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Research Article

## The Influence of Particle Size and Reinforcement Rate of B<sub>4</sub>C on Mechanical and Microstructure Properties of Al-B<sub>4</sub>C Composites

 Mahmut Can ŞENEL\*,  Mevlüt GÜRBÜZ

Mechanical Engineering Department, Faculty of Engineering, Ondokuz Mayıs University, Samsun, TURKEY

\* Corresponding author: mahmutcan.senel@omu.edu.tr

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### ABSTRACT

In this study, Al-B<sub>4</sub>C composites were produced with various particle sizes (B<sub>4</sub>C: 3.5 and 20 µm) and reinforcement rates (B<sub>4</sub>C%: 1, 3, 6, 9, 12, 15, 30%) by the powder metallurgy method. The apparent density, compressive strength, and Vickers hardness of the composites were determined by Archimedes' density meter, universal test machine, and micro Vickers hardness measurement device, respectively. The phase and microstructural analysis of the fabricated composites were analyzed using an X-ray diffraction device and scanning electron microscope, respectively. From the test results, the highest micro Vickers hardness (68.8 HV), apparent density (2.61 g/cm<sup>3</sup>), compressive strength (242 MPa), and minimum porosity (1.4%) were determined at 3.5 µm particle size and 30% reinforcement rate of B<sub>4</sub>C. The enhancement in Vickers hardness and compressive strength of Al-30%B<sub>4</sub>C composite was detected as +129.3% and +165.9% compared with pure aluminum.

**Keywords:** Aluminum, Boron Carbide, Composite, Hardness, Microstructure

## Partikül Boyutunun ve B<sub>4</sub>C Katkı Oranının Al-B<sub>4</sub>C Kompozitlerin Mekanik ve Mikroyapı Özellikleri Üzerine Olan Etkisi

### ÖZET

Bu çalışmada, Al-B<sub>4</sub>C kompozitler farklı partikül boyutlarında (B<sub>4</sub>C: 3.5 ve 20 µm) ve takviye oranlarında (% B<sub>4</sub>C: 1, 3, 6, 9, 12, 15, 30) toz metalürjisi yöntemiyle üretilmiştir. Kompozitlerin görünür yoğunluğu, basma dayanımı ve Vickers sertliği sırasıyla; Arşimet yoğunluk ölçüm cihazı, universal test makinesi ve mikro Vickers sertlik ölçüm cihazıyla belirlenmiştir. Üretilen kompozitlerin faz ve mikro-yapı analizi sırasıyla; X-ışını kırınımı cihazı ve taramalı elektron mikroskobu kullanılarak analiz edilmiştir. Test sonuçlarına göre; en yüksek mikro Vickers sertliği (68.8 HV), görünür yoğunluk (2.61 g/cm<sup>3</sup>), basma dayanımı (242 MPa) ve en düşük gözeneklilik oranı (% 1.4) %30 B<sub>4</sub>C katkı oranına ve 3.5 µm B<sub>4</sub>C partikül boyutuna sahip Al-B<sub>4</sub>C kompozit yapıda elde edilmiştir. Saf alüminyum ile karşılaştırıldığında Al-%30B<sub>4</sub>C kompozitin Vickers sertliğinin ve basma dayanımının sırasıyla; % 129.3 ve % 165.9 oranında arttığı tespit edilmiştir.

**Anahtar Kelimeler:** Alüminyum, Bor Karbür, Kompozit, Sertlik, Mikroyapı

## **I. INTRODUCTION**

Metal matrix composites (MMCs) can be reinforced with a ceramic or a carbon-based material into a metal matrix material to improve its strength, corrosion resistance, and wear resistance [1-3]. The most preferred matrix materials are magnesium (Mg), aluminum (Al), and its alloys. Among matrix materials, aluminum is the most commonly used metal for their good elasticity modulus (~68 GPa), excellent machinability, low density (~2.7 g/cm<sup>3</sup>), excellent corrosion resistance, and high toughness, especially in automotive and aerospace applications such as construction and engine blocks [4-6]. Also, metal oxides (Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub>, etc.), metal nitrides (TiN, Si<sub>3</sub>N<sub>4</sub>, TaN, ZrN, etc.), metal carbides (B<sub>4</sub>C, SiC, WC, etc.), and carbon-based materials (graphene nanoplatelets (GNPs), fullerenes, carbon nanotube (CNT)) can be used as the reinforcement material [7]. Among ceramic materials, boron carbide (B<sub>4</sub>C) has high Young's modulus (~441x10<sup>3</sup> MPa), high hardness (~3800 HV), high melting point (~2400 °C), and low density (~2.52 g/cm<sup>3</sup>) [8, 9]. The use of pure boron carbide (B<sub>4</sub>C) as a structural material is restricted because of its brittle structure. Hence, B<sub>4</sub>C was preferred as a particle reinforcement material in aluminum matrix composites. B<sub>4</sub>C reinforced aluminum composites have been utilized from the fields of cycling industrial, aviation industry, and electronic communication due to the functional damping capacity, excellent thermal conductivity, high strength, low density, and high specific stiffness [10, 11].

