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Mechanical and Thermal Neutron Absorbing Properties of B₄C/Aluminum Alloy Composites Fabricated by Stir Casting and Hot Rolling Process

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Abstract: In this study, to fabricate neutron shielding material, boron carbide (B₄C)-reinforced aluminum (Al) alloy composites were successfully fabricated by stir casting followed by a hot rolling process. Microstructural analysis of B₄C/Al6061 composites with different volume fractions (5, 10, 20, 25, and 30%) revealed that the composites had volume ratios similar to the target volume ratios of B₄C. Furthermore, B₄C reinforcements were uniformly dispersed in the Al matrix, forming multi-interfacial layers of Al₄C₃/(Ti,Cr)B₂. The interfacial layer generated during stir casting maintained its own structure after the hot rolling process, indicating strong interfacial bonding strength. The tensile strengths of the B₄C/Al6061 composites increased to 20 vol.% and stayed above the value for Al6061, even reaching 30 vol.%. The measured thermal neutron shielding rate increased with increasing B₄C content, and the highest thermal neutron shielding rate was observed at 30 vol.% composite, which corresponds to 95.6% neutron shielding at 0.158-cm thickness.

Keywords: Al matrix composite; boron carbide; hot rolling; stir casting; thermal neutron shielding



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1. Introduction

Aluminum matrix composites (AMCs), due to their high specific strength, thermal stability, good wear, and weldability, have many advantages in applications in the electronics, vehicle, aerospace, military, marine, and nuclear industries [1]. By changing the type of aluminum (Al) alloy and the reinforcing material, AMCs can be manufactured according to the needs of the application in question [1,2]. Recently, AMCs, which are lighter and more suitable than the conventional materials used in certain industries, have been replaced, and ceramic particles such as B₄C [3], SiC [4], SiO₂ [5], TiC [6], TiB₂ [7], TiO₂ [8], Al₂O₃ [9], MgO [10], AlN [11], Gd₂O₃ [12], BN [13], etc., are used as reinforcement materials [1,14]. Among neutron absorber composites (NACs), B₄C/Al composites are used as shielding materials in transportation baskets, storage racks, and storage casks for spent nuclear fuel. Materials with excellent neutron absorption include Gd (44,000 barn) [15], ¹⁴⁹Sm (41,500 barn) [16], ¹⁰B (3850 barn) [17], and natural boron (760 barn) [15]. B₄C has a considerable amount of ¹⁰B (19.9%), and the neutron absorption cross-section is quite large [15]. It is reported that ceramics with excellent neutron absorption capacity, other than B₄C, have high density and poor mechanical stability during the composite manufacturing process, as well as being expensive [17]. B₄C, the reinforcing material used in this study, has the advantage of being easy to manufacture because its density is similar to that of aluminum (Al density: 2.7 g/cm³; B₄C density: 2.52 g/cm³). For this reason, B₄C-reinforced Al composites are often used in the nuclear shielding field.

Major fabrication methods of NACs include powder metallurgy, hot pressing, hot isostatic pressing, spark plasma sintering, coating, and stir casting [1,18]. Zhang et al. reported that B₄C/Al composites could be prepared using a powder metallurgy process, i.e., ball milling, followed by pressing, sintering, hot extrusion, and hot rolling [19]. Microstructure analysis and neutron radiography revealed that high-energy ball milling induced homogeneous distribution of B₄C particles in the Al matrix and good bonding between the Al matrix and the B₄C particles. Tariq et al. used cold spray technology to deposit a neutron shielding B₄C/Al composite coating on a 6061-t6 cylindrical substrate [20]. The microstructure, mechanical behavior, and neutron shielding performance of the coating were analyzed according to various heat treatment conditions. Abenojar et al. studied the effect of optimum compression pressure, internal density, and sintering atmosphere on powder metallurgy composite fabrication [21]. Harri Junaedi et al. investigated various strain rates and temperatures for the high-temperature deformation of the Al-15vol.% B₄C composite [22]. The analysis showed the presence of threshold stress and that its value increases with decreasing the testing temperature.

Among the various fabrication process of NACs, stir casting is an attractive method because it is simpler than other manufacturing methods and has the advantage of easy mass production. However, the B₄C content of stir cast composites was generally low (less than 20 vol.%) [23–25]. It is necessary to fabricate a B₄C/Al composite containing high B₄C content using a cost-effective stir casting process to increase functional properties such as neutron shielding, hardness, and modulus. Furthermore, uniform dispersion and precise content control of B₄C are extremely difficult due to poor wetting between B₄C/Al and loss of B₄C particles during the stir casting process. In this study, B₄C-reinforced Al6061 composites were manufactured by stir casting with the designed mold and stirring system followed by hot rolling.

2. Experimental Procedure

Al6061 alloy (ThyssenKrupp materials Korea Co., Ltd., Seoul, Korea) was used as a matrix to fabricate metal matrix composites (MMCs). The chemical composition of the Al6061 provided by the company is shown in Table 1. B₄C powders (Dunhua Zhengxing Abrasive Co., Ltd., Dunhua, China) with a commercial grade of F320 (Federation of European Producers of Abrasives (FEPA)-specified size distributions, 39.0 ± 1.5 µm) were used as reinforcement. The particle size (D₅₀) of the B₄C reinforcement, measured using laser diffraction spectroscopy (Sympatech HELOS, Clausthal-Zellerfeld, Germany), was 42.95 µm.

