Al_{18}B_4O_{33}. \quad (1)$$

4. Conclusions

In this study, the mechanical properties of high-frequency induction heated sintered B_4C powder of different weights were studied. The effect of B_4C on alumina was investigated. The following results were obtained:

– The surface roughness value reached a maximum level at 50wt.%B_4C-50Al_2O_3 compacts.

– Highest density was obtained for pure alumina and highest hardness result was obtained for Al_2O_3-10wt.%B_4C.

– As the amount of B_4C increased, the compos-

ite structure became more brittle. Hence, fracturing occurred during the measurement of hardness.

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