In general, the squeeze casting, melting, and powder metallurgy (PM) can be used to produce the MMCs [12]. Among these methods, the PM method is remarkable because of its ability to fabricating large-size and complex machine parts [13-15]. Especially, aluminum-based composites might be produced by the PM method due to the widespread use of aluminum in the industrial areas such as in the automotive, aviation, transportation, and space industries [16, 17].

Many researchers have made successful attempts to investigate the production, mechanical, and microstructural properties of Al-B<sub>4</sub>C composites [18-24]. Khademian et al. [18] examined the influences of pouring temperature (700, 850, 950 °C) and stirring times (10, 15, 20 min) on the mechanical properties of A356-3%B<sub>4</sub>C using stir casting method. The optimum pouring temperature and stirring time were determined as 850 °C and 15 min, respectively. Pang et al. [19] studied the tensile properties of Al6061 alloy and Al6061-12%B<sub>4</sub>C-2.5%Al<sub>2</sub>O<sub>3</sub> composites. According to test results, the tensile and yield strength determined an increase of ~113% and ~246% when compared to those of the Al6061 alloy. Ipekoglu et al. [20] researched the effect of the reinforcement rates of B<sub>4</sub>C (0, 3, 5, 10wt%) and squeeze pressure (P=0, 75, 150 MPa) on the mechanical strength of Al-B<sub>4</sub>C composites using the squeeze casting method. The highest mechanical properties (yield stress, hardness, and tensile stress) were detected at the amount of 5wt%B<sub>4</sub>C and the squeeze pressure of 150 MPa. To the best of our knowledge, no study is available to investigate the microstructural and mechanical properties of B<sub>4</sub>C reinforced aluminum composites with different particle sizes and contents of B<sub>4</sub>C fabricated by the powder metallurgy method.

The purpose of this study was to fabricate pure Al and B<sub>4</sub>C reinforced aluminum composites with different content (B<sub>4</sub>C: 1, 3, 6, 9, 12, 15, 30wt%;) and particle size (B<sub>4</sub>C: 3.5 and 20 μm) by the powder metallurgy method. The present study aimed to examine the influences of reinforcement rate and particle size of B<sub>4</sub>C on the microstructure, porosity, hardness, apparent density, and compressive strength of Al-B<sub>4</sub>C composites.

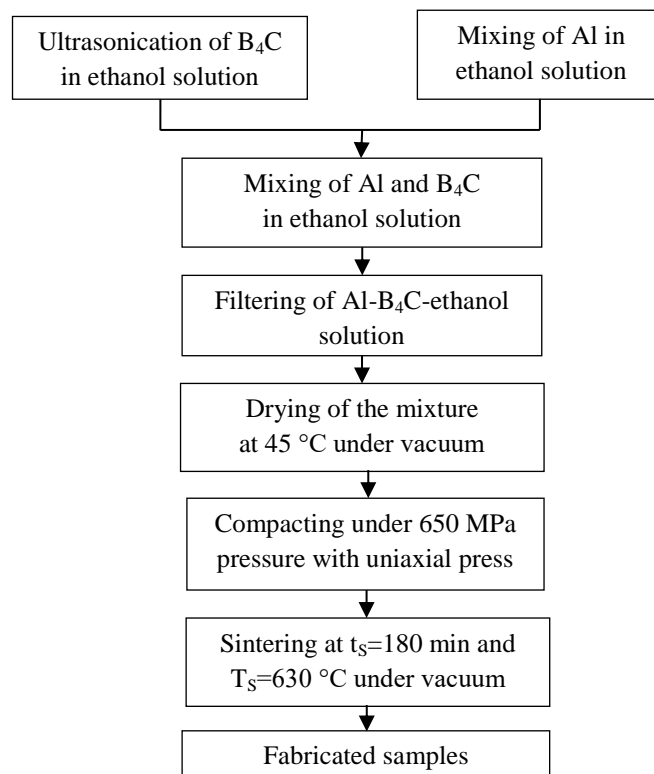
## II. MATERIALS AND METHOD

### A. MATERIALS

In the present study, B<sub>4</sub>C powders (the theoretical density of 2.52 g/cm<sup>3</sup>, the mean particle sizes of ~3.5 μm and ~20 μm, and the purity of 99%) were used as the reinforcement material while atomized pure aluminum powders (the theoretical density of 2.7 g/cm<sup>3</sup>, the particle diameter of ~10 μm, the purity of 99%) were used as the matrix material. Aluminum and B<sub>4</sub>C powders were provided by Alfa Aesar Inc. (United Kingdom), respectively.

### B. METHOD

Figure 1 shows the fabrication scheme of boron carbide reinforced aluminum-based composites by powder metallurgy method. Al powders were mixed in ethanol using the mechanical mixer. At the same time, boron carbide powders (1, 3, 6, 9, 12, 15, 30 wt.%) were ultrasonicated in ethanol by using an ultrasonic dispenser. Then, the boron carbide solution was added to the aluminum solution. The solution was filtered and dried at 45 °C for 12 h. The powders were compacted in stainless steel die under a pressure of 650 MPa. Then, the samples were sintered in a tube furnace for the sintering time ( $t_s=180$  min) and the sintering temperature ( $T_s=630$  °C) under vacuum.