Table 1. Chemical composition of Al6061 alloy (wt.%).

Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.529	0.549	0.172	0.086	0.954	0.142	0.0061	0.031	97.450

B₄C/Al6061 composites with different volume fractions (5, 10, 20, 25, and 30%) were prepared by stir casting process, a well-known approach used to fabricate MMCs. The theoretical volume fraction of B₄C was calculated using the next equation.

$$V_{matrix} : V_{reinforcement} = \frac{W_{matrix}}{\rho_{matrix}} : \frac{W_{reinforcement}}{\rho_{reinforcement}} \quad (1)$$

where V is volume fraction, W is weight of the matrix and reinforcement, and ρ is the density of each material. We fabricated the stir casting equipment in a chamber system for environment control with the designed mold and stirring system. A schematic sketch of the experimental setup is provided in Figure 1. A pitched blade turbine type of impeller, suitable for suspending particles by strong down-pumping due to generation of axial flow with medium turbulence energy, was used in this research. Even though a propeller type impeller is more advantageous for stirring, a pitched blade turbine type was used due to

its simple structure and easy manufacturing. The size of the impeller was made to be $2/3$ the diameter of the crucible. Baffles were installed to prevent swirling and to convert the horizontal flow of molten metal into vertical flow when rotating the impeller.

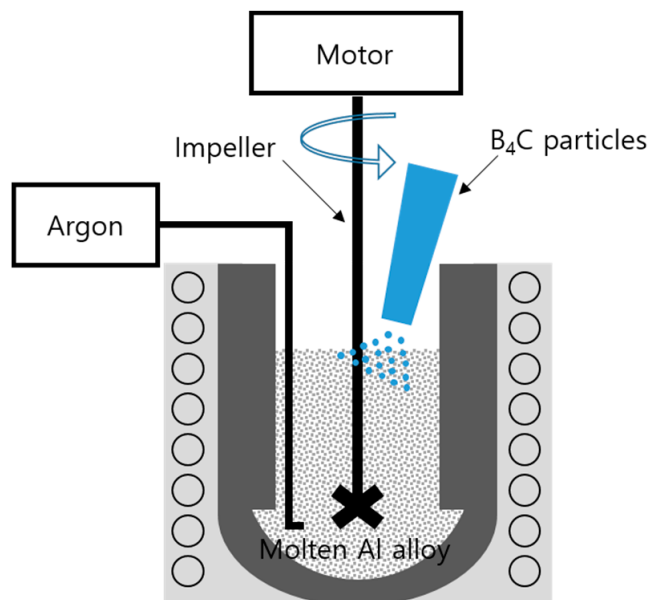


Figure 1. Schematic of stir casting equipment.

Al6061 alloy was melted in a graphite crucible by induction heating under temperatures of about 750 °C for 20–30 min. Degassing of the melt was performed with argon gas. After degassing, B₄C particles preheated to 200 °C were added to the graphite crucible while continuing the stirring process. Stirring was maintained for 10 min at 800 rpm after finishing B₄C addition. After stirring, the melt was poured into the pre-heated steel mold at 200 °C. The fabricated composites were cut into 100 mm × 100 mm × 10 mm for the hot rolling process.

For hot rolling, the composites fabricated by the stir casting process were cross-rolled in atmospheric environments after preheating at 500 °C. A pre-heated composite plate was hot rolled and then heat-treated again for 5 min per one pass. The rolling speed was 10 m per min (MPM) and the final degree of deformation was 84.2%. The fabricated hot-rolled B₄C/Al6061 composites were cut into 250 mm × 350 mm sheets.

The microstructures of the composites were observed with a scanning electron microscope (SEM, LEO-1450, Zeiss, Oberkochen, Germany), a Cs-corrected field-emission transmission electron microscope (FE-TEM, JEM-ARM200F, JEOL, Akishima, Japan), and a field emission electron probe microanalyzer (EPMA, JXA-8530F, JEOL, Akishima, Japan). Sample densities were measured using Archimedes' principle. The tensile tests of the stir-cast B₄C/Al6061 and the hot-rolled B₄C/Al6061 composites were performed at room temperature (RT) using a universal testing machine (5882 model, INSTRON, Norwood, MA, USA) with $5 \times 10^{-3} \text{ s}^{-1}$ head speed. All specimens of the tensile test were prepared in a dog bone shape (sub-size ASTM E8) and the test conditions were the same for all kinds of specimens. The B₄C reinforcement volume fraction was calculated from the SEM images using an in-house code developed by the authors' group [3]. This in-house code automatically extracts reinforcement information from a given composite image, and the volume fraction is calculated as the ratio between the reinforcement area and the total image area. Five images were used for each volume fraction calculation and the average percentage is expressed in this research.

