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Research Article

PRODUCTION OF AA7075/B_4C COMPOSITE MATERIALS BY THE SEMI-SOLID STIRRING METHOD

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AA7075 aluminum alloy was reinforced with B_4C particles to produce aluminum composite materials. In the production of AA7075/B_4C composite materials, the semi-solid mixing method was used for homogenous reinforcement dispersion and low-pressure solidification. For determination of the mechanical properties of the obtained samples, three-point bending, compression and hardness tests were performed. Additionally, the microstructure, reinforcement matrix interface and chemical structure of the produced composite material were investigated by scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDS) and X-ray diffraction (XRD) analyses. As a result, it was observed that, by using the semi-solid mixing method, the reinforcing powders could be distributed homogeneously into the matrix, both chemical and mechanical bonding could be facilitated between the matrix and the reinforcing material, and this mixing technique could be easily used in production of particle-reinforced metal composites. As the reinforcement rate increased, bending strength increased, but there was a reduction in comparison to the non-reinforced aluminum alloy. As the reinforcement rate increased, compressive strength was observed to decreased, while the mean hardness value increased.

Key words*: Aluminum, B_4C, Metal Matrix Composites, Semi-Solid Stirring*

1. Introduction

Studies on metal matrix composite materials (MMCs) began in the late 1950s with the aim of maintaining the superior characteristics of metallic materials and improving their structural properties [1]. These materials may be used in different areas based on their intended use [2]. These materials are advanced materials which show superior properties due to ceramic reinforcements that are dispersed into metals or metal alloys. They have found a broad area of application especially in the aerospace and automotive industries. Ceramics such as SiC, Al_2O_3, C, SiO_2, MgO, TiC, TiB_2 and B_4C are the leading types of reinforcements incorporated into MMCs to improve their mechanical properties. Among these reinforcements, B_4C has been a subject to many fields recently due to its superior physical and chemical properties, and it is a highly strategic material for Turkey, with its rich boron resources [3, 4, 5, 6, 7]. Due to the characteristics of metal matrix composite (MMC) materials

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consisting of a metal matrix and ceramic reinforcements such as high strength, lightness, high abrasion resistance and good thermal expansion coefficients, their usage areas in the automotive, aviation and defense industries are increasingly more frequent [8]. High-tech ceramics, polymers, metals and composites that entered the world's economy with a high market share in the second half of the twentieth century are high-added-value materials [9]. In recent years, particularly aluminum MMCs (Al-MMCs) have been widely investigated and are used in new industrial applications. The widespread use of aluminum as a matrix material is increasing due to its low density and low cost [10, 11, 12]. Moreover, aluminum is an attractive material for production of MMCs due to its corrosion resistance, low electrical resistance and perfect mechanical properties [13, 14]. Production and use of MMCs are increasing with technological advancements, especially in the automotive industry and the aerospace and aviation sectors. By combining the desired properties of two or more identical or different groups of materials one in the form of at least one metal and metal alloy and the other in the form of continuous fiber, capillary crystal or particle, superior MMCs may be produced [15, 16, 17, 18, 19]. MMCs are high-tech materials which can display superior properties such as high tensile strength, high modulus of elasticity, abrasion, compression and creep strength, ability to maintain its stability at high temperatures, ductility and toughness, low specific gravity, low sensitivity to thermal shocks and high electrical and thermal conductivity [20].

2. Research Significance

Due to their low density, high melting temperatures, high elasticity and high strength properties, B_4C ceramic powders are highly preferred in production of high-strength aluminum-based composite materials. In this study, it was firstly aimed to increase the wettability of B_4C ceramic powders by the AA7075 Aluminum alloy by using the semi-solid stirring method and heat treatment of B_4C powders. It was targeted to minimize problems such as non-uniform reinforcement distribution in the matrix, agglomeration of the reinforcements and rejection of reinforcement from mixture and improve the properties of the composite material by the using semi-solid stirring method.

3. Material And Method

3.1. Matrix Material

The AA7075 alloy was selected as the matrix material the production of B_4C-reinforced composite materials. The density of this alloy is 2.8 (g/cm^3), and it has high strength. Copper is the main alloying element in this alloy, where magnesium, chromium and zirconium are additional alloying elements. Tables 1-2 show the properties of the AA7075 aluminum alloy [21].