*Figure 1. The fabrication scheme of Al-B<sub>4</sub>C composites by the PM method [25]*

The microstructure of composites and powders was determined by using a scanning electron microscope (SEM, Jeol JSM-7001F). SEM analyses were actualized to determine the distribution of boron carbide in the Al matrix. X-ray Diffraction (XRD, Rigaku Smartlab) analysis was used to examine the phases in composites and powders between 20 and 80°. In XRD analysis, raw data were

analyzed afterward, refined by MDI Jade 6.0 program. The apparent densities of the composites were determined by Archimedes' method. The density results were averaged over at least six measurements. The apparent density ( $\rho_D$ ) can be stated by Eq. (1) [26]:

$$\rho_D = [m_K / (m_D - m_A)] \rho_W \quad (1)$$

where  $\rho_W$  is the water density,  $m_D$  is the mass of water-saturated composite,  $m_K$  is the mass of fabricated composite, and  $m_A$  is the mass of the composite submerged in the water.

The theoretical density of the composites ( $\rho_T$ ) can be determined by Eq. (2):

$$\rho_T = (m_{Al}\% \times \rho_{Al}) + (m_{B_4C}\% \times \rho_{B_4C}) \quad (2)$$

where  $m_{B_4C}\%$  and  $m_{Al}\%$  are defined as the mass fraction of  $B_4C$  and pure Al, respectively. Also,  $\rho_{B_4C}$  ( $2.52 \text{ g/cm}^3$ ) and  $\rho_{Al}$  ( $2.7 \text{ g/cm}^3$ ) are defined as the theoretical density of  $B_4C$  and pure aluminum, respectively.

The percentage of porosity for the Al- $B_4C$  composites can be calculated by Eq. (3):

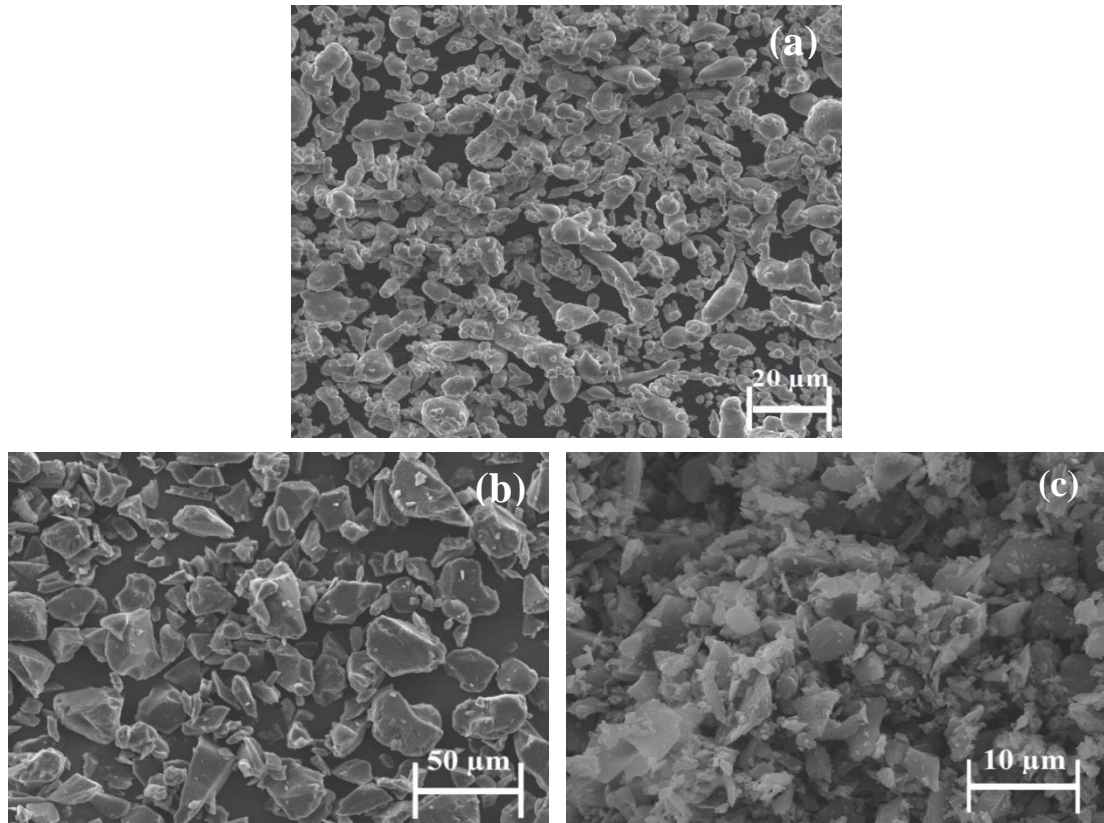
$$P\% = (1 - \rho_D / \rho_T) \times 100 \quad (3)$$

The hardness of composites was determined by using a micro Vickers hardness tester under a dwell time of 15 s and a load of 200 g. The measurement was actualized six times from random locations of the polished cross-sections, and then they were averaged. The compressive strength of the composites was detected by the compressive test unit (Mares Test-10 tons). By using this test unit, each sample was tested at least five times with the compression rate of 10 mm/min.