The neutron shielding test is designed to identify the thermal neutron density in a neutron absorber [21]. This test was performed by the Korea Research Institute of Standards and Science (KRISS). The neutron source was Am²⁴¹-Be (α, n). The device produces about

60 neutrons, less than 0.6 eV, per second and irradiates neutrons on the surface of a neutron absorber for 20 min for each sample [21]. In neutron absorber composites, thermal neutron shielding is facilitated by ^{10}B isotope through the $^{10}\text{B}(\text{n}\alpha)^7\text{Li}$ transmutation reaction. Neutron shielding ability is defined as the ^{10}B isotope content present in B_4C within the composite [2]. To estimate the neutron shielding ability according to the B_4C content, the neutron transmittance for the $\text{B}_4\text{C}/\text{Al}$ composite was calculated using the Beer–Lambert law [7].

$$I_t = I_0 e^{(-\Sigma d)} \quad (2)$$

where I_t and I_0 are the intensities of incoming and transmitted neutrons, respectively, Σ is the macroscopic neutron transmission cross-section, and d is the thickness of the composite. In the neutron shielding performance experiment, an $\text{SP}^9\text{--He}^3$ proportional counter was installed in the neutron table weight, and neutron transmittance was determined by measuring the neutron coefficient before and after installation of the composite to be tested.

3. Results and Discussion

3.1. Microstructures of $\text{B}_4\text{C}/\text{Al6061}$ Composites Fabricated by Stir Casting and Hot Rolling Process

Microstructures of the $\text{B}_4\text{C}/\text{Al6061}$ composites are shown in Figure 2. For $\text{B}_4\text{C}/\text{Al6061}$ composites fabricated by stir casting, some B_4C particles were clustered (yellow circles), but most B_4C particles were relatively well dispersed in the Al6061 matrix, as shown in Figure 2a–e. When $\text{B}_4\text{C}/\text{Al6061}$ composites are manufactured by stir casting, the B_4C particles are randomly and three-dimensionally arranged. Because part of the B_4C particles (irregular shape) are exposed in the 2D SEM image, they look small. B_4C aggregation in the $\text{B}_4\text{C}/\text{Al6061}$ composites fabricated by stir casting has two main causes. First, when the turbulent energy dissipation rate, which is related to the size of the impeller and the revolutions per minute (RPM), is insufficient, B_4C particles can be partially clustered in the Al6061 matrix. In addition, even if B_4C is uniformly dispersed in the molten Al during the stir casting process, it can aggregate at the grain boundaries due to grain growth of the Al matrix during solidification. Therefore, it will be possible to improve the dispersibility of B_4C through control of the stirring process and solidification condition. The measured volume fractions of the B_4C reinforcement of the stir-cast $\text{B}_4\text{C}/\text{Al6061}$ composites, obtained using an image tool program, were 4.89, 9.55, 19.25, 24.01, and 26.35% (Figure 2). Microstructures of the $\text{B}_4\text{C}/\text{Al}$ composite plates after hot rolling are shown in Figure 2f–j. After the hot rolling process, the distribution of B_4C particles was highly improved, with less agglomeration and fewer voids. Therefore, it is confirmed that uniformly dispersed $\text{B}_4\text{C}/\text{Al6061}$ composites with negligible defects can be fabricated by a combination of stir casting and hot rolling. Measured volume fractions of B_4C reinforcement were 4.82, 10.90, 20.02, 25.56, and 29.01% for 5, 10, 20, 25, and 30% $\text{B}_4\text{C}/\text{Al6061}$ composites, respectively, values similar to the target volume ratios. The measured volume fractions of B_4C were within 1% compared to the target volume ratios. This result revealed that $\text{B}_4\text{C}/\text{Al6061}$ composites with relatively well controlled B_4C volume fraction were successfully fabricated by self-designed stir casting followed by the hot rolling process.