Table 1. Mechanical characteristics of AA7075 alloy

Heat Treatment	Tensile Strength, Rm MPa	Elongation Strength %	Shear Modulus MPa	Elasticity Modulus GPa
T651	572	11	331	72
T7351	503	13	303	72
T7651	503	13	303	72

Table 2. Chemical composition of AA7075 alloy

Weight %	Weight %	Si	Fe	Cu	Mg	Mn	Cr	Zn	Ti
Minimum	Minimum	-	-	1.2	2.1	-	0.18	5.1	-
Maximum	Maximum	0.4	0.5	2	2.9	0.3	0.28	6.1	0.2

3.2. Reinforcement Materials

Due to its lightness and superior mechanical properties, B_4C is used as the reinforcing material to increase the abrasion and impact resistance of materials [22]. The B_4C material is among the advanced-technology ceramics of today with its characteristics such as high melting temperature, high hardness, high abrasion strength, low density and superior resistance against chemical substances. Its hardness values do not decrease even at temperatures around 1300℃ [23]. Table 3 shows the properties of B_4C reinforcing materials.

Table 3. Some mechanical and physical characteristics of B_4C materials [21].

Reinforcement Material	**B_4C**
Density (x10^3kgm^3)	2.52
Thermal Expansion Coefficient ($10^{-6}C^{-1}$)	6.08
Melting Point (°C)	2420
Compression Strength (MPa)	2900
Elasticity Modulus (GPa)	460
Knoop Hardness	2800

3.3. Composite Material Production by the Method of Semi-Solid Stirring

The temperature of the melting furnace (Figure 1 (a)) was kept constant in the range of 700 ºC-720 ºC. The nitrogen gas introduction setup was installed by means of the mechanism to allow nitrogen gas to be emitted onto the molten metal. After the matrix material was put into a SiC crucible where melting and mixing processes were performed, it was increased to the semi-solid temperature range, and the reinforcement material, heated up to 220 ℃, was added with a speed of 5 gr/min. The B_4C reinforcement was mixed into the matrix at constant temperature by using steel bars with specially profiled stirrers. Homogeneous mixing of the matrix material and reinforcement in the matrix material was ensured without any flocculation by mixing at a low speed by the mixer at a semi-solid temperature. After the reinforcement process was completed, a homogenization process was carried out by applying high-speed mixing to the semi-solid melt for a short time. After the mixing and homogenization processes, the lowest temperature range that provided fluidity was selected as the casting temperature, and the mixture was removed from the melting unit and transferred to the steel molds which had previously been heated to 540-550 ºC (Figure 1 (b)). The mold cap of the melted steel mold was closed, put under a pressure tray and compressed under a pressure process that was started within a few seconds. After applying compressive pressure under 10 MPa for 3-4 seconds, the whole mold was removed from the press, the composite material was allowed to cool in the mold for 15 minutes, and it was removed from the mold and left for cooling at the ambient temperature. The removal process of the prismatic metal matrix composite specimen material from the mold is shown in Figure 1 (c). AA7075/B_4C composite materials were produced by adding B_4C reinforcement into the semi-solid aluminum alloy in the ratios specified in Table 4. Temperature measurements were performed by two K-type thermocouples. One of the thermocouples contacted the furnace's interior, and the other was dipped into the mixture in the crucible. Three specimens were produced for each reinforcement ratio. Control specimens were produced under the same production conditions but without reinforcements.

a) The furnace for semi solid stirring and melting

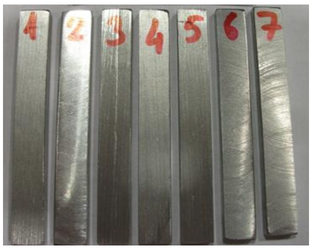
b) Female mold temperature before casting process

c) Sample that came out of mold (110x110x20 mm)

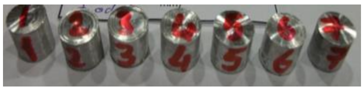
Figure 1. Experiment details from the AA7075/B_4C composite production process

Three-point bending test (TS-205), compression (TS EN ISO 6506-1) and hardness specimens (ASTM-E9) were prepared in accordance with the relevant standards. All specimens were produced with Wire Electrical discharge machining (WEDM) in Hema Industrial Inc. Figure 2 (a), (b), (c) shows the test sample types and preparation details.

a) Preparation of test samples with WEDM process.



b)Three-point bending test samples (4x10x80mm)



c) Hardness, porosity and compression test samples (20x20x50mm)

Figure 2. Test sample types and preparation details

4. Result And Discussion

In this study, due to its widespread use in industrial fields, the AA7075 aluminum alloy was selected as the matrix material in particular. The B_4C material selected as the reinforcing material was mixed into the semi-solid aluminum alloy, and composites with different reinforcing ratios were obtained. The effects of the reinforcing ratios of the obtained composites on mixing performance and mechanical properties were investigated.