### **III. RESULTS AND DISCUSSION**

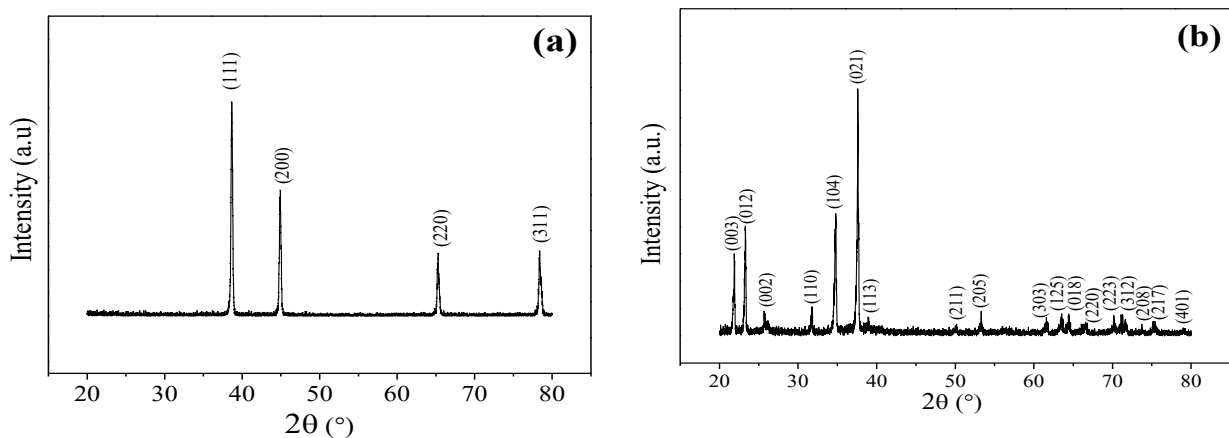
#### **A. CHARACTERIZATION OF POWDERS**

Scanning electron microscopy images of pure aluminum and boron carbide powders are presented in Figure 2. As seen from the figure, Al powders have nearly spherical morphology. On the other hand,  $B_4C$  powders have an irregular form with sharp edges (Figures 2(a-c)). The mean particle size of aluminum is detected as  $\sim 10 \text{ }\mu\text{m}$ . Also, the mean of boron carbide powders is determined as  $\sim 20 \text{ }\mu\text{m}$  and  $\sim 3.5 \text{ }\mu\text{m}$ , respectively (Figures 2(b) and (c)).



**Figure 2.** SEM images of pure Al (a) and B<sub>4</sub>C particles (particle sizes of B<sub>4</sub>C: 20 μm (b) and 3.5 μm (c))

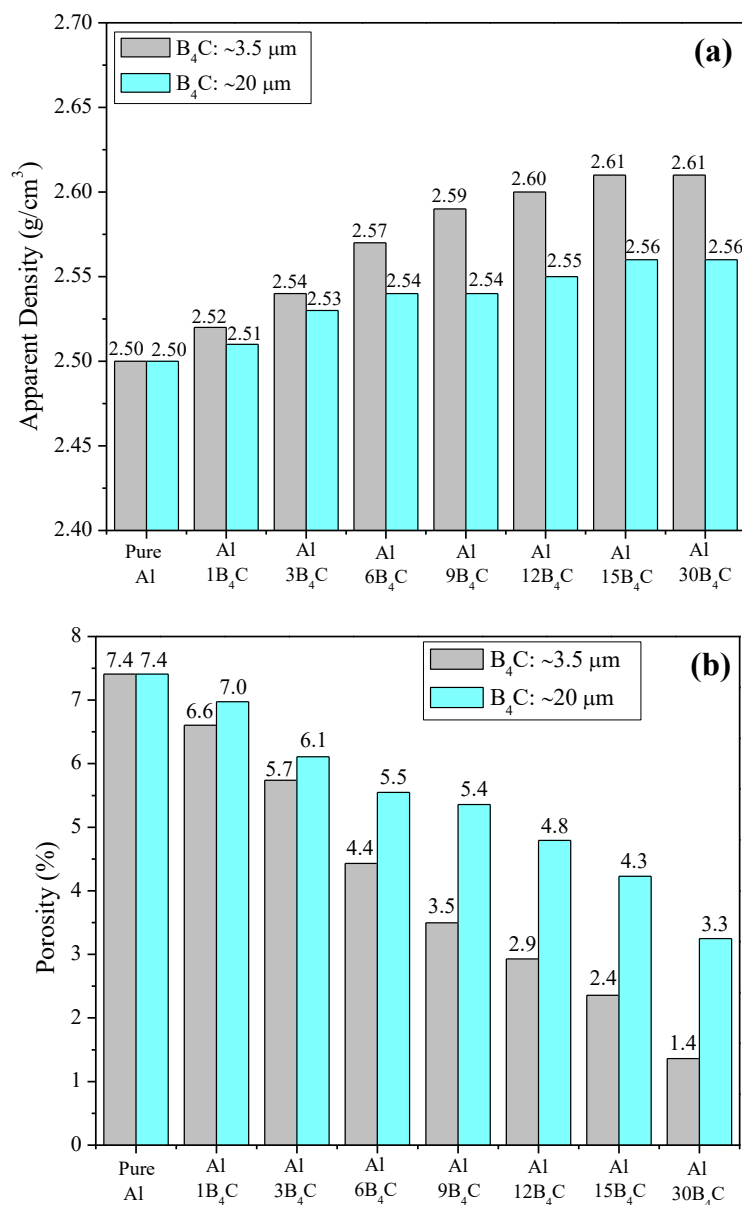
XRD plots of pure Al and B<sub>4</sub>C were given in Figures 3(a-b), respectively. As shown in these figures, aluminum and B<sub>4</sub>C peaks are presented at  $2\theta \approx 39^\circ, 45^\circ, 65^\circ, 78^\circ$  and  $2\theta \approx 22^\circ, 23^\circ, 26^\circ, 32^\circ, 35^\circ, 38^\circ, 39^\circ, 50^\circ, 54^\circ, 62^\circ, 64^\circ, 65^\circ, 67^\circ, 70^\circ, 72^\circ, 74^\circ, 75^\circ, 78^\circ$ , respectively. The phase analyses of pure Al and B<sub>4</sub>C particles are an important process to determine the phases of composites after sintering. Also, these analyses give information related to the reaction between the reinforcement and matrix materials after sintering.