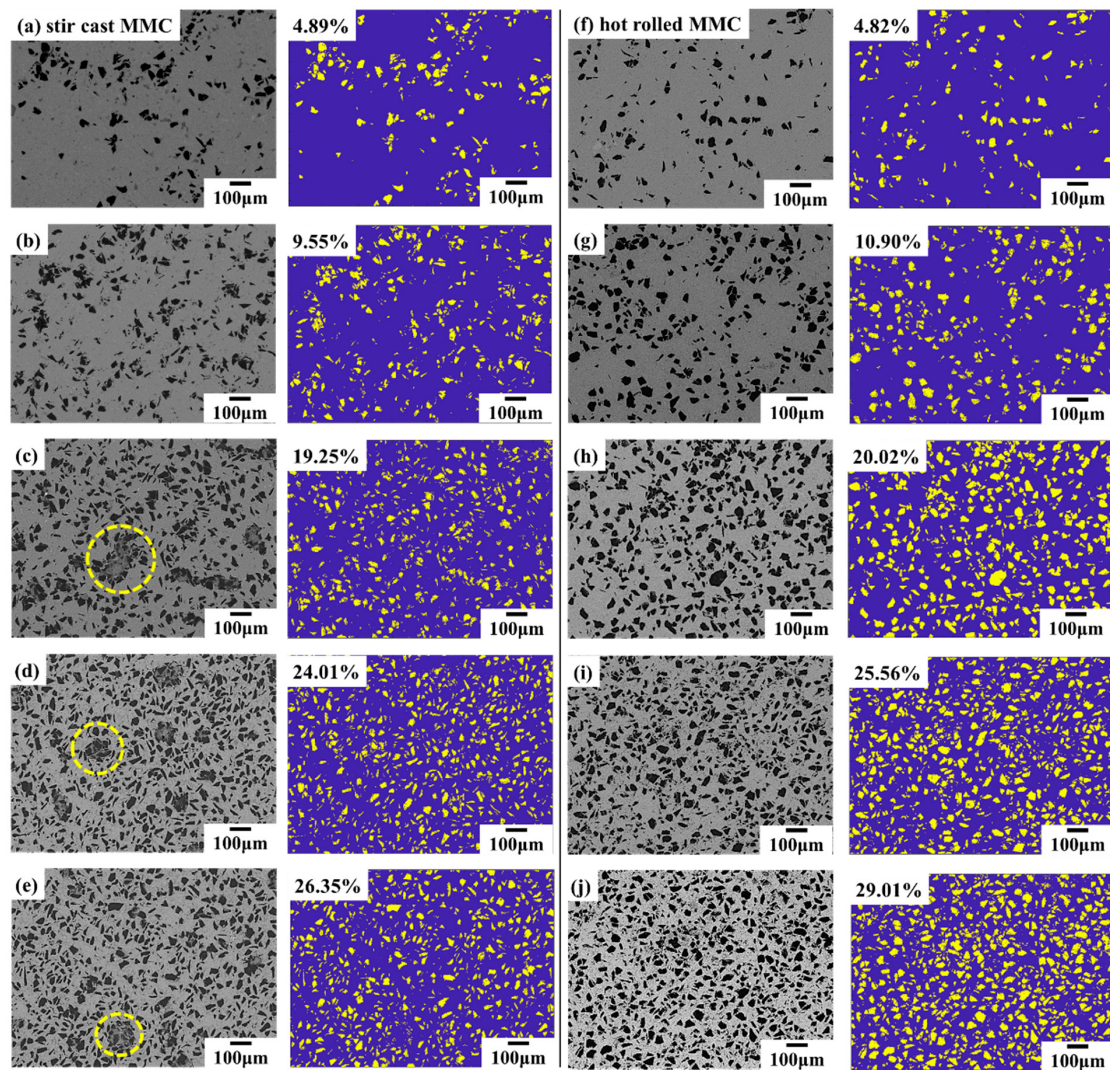


Figure 2. Microstructures of B_4C /Al6061 stir-cast metal matrix composites (MMCs) with (a) 5, (b) 10, (c) 20, (d) 25, and (e) 30 vol.% B_4C and hot-rolled MMCs with (f) 5, (g) 10, (h) 20, (i) 25, and (j) 30 vol.% B_4C .

3.2. Interfacial Analysis of B_4C /Al6061 Composites

EPMA analysis was performed to analyze the elemental distributions of stir-cast B_4C /Al6061 composites and hot-rolled B_4C /Al6061 composites. Figure 3a shows the EPMA analysis results for the 5 vol.% B_4C /Al6061 stir-cast AMCs. Since these were manufactured by the casting process, defects were not observed at the B_4C /Al interface. The EPMA image shows large amounts of elemental boron (B) and carbon (C), indicating that the crystal structures of the B_4C particles are maintained in molten Al during stir casting. Elemental titanium (Ti) and chromium (Cr) appeared continuously along the interface between the B_4C reinforcement and Al matrix. It is known that AlB_2 and Al_3BC precipitates are formed in the B_4C /Al system [26]. However, these did not appear in the B_4C /Al6061 composite manufactured with the stir casting process due to the presence of Ti and Cr in the Al6061 alloy. Therefore, the interfacial layer entirely generated at the B_4C /Al interface can limit the decomposition of B_4C reinforcements and promote their wettability in liquid Al in the stir casting process. Figure 3b shows hot-rolled AMCs, indicating the presence of an interfacial layer after hot rolling. There was no interfacial debonding between the Al matrix and B_4C reinforcement. This result indicates that only the Al matrix undergoes plastic deformation during the hot rolling process, and the B_4C /Al interface maintains a strong bonding by the formation of an interfacial layer.