4.1. Specific Weight and Porosity Measurement Results

The density of the B_4C-particle-reinforced samples was calculated according to the Archimedes principle. The variation of the theoretical and experimental densities of the samples produced depending on the particle ratio is given in Table 4.

Table 4. Theoretical, specific gravities and porosity ratios of the samples

Material	%B_4C (by Volume)	Theoretical Specific Weight (g_f/cm^3)	Experimental Specific Weight (g_f/cm^3)	% Porosity
AA7075	-	2.80	2.74	2.0
10% B_4C-90% AA7075	10	2.77	2.50	9.8
20% B_4C-80% AA7075	20	2.74	2.43	11.4

It was observed that, as the volumetric reinforcement ratios increased, the experimental specific gravities of the composite materials decreased. Specific weight and porosity experiments were carried out at the Marmara Research Center of TÜBİTAK, and the theoretical and experimental specific weight results of the experiment specimens were compared. As a result of this comparison, the porosity levels were found to be high. This was thought to be due to the length of mixing time and low application rate of compression pressure.

4.2. Mechanical Test Results

4.2.1. Three-Point Bending Test

In this study, three composite materials were produced from each reinforcement ratio to be used in the experiments, and additionally, to make a comparison, three test specimens were produced from the non-reinforced composite material under the same production conditions. Three-point bending tests were applied to these test samples. The bend strength of the samples was calculated with the help of the equation specified in the ASTM B528-05 standard.

When the three-point bending test results of the non-reinforced AA7075 aluminum alloy were examined, the mean bending strength was found to be 268 N/mm^2. When the three-point bending test results applied to the 10% and 20% by volume particle-reinforced AA7075/B_4C composites were examined, it was seen that the mean bending strength of the 10% reinforced composites was 194.6 N/mm^2, and the mean bending strength of the 20% reinforced composites was 206.3 N/mm^2 (Figure 3). As the reinforcement ratio increased, bending strength increased, but due to the high porosity rates, there was a reduction in comparison to the non-reinforced aluminum alloy.

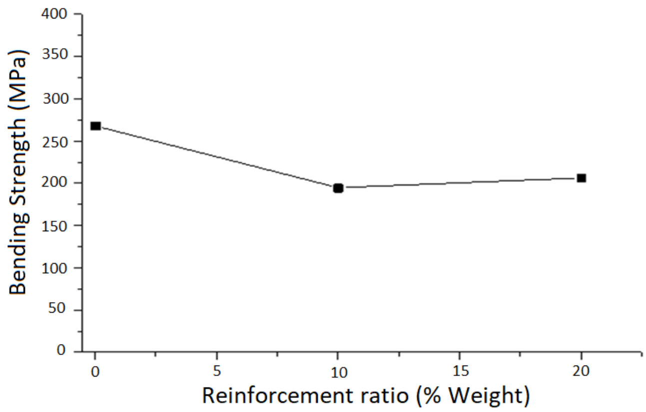


Figure 3. The effect of reinforcement ratio on bending strength in AA7075/B_4C composites

4.2.2. Compression Experiments

When the compression test results of the AA7075 non-reinforced aluminum alloy were examined, it was seen that the mean compressive strength was 500 N/mm^2. When the compression test results applied to the 10% and 20% by volume particle-reinforced AA7075/B_4C composites were examined, it was observed that the mean compression strength of the 10% reinforced composites was 493.7 N/mm^2, and similarly, the mean compression strength of the 20% reinforced AA7075/B_4C composites was found to be 360 N/mm^2 (Figure 4). It was seen that there was a decrease in compression strength after increasing the B_4C reinforcement ratio to 20% by volume.