**Figure 3.** XRD analyses pure Al (a) and B<sub>4</sub>C (b) particles

## B. MECHANICAL PROPERTIES OF COMPOSITES

Figure 4 gives the variation of apparent density and porosity for pure Al and Al-B<sub>4</sub>C composites, respectively. As illustrated in Figure 4(a), increasing B<sub>4</sub>C content in the aluminum matrix improved the apparent density of Al-B<sub>4</sub>C composites. Besides, the decrease in the particle size of the reinforcement material increased the apparent density and decreased the porosity of Al-B<sub>4</sub>C composite (Figure 4(b)). The highest density and lowest porosity of the Al-B<sub>4</sub>C composite were determined as 2.61 g/cm<sup>3</sup> and 1.4% at Al-B<sub>4</sub>C composite with the B<sub>4</sub>C particle size of 3.5 μm, respectively. There is an inverse relationship between the porosity and the mechanical properties of the composite. In other words, minimum porosity implies maximum mechanical properties. The density of the Al-B<sub>4</sub>C composites improved due to the homogeny dispersion of B<sub>4</sub>C in the matrix. It causes the increase of the contact area between the matrix and reinforcement element, which led to the lower porosity and higher density [27, 28].



**Figure 4.** Apparent density (a) and porosity (b) variation of pure Al and Al-B<sub>4</sub>C composites for different sizes of B<sub>4</sub>C particles

Figure 5 gives the Vickers hardness variation of pure Al and Al-B<sub>4</sub>C composites for different sizes of B<sub>4</sub>C particles. As shown in the figure, the Vickers hardness of Al-B<sub>4</sub>C composites improves with the increase in B<sub>4</sub>C amount due to the hard structure of B<sub>4</sub>C particles. The highest Vickers hardness (68.8 HV) was obtained at Al-30%B<sub>4</sub>C composite. The improvement in Vickers hardness is determined as ~129.3% compare with pure aluminum. Also, an increase in the particle size of B<sub>4</sub>C decreases the Vickers hardness of Al-B<sub>4</sub>C composites. The increase in the hardness of the composites can be expressed theoretically with the rule of mixtures by Eq. (4) [29].

$$H_c = H_m f_m + H_r f_r \quad (4)$$

where  $f_m$  and  $f_r$  are the volume fraction of matrix and reinforcement element,  $H_m$ ,  $H_c$ , and  $H_r$  are the hardness of the matrix, composite, and reinforcement element, respectively.

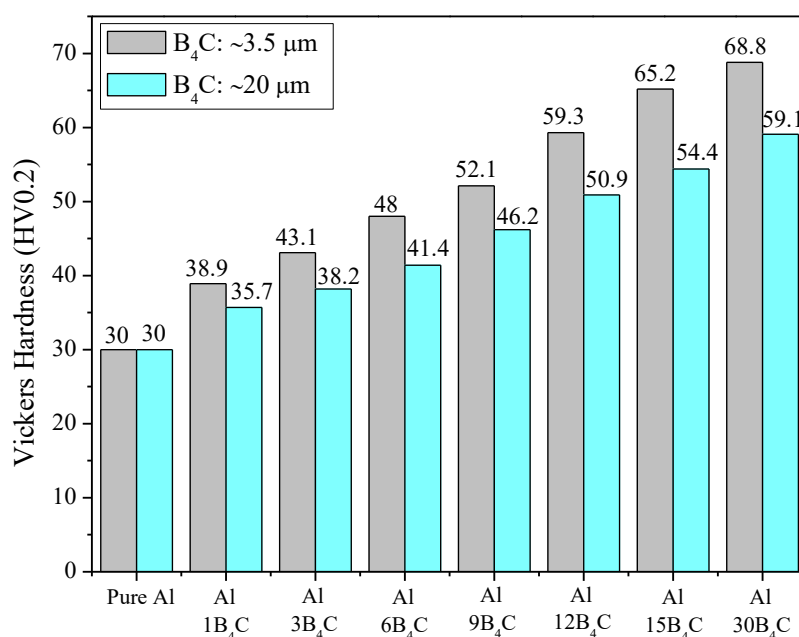
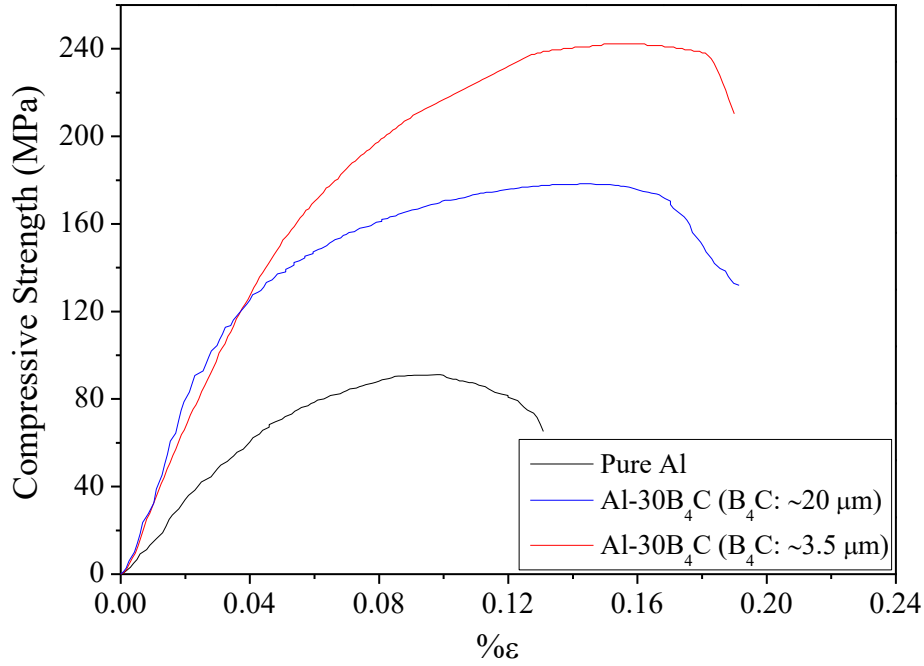