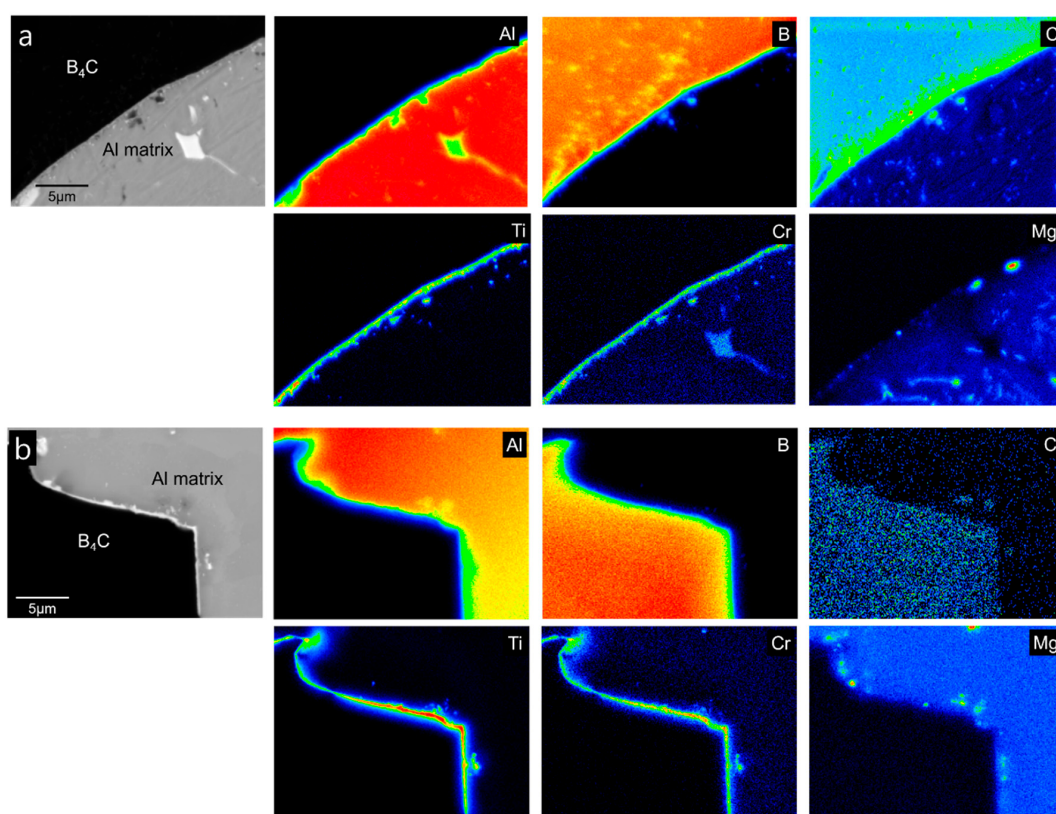


Figure 3. Electron probe microanalyzer (EPMA) analysis of (a) B_4C /Al6061 stir-cast aluminum matrix composites (AMCs) and (b) hot-rolled AMCs at 5 vol.% B_4C .

Figure 4 shows EPMA analysis results for the 30 vol.% stir-cast B_4C /Al6061 and hot-rolled B_4C /Al6061 composites. Ti and Cr mapping images revealed that an interfacial layer was not perfectly generated at the B_4C /Al interface. This is mainly due to insufficient Ti and Cr contents of the Al6061 matrix. Because the interfacial area is considerably increased with increasing B_4C contents, the amount of alloying elements such as Ti and Cr required for interfacial layer generation also increases considerably. Further research is, therefore, required to totally generate an interfacial layer by controlling alloying elements according to B_4C contents.

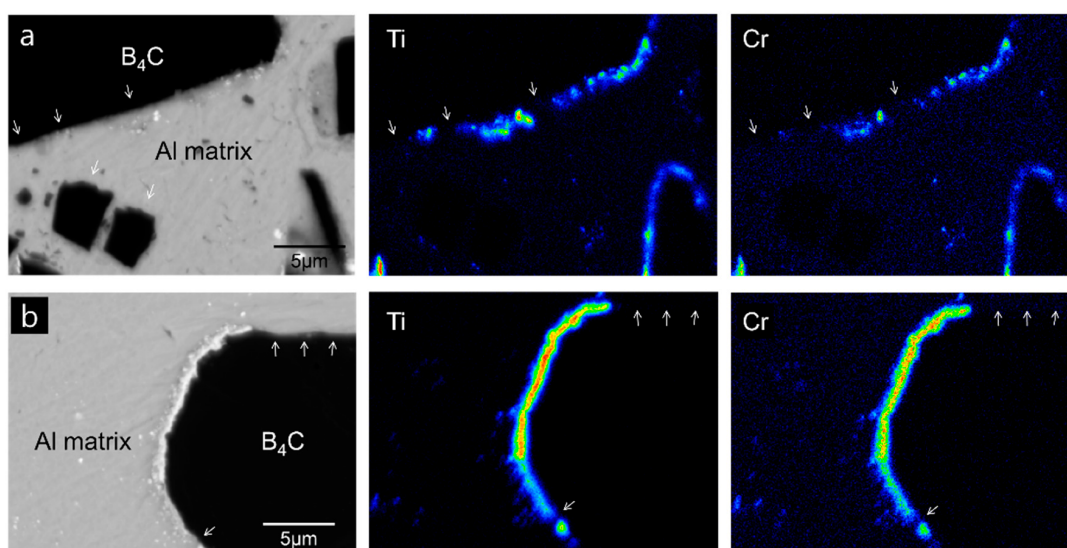


Figure 4. EPMA analysis of (a) B_4C /Al6061 stir-cast AMCs and (b) hot-rolled AMCs at 30 vol.% B_4C .