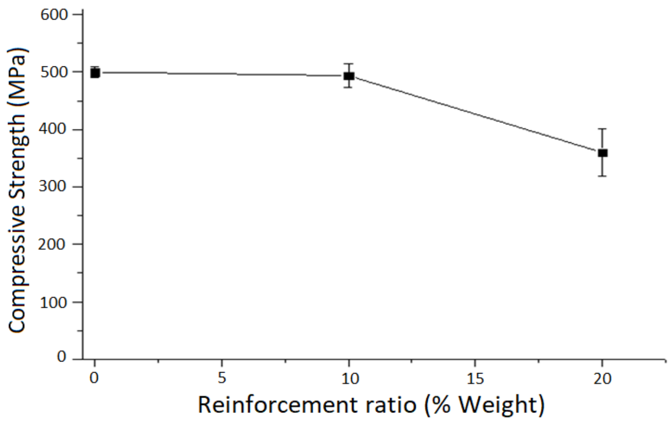


Figure 4. The effect of reinforcement ratio on compression strength in AA7075/B_4C composites

4.2.3. Hardness Experiments

When the hardness test results of the non-reinforced AA7075 aluminum alloy were examined, the mean hardness was found to be 70 BSD. When the hardness test results applied to the 10% and 20% particle-reinforced AA7075/B_4C composites were examined, the mean hardness of the 10% reinforced composites was found to be 107 BSD, while it was 127 BSD for the 20% reinforced composites (Figure 5). In the study, as the reinforcement material had a good wettability by aluminum, it was seen that the mean hardness value increased as the reinforcement ratio increased.

In their study, Hasırcı and Gül (2010) produced averagely 25-μm-sized Al by means of powder metallurgy, they produced 10% and 20% B_4C-reinforced B_4C/Al composites, and they investigated the change in hardness depending on the reinforcement volume ratio. They found the results of approximately 46 HV for the 10% B_4C-Al reinforced composite and about 55 HV for the 20% B_4C-Al reinforced composite [2]. Since the hardness values of composites produced by powder metallurgy are less than 80 HV and 76 BSD, which is the lowest hardness value in hardness conversion tables, no exact comparison could be made with the hardness values found in this study. However, the fact that the hardness values in this study were seen in the hardness conversion tables shows that the hardness values of the composites produced by powder metallurgy were lower than the hardness values in this study.

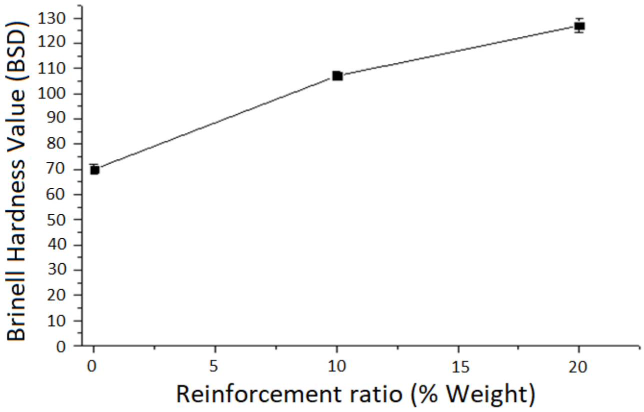
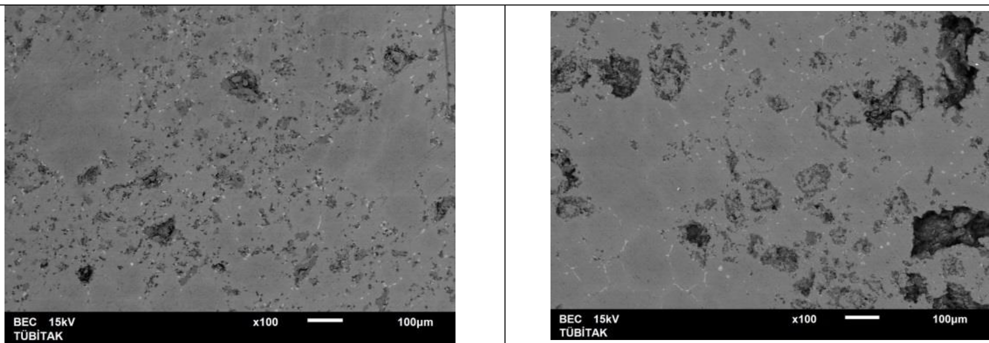


Figure 5. Change in BSD values of AA7075/B_4C composites by reinforcement ratio

4.2.4. Microstructure Examinations

Whether there is a mechanical or chemical bond between the reinforcement phase and the matrix in the composite materials produced may be understood by examining whether the reinforcement phase is homogeneously dispersed in the matrix. The microstructures of the test samples were examined by SEM images taken at different magnification rates.