Figure 5. Vickers hardness variation of pure Al and Al-B<sub>4</sub>C composites for different sizes of B<sub>4</sub>C particles

The compressive strength variation of Al-B<sub>4</sub>C and pure Al particles is illustrated in Figure 6. As given in the figure, compressive strength was increased from 91 MPa (pure Al) to 242 MPa (Al-30B<sub>4</sub>C). Also, the decrease in the particle size of B<sub>4</sub>C from 20 μm to 3.5 μm improves the compressive strength of the fabricated composites from 178 MPa to 242 MPa. In the Al-B<sub>4</sub>C composite structure, small B<sub>4</sub>C particles act as an obstacle at the aluminum grain boundaries. It prevents the dislocation movement. The decrease between particles by the addition of B<sub>4</sub>C particles to the Al matrix declined, and then the dislocation movement blocked. Hence, the mechanical strength of Al-B<sub>4</sub>C composites improved. The increase in the strength of the composite can be stated as given in Eq. (5) [29]:

$$\sigma_c = \sigma_m f_m + \sigma_r f_r \quad (5)$$

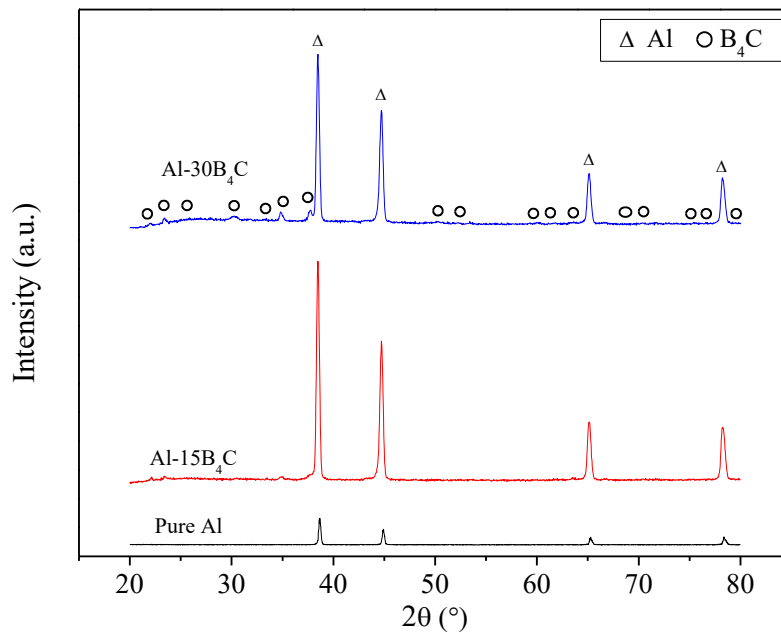
where  $f_m$ ,  $f_r$ , and  $f_c$  are the volume fraction of matrix, reinforcement, and composite material,  $\sigma_m$ ,  $\sigma_r$ , and  $\sigma_c$  are the strength of the matrix, reinforcement, and composite material, respectively.