FE-TEM observations were used to determine the nature of the B_4C /Al6061 composite interface. Figure 5 shows a TEM image and energy dispersive spectroscopy (EDS) element mapping images of the interfacial layer. Element mapping images of Al, B, C, Ti, and Cr provide information on the correlation and distribution of different elements. Ti, Cr, and B formed an approximately 200-nm thick layer surrounding B_4C , and a small precipitate formed in the layer. Furthermore, an Al-based thin layer was observed between the B_4C and Ti-Cr-B layers.

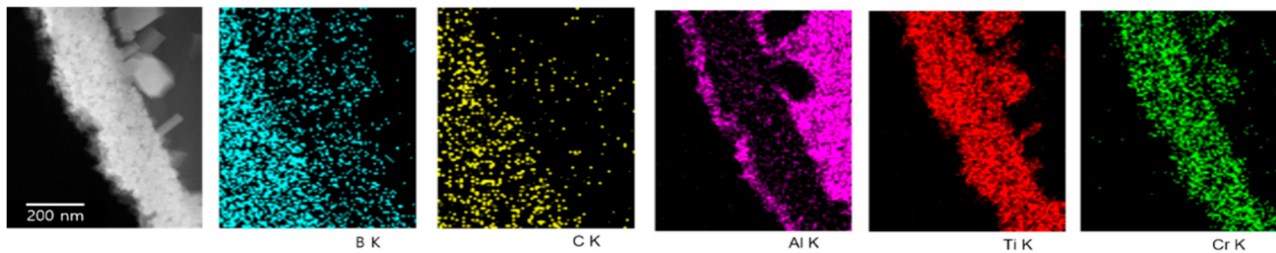


Figure 5. Transmission electron microscope (TEM)—energy dispersive spectroscopy (EDS) mapping images of B_4C /Al6061 composites fabricated by stir casting, indicating the presence of an interfacial layer.

Figure 6 shows a FE-TEM image and selected area electron diffraction (SAED) pattern of the interfacial layer. As a result of pattern analysis, the presences of TiB_2 and Al_4C_3 were confirmed. From the EPMA and TEM results, the outer interfacial layer is known to be mainly composed of TiB_2 , with a small amount of CrB_2 . Therefore, because CrB_2 is isomorphic to TiB_2 , it was expected that a substitutional solid solution would form with the same crystalline structure and similar lattice parameters [27]. It was predicted that molten Al in the casting process will react quickly with B_4C . Indeed, the Al atom rapidly diffused toward B_4C and reacted during the casting process to produce Al_4C_3 . Thereafter, Ti and Cr atoms of the Al alloy diffused to B_4C and reacted with the B atoms in the interface layer to form a thermodynamically stable and continuous $(Ti,Cr)B_2$ layer on the Al_4C_3 layer.

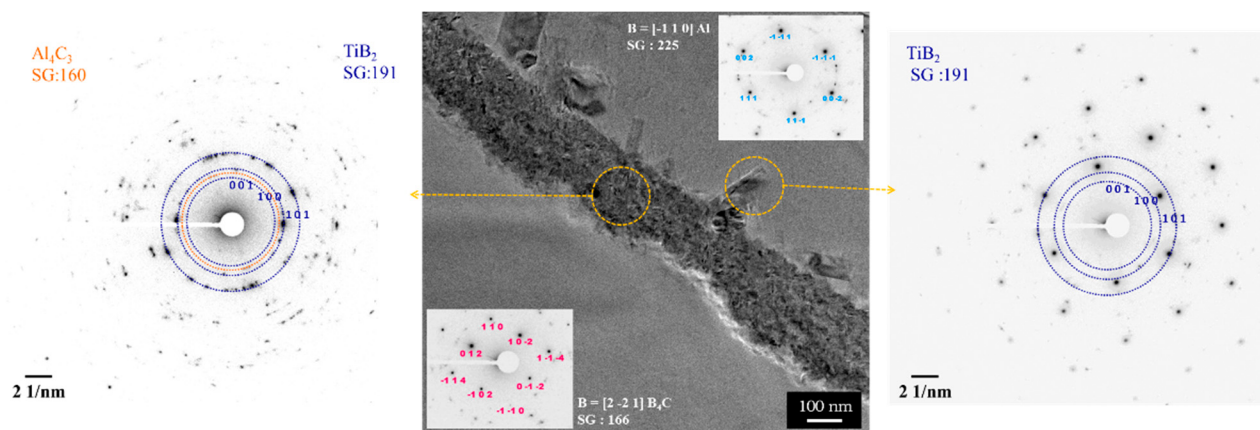


Figure 6. Representative TEM image of interfacial layer in hot-rolled B_4C /Al6061 matrix and selected area electron diffraction (SAED) patterns of the marked areas.

3.3. Mechanical Properties of B_4C /Al6061 Composites

Table 2 summarizes the tensile strengths and relative densities of the Al6061 and B_4C /Al6061 composites. The relative densities of the composites increased after hot rolling. It can be seen that the porosity in the composites significantly decreased after hot rolling. For the hot-rolled MMCs, the relative density was over 99%, indicating that the rolling process reduces the apparent porosity to less than 1%.