The SEM microstructure images of the 10% and 20% B_4C-reinforced AA7075/B_4C composites are given in Figure 7. In the composites shown in Figure 7 (a) and (b), it is seen that the reinforcing material was homogeneously distributed, and while looking at x40 and x2500 magnifications, it is seen that the B_4C particles were wetted well by aluminum. When the images are examined, it is observed that the B_4C particles were well-surrounded by aluminum, and there were no images related to pores and flocculation. This shows that the reinforcement was distributed homogeneously in the matrix, and a mechanical bond was formed between the reinforcing material and the matrix material.



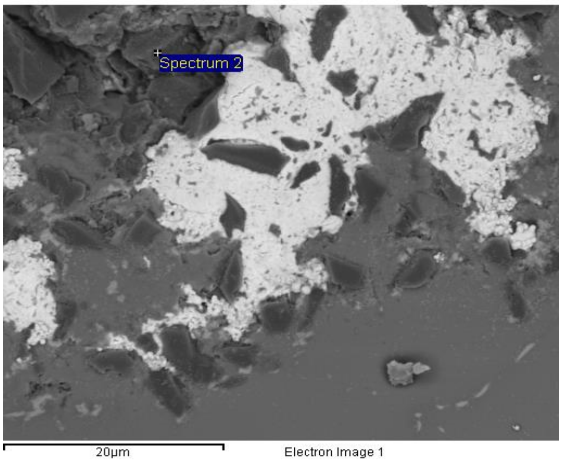
a) AA7075/B_4C composite (vol 10%, x100) | b) AA7075/B_4C composite (vol 20%, x100)

Figure 7. SEM microstructure images of AA7075/B_4C composites

In the EDS analysis given in Figure 8, it was observed that the reaction products formed at the interface of the matrix material with the reinforcement. Thus, the formation of a chemical bond was confirmed. We observed Al peaks that represented the aluminum constituting the matrix material, B and C peaks that constituted the B_4C material and O peaks that confirmed the reaction products.

Spectrum processing:
Peaks possibly omitted: 1.037, 2.141, 2.405, 2.842, 3.020 keV
Processing option: All elements analyzed (Normalized)
Number of iterations = 4
Standard:
B B 20-Jul-1999 12:00 AM
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Al Al2O3 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
B	81.24	84.31
C	13.79	12.88
O	2.61	1.83
Al	2.36	0.98
Totals	100.00	



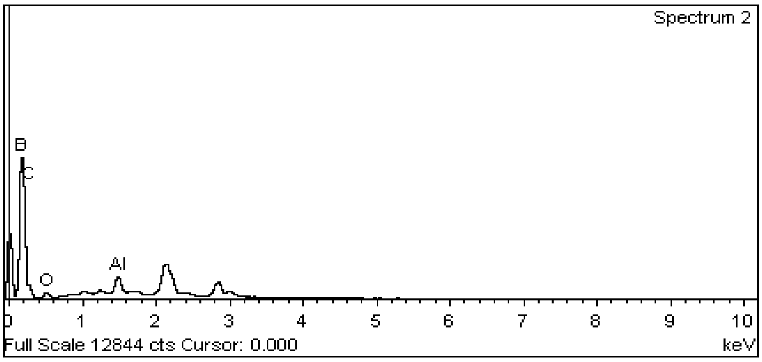


Figure 8. The EDS analyses of the produced 20% reinforced AA7075/B4C composites at the reinforcement region

With the XRD analysis for produced 20% AA7075/B_4C composites with 20% reinforcement, it was determined that Al_3BC and B_2O_3 weighted reaction products were formed at the interface of the reinforcing material and both matrix materials, and there was a chemical bond between the reinforcement and matrix materials as shown in Figure 9. Additionally, it was seen in the XRD analysis given in Figure 10 that the heat treatments performed to increase the wettability of B_4C powders by Aluminum were successful. In this analysis, $B(OH)_3$ reaction products were formed on the B_4C powders.