**Figure 6.** Compressive strength variation of pure Al and Al-B<sub>4</sub>C composites for different sizes of B<sub>4</sub>C particles

### C. CHARACTERIZATION OF SINTERED COMPOSITES

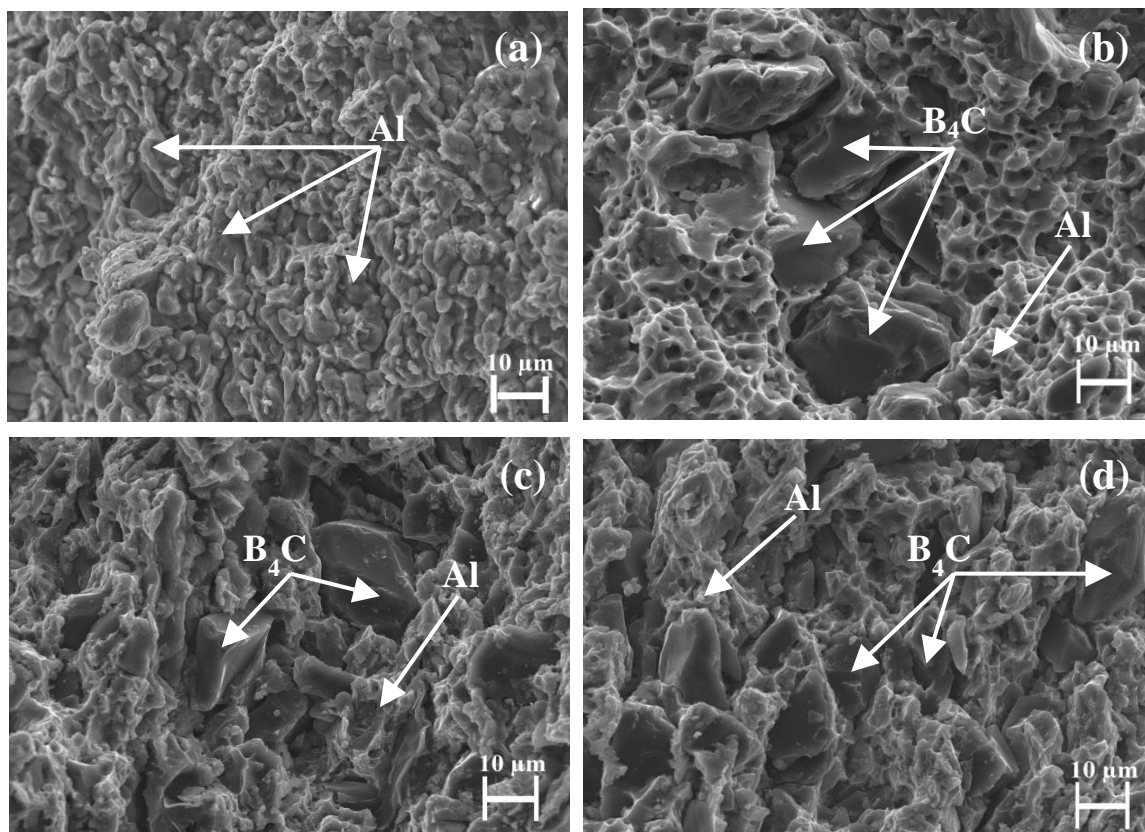
XRD patterns of Al-B<sub>4</sub>C composites and pure Al are presented in Figure 7. In order to determine the phase formation in Al matrix composites, XRD analysis was detected for pure Al, Al-15B<sub>4</sub>C, and Al-30B<sub>4</sub>C. As indicated in the figure, the intensity of B<sub>4</sub>C peaks in Al composites increased with increasing B<sub>4</sub>C content. The peak angles ( $2\theta \sim 22^\circ, 23^\circ, 26^\circ, 32^\circ, 35^\circ, 38^\circ, 39^\circ, 50^\circ, 54^\circ, 62^\circ, 64^\circ, 65^\circ, 67^\circ, 70^\circ, 72^\circ, 74^\circ, 75^\circ, 78^\circ$ ) of B<sub>4</sub>C were confirmed by the existence of B<sub>4</sub>C in Al-B<sub>4</sub>C composites. According to the XRD analysis, the undesired phase formation, such as Al<sub>4</sub>C<sub>3</sub> phases were not observed in any composition.