Table 2. Summarized tensile strengths and relative densities of Al6061 and B₄C/Al6061 composites.

B ₄ C vol.%	Tensile Strength (MPa)		Relative Density (%)	
	Stir Casting	Hot Rolled	Stir Casting	Hot Rolled
0	173.5 ± 0.7	188.9 ± 0.4	-	-
5	127.8 ± 7.5	249.4 ± 10.3	96.6	99.3
10	123.4 ± 3.7	272.5 ± 4.9	94.1	99.3
20	103.6 ± 2.3	289.2 ± 1.7	95.1	98.9
25	97.1 ± 6.0	241.9 ± 1.0	92.8	99.2
30	102.6 ± 13.9	224.5 ± 2.4	93.8	100.0

When a B₄C/Al6061 composite was manufactured by stir casting without additional heat treatment, the tensile strength decreased as the volume ratio increased due to casting defects such as pores and also due to particle agglomeration. However, B₄C/Al6061 composites after hot rolling showed relatively higher tensile strengths than those of stir-cast B₄C/Al6061 composites and Al6061. The tensile strength of the hot-rolled B₄C/Al6061 composites clearly increased up to 20 vol.% and stayed above the value of Al6061, even increasing to 30 vol.%. This higher tensile strength of the composites than that of Al6061 means that applied load in the Al matrix is effectively transferred to the B₄C in the composites. In the cases of 25 and 30 vol.% composites, the tensile strengths decreased compared to that of the 20 vol.% composite, even though the relative density was more than 99%. The viscosity of molten Al increased highly with the addition of over 20 vol.% B₄C during the stir casting process, so more defects and B₄C agglomeration were generated in the stir-cast 25 and 30 vol.% composites than were generated for the 5, 10, and 20 vol.% composites. In addition, even though the interfacial area increased with increasing B₄C content, Cr and Ti contents of the Al6061 matrix were fixed. Therefore, interfacial layers were not fully generated at the B₄C/Al interface in the 25 and 30 vol.% composites as shown in Figure 4. Therefore, if the stir casting process is optimized by controlling the dispersibility and alloying elements (Ti, Cr) of the composites, the tensile strength can be expected to improve.

The fracture surface of the 5 vol.% B₄C/Al6061 composites after tensile testing using sub-size ASTM E8 specimens (Figure 7a) at room temperature was examined by SEM to study the fracture mechanism during tensile deformation. Figure 7b,c show typical SEM micrographs of the fracture surfaces of the tested B₄C/Al6061 composites. It can be confirmed that fractured B₄C particles are uniformly distributed on the fracture surfaces of the composites. Both ductile fractures of the Al matrix and brittle fractures of the B₄C particles were observed. Although there were not many, dimples as large as the B₄C particle size were also observed in the Al matrix, indicating the possibility of B₄C pull-out. However, it can be clearly observed that most of the B₄C incurred cleavage fracture. For intergranular fractures, the interfacial layer should be observed throughout the fractured B₄C surface. Therefore, the presence of an interfacial layer at the B₄C/Al interface in most fractured B₄C samples is evidence of cleavage fracture. Large cracks on the fractured surface of B₄C particles were occasionally observed. In the case of B₄C/Al6061 composites, therefore, interfacial layer formation at the B₄C/Al interface by reaction of B₄C and Al, Ti, and Cr might cause an effective load transfer from the Al matrix to B₄C, which would increase the tensile strength of the B₄C/Al composites.

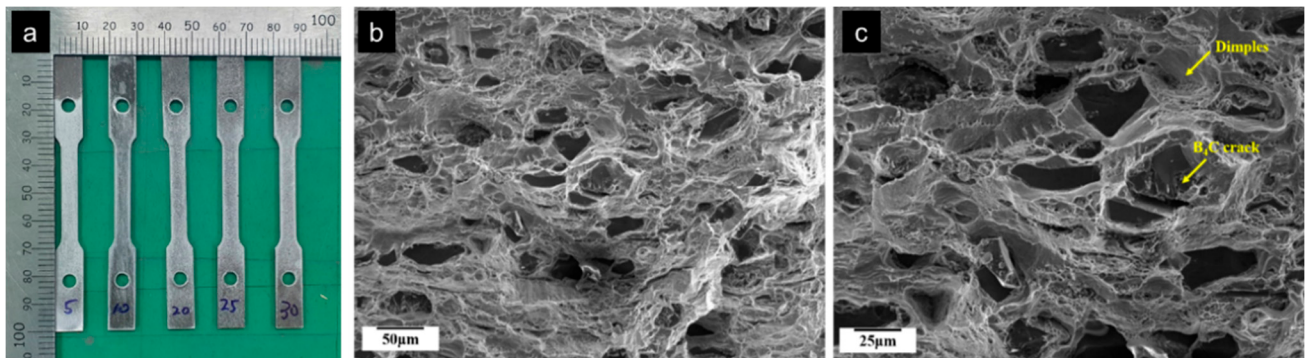


Figure 7. (a) Photograph of tensile specimens (sub-size ASTM E8) and (b,c) SEM images of tensile fracture surface of hot-rolled B₄C/Al6061 composites.