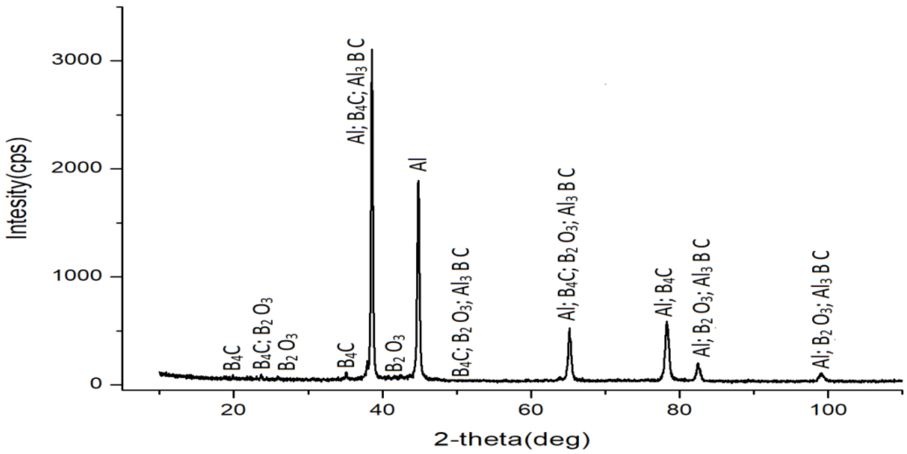


Figure 9. XRD analysis on AA7075/B_4C composite

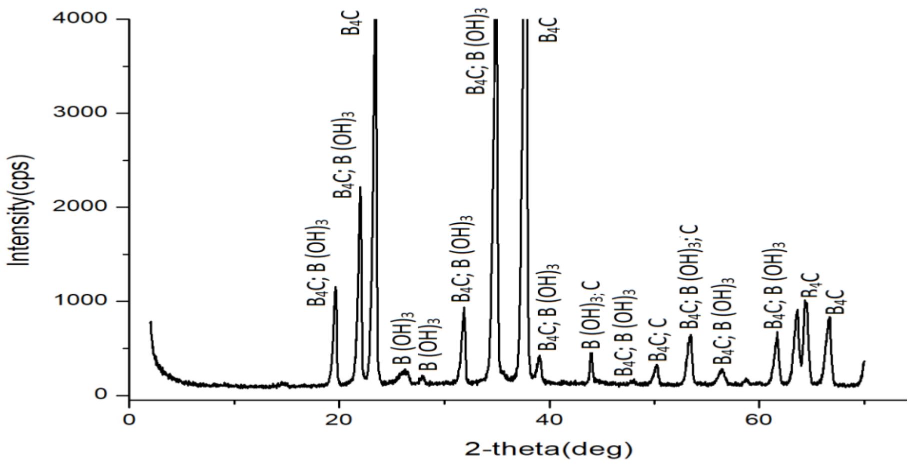


Figure 10. XRD analysis on B_4C powders that were heat-treated

5. Conclusion

In this study, composite materials were produced by reinforcing the AA7075 Aluminum alloy with the B_4C reinforcing material. Composite materials were produced by applying the semi-solid stirring method and a solidification process under pressure in the mold. Mechanical tests were performed to determine the bending and compression strength of the composite materials that were produced, and as a result, microstructure analyses were performed. The results that were obtained are summarized as follows.

a) It was found that the reinforcing material could be mixed homogeneously into the matrix material by using the semi-solid stirring method, and it was seen that no problems such as flocculation, resolution, sedimentation and excluding the reinforcement out of the matrix occurred.
b) Heat treatment of the B_4C reinforcing material resulted in formation of reaction products on B_4C, and thus, the good wettability of the reinforcing material by the aluminum was improved.
c) When the SEM images of microstructures were examined, it was seen that the B_4C reinforcement particles were well-surrounded by the matrix material with the effects of semi-solid stirring and solidification under pressure, and there was a mechanical bonding between the reinforcement and the matrix.
d) It is thought that the interface properties of the composites produced were improved; however, depending on the high porosity ratio, the improvement in the interface properties could not be transferred onto the mechanical properties sufficiently.
e) As in conventional alloys, porosity affects the mechanical properties negatively in the composite materials produced, and this effect is considerably higher than that of conventional alloys.
f) While a liquid or semi-solid stirring process leads to increased gas solubility, the vortex formed during stirring causes the gas to be confined to the liquid alloy. Hence, this shows that the speed of the stirrer and its position in the melt have importance.
g) When the hardness values of the 10% and 20% B_4C-reinforced composites produced by the semi-solid stirring method were investigated, the semi-solid mixing method was found to be successful.

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