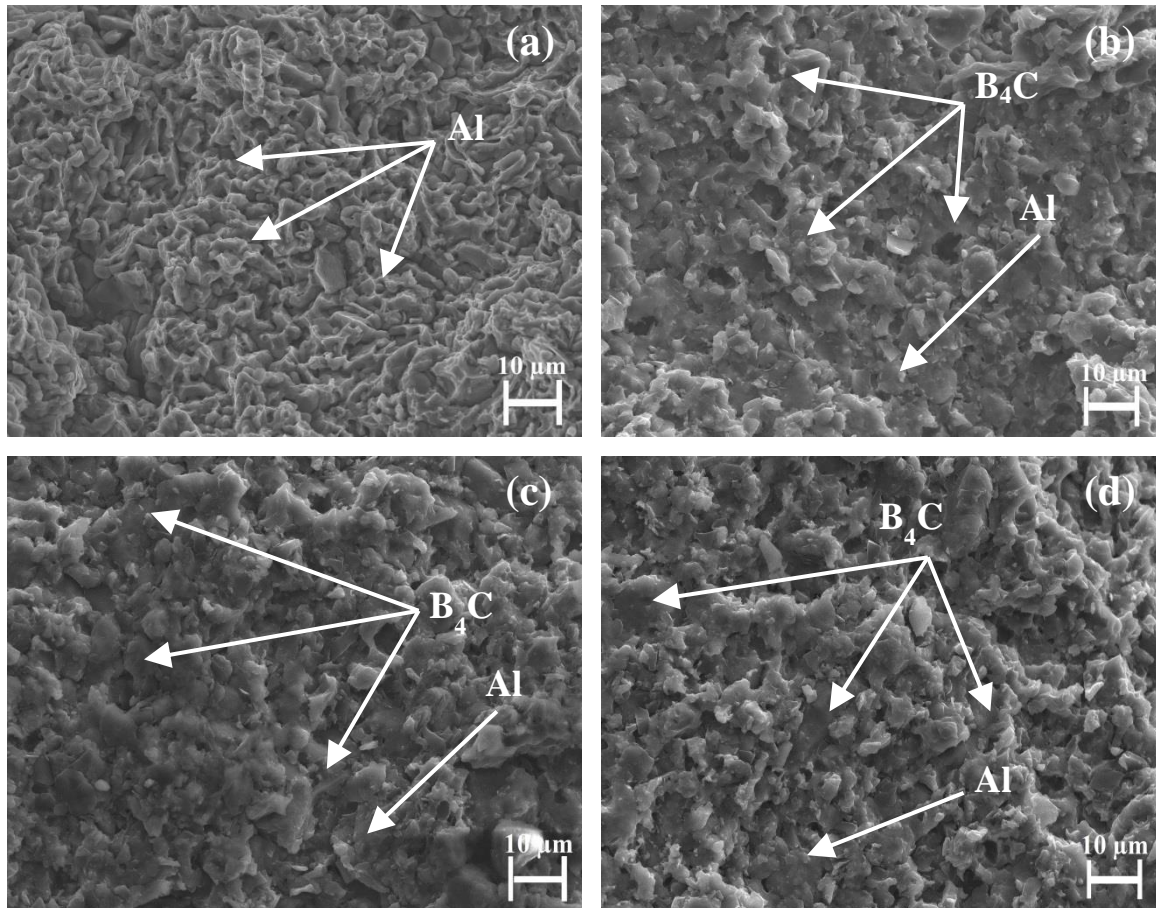
**Figure 7.** XRD plots of pure Al, Al-15B<sub>4</sub>C, and Al-30B<sub>4</sub>C composites



Scanning electron microscope (SEM) images of pure aluminum, Al-6B<sub>4</sub>C, Al-15B<sub>4</sub>C, and Al-30B<sub>4</sub>C composites for different sizes of B<sub>4</sub>C particles (B<sub>4</sub>C: 20 μm and 3.5 μm) are shown in Figures 8 and 9, respectively. As illustrated in Figure 8, it was clearly seen that B<sub>4</sub>C particles were distributed homogeneously and positioned at between aluminum boundaries. On the other hand, many pores were detected in the microstructure of the Al-B<sub>4</sub>C composite due to bigger particle sizes of B<sub>4</sub>C (Figure 8(b-d)). Figure 9 gives the SEM images of pure Al, Al-6B<sub>4</sub>C, Al-15B<sub>4</sub>C, and Al-30B<sub>4</sub>C with a smaller particle sizes of B<sub>4</sub>C (3.5 μm), respectively. The homogeneity distribution of B<sub>4</sub>C particles was detected from the SEM images of Al-B<sub>4</sub>C composites. Also, less porosity was observed due to the smaller size of B<sub>4</sub>C particles (Figure 9(b-d)). As a result, the mechanical properties (compressive strength and Vickers hardness) of B<sub>4</sub>C reinforced aluminum composites with smaller B<sub>4</sub>C particle size were better than the mechanical strength of B<sub>4</sub>C reinforced aluminum composites with larger B<sub>4</sub>C particle size.



**Figure 8.** SEM images of pure Al (a), Al-6B<sub>4</sub>C (b), Al-15B<sub>4</sub>C (c), and Al-30B<sub>4</sub>C (d) composites (particles size of B<sub>4</sub>C: 20 μm)



**Figure 9.** SEM images of pure Al (a), Al-6B<sub>4</sub>C (b), Al-15B<sub>4</sub>C (c), and Al-30B<sub>4</sub>C (d) composites (particles size of B<sub>4</sub>C: 3.5 μm)

## **IV. CONCLUSIONS**

In the present study, B<sub>4</sub>C reinforced aluminum matrix composites with various contents (1, 3, 6, 9, 12, 15, 30wt.%) and particle size (3.5 and 20 μm) of B<sub>4</sub>C were fabricated by the powder metallurgy method. The effect of the particle sizes and reinforcement rates of B<sub>4</sub>C on the Vickers hardness, the apparent density, porosity, compressive strength, and microstructure of Al-B<sub>4</sub>C composites were investigated. According to the test results, the highest apparent density (2.61 g/cm<sup>3</sup>), Vickers hardness (68.8 HV), compressive strength (242 MPa), and minimum porosity (1.4%) were obtained at Al-30%B<sub>4</sub>C composite with 3.5 μm particle size of B<sub>4</sub>C. Well-distributed small B<sub>4</sub>C particles act as an obstacle at the grain boundaries. Also, these particles prevent the grain growth during sintering. Hence, reinforcement of small B<sub>4</sub>C particles enhances the mechanical strength of Al-based composites owing to the hard structure and high compressive strength of B<sub>4</sub>C particles.

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