3.4. Neutron Shielding Properties of B₄C/Al6061 Composites

Figure 8 shows the neutron attenuation test facility, consisting of a graphite layer, an SP⁹–He³ detector, and a neutron source. The graphite layer regulates fast neutrons and produces thermal neutrons. The SP⁹–He³ detector ascertains the presence of thermal neutrons. The neutron attenuation test verifies the neutron absorption performance by comparing the measured thermal neutron density to the pre-irradiation thermal neutron density [21].

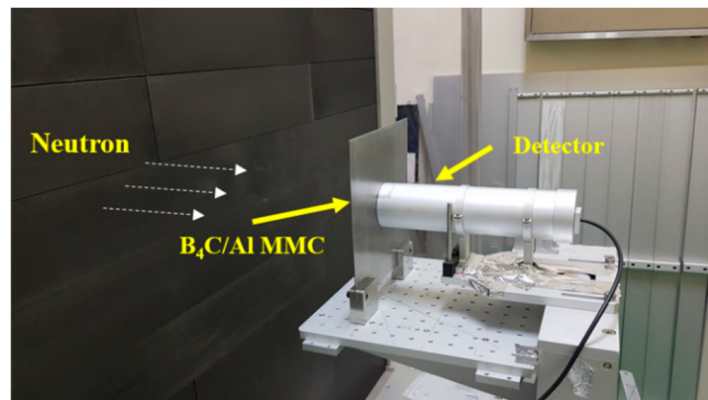


Figure 8. Photograph of neutron attenuation test facility for B₄C/Al composites at Korea Research Institute of Standards and Science (KRISS).

Table 3 presents the measured thermal neutron shielding rate and calculated macroscopic cross-section of B₄C/Al6061 composites considering the thickness of each sample. As the B₄C content increases, the values of the thermal neutron shielding rate and macroscopic neutron transmission cross-section tend to increase. In the case of the 30 vol.% composite, 95.6% of thermal neutrons are shielded at 0.158-cm thickness. The calculated macroscopic cross-section of the 30 vol.% B₄C/Al composite was 19.7 cm^{−1}. Tariq et al. reported that the theoretical calculated neutron shielding ratios of 10, 20, and 30 vol.% B₄C/Al composites are about 65, 90, and 95% at 0.15-cm thickness, respectively [20]. The measured neutron shielding ratios of B₄C/Al composites in this research were similar to reported theoretical values. The results indicate that B₄C/Al composites having superior neutron shielding properties similar to theoretical values are successfully fabricated by stir casting followed by rolling process.

Table 3. Measured thermal neutron shielding rate and calculated macroscopic cross-section of B₄C/Al6061 composites considering sample thickness.

B ₄ C vol. %	Thermal Neutron Shielding Rate (%)	Macroscopic Cross-Section (cm ⁻¹)	Thickness (cm)
5	50.9	4.50	0.158
10	75.4	8.25	0.170
20	90.6	13.91	0.170
25	94.8	18.70	0.158
30	95.6	19.70	0.158

4. Conclusions

In this paper, 5–30 vol.% B₄C/Al6061 composites with relatively well controlled B₄C volume fractions were successfully fabricated by self-designed stir casting followed by the hot rolling process. Microstructures, tensile strengths, and neutron shielding rates of the composites according to the volume fraction of B₄C were investigated. As the B₄C content increases, the values of the thermal neutron shielding rate tend to increase. However, strengthening effect of B₄C was decreased at over 20 vol.% B₄C content. The tensile strength is expected to be improved by controlling the dispersibility and alloying elements (Ti, Cr) of the composites. Process optimization for high volume fraction of B₄C/Al composites is now under way. The following conclusions can be drawn:

- The measured volume fractions of B₄C reinforcement were 4.82, 10.90, 20.02, 25.56, and 29.01% for 5, 10, 20, 25, and 30% B₄C/Al6061 composites, respectively—values similar to the target volume ratios.
- Without AlB₂ and Al₃BC phases, a continuous Al₄C₃/(Ti,Cr)B₂ layer was generated at the B₄C/Al interface in the stir-cast B₄C/Al6061 composites.
- The interfacial layer generated during stir casting maintained its own structure after the hot rolling process, indicating strong interfacial bonding strength.
- The tensile strengths of the B₄C/Al6061 composites clearly increased to 20 vol.% and stayed above the value for Al6061, even reaching 30 vol.% due to partial lack of an interfacial layer at high vol.% B₄C/Al6061 composites.
- The measured neutron shielding values of the composites increased with increasing B₄C content. The 30 vol.% B₄C/Al6061 composite had a 95.6% thermal neutron shielding ratio at 0.158-cm thickness with 19.7 cm⁻¹ macroscopic cross-section.

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Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

AMCs	Aluminum matrix composites
NACs	Neutron absorber composites
MMCs	Metal matrix composites
Volume fraction	%
Weight fraction	%
Tensile strength	MPa
Relative density	%
Thickness	cm
Thermal neutron shielding rate	%
Macroscopic cross-section	cm ^{−1}

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