



The effect of B₄C amount on wear behaviors of Al-Graphite/B₄C hybrid composites produced by mechanical alloying

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ABSTRACT

In this study, wear behavior of composite materials produced by mechanical alloying method adding different amounts of B₄C in the Al-Gr matrix were investigated. After adding 2% (vol.) graphite to the aluminum matrix, 3 different amounts (3%, 6% and 9%) of B₄C were added. The composite powders prepared were mechanically alloyed for 60 minutes. The milled powders were cold-pressed under 700 MPa pressure. The green compacts produced were sintered at 600 °C for 120 minutes. The sintered B₄C reinforced aluminum composite materials were characterized by the Scanning Electron Microscope, X-ray diffraction, and hardness and density measurements. Wear tests were performed on a standard pin-on-disc wear testing device with a load of 20 N at a sliding speed of 0.5 ms⁻¹ and four different sliding distances (between 250-1000 m) according to ASTM G99 standard. As a result of the studies, the hardness increases as the amount of B₄C in the composite material increases, while the density of AMC decreases. As a result of the wear tests, the highest weight loss was obtained in the non-reinforced Al-Gr matrix alloy, while the lowest weight loss was obtained in 9% B₄C reinforced composite materials. However, it was observed that there was a decrease in the friction coefficient with increasing amount of reinforcement.

1. Introduction

Particle reinforced aluminum matrix composites (AMCs) are considered as a group of materials that can be developed due to their lightness, high strength, low thermal expansion coefficient and good wear resistance [1, 2]. Different reinforcing elements such as SiC [3], Al₂O₃ [4], TiB₂ [5, 6], TiC [7] and B₄C [8, 9] are widely used in the production of these composites. Boron carbide (B₄C), which is among the lightweight ceramic material groups, has high neutron absorbing characteristics as well as high melting temperature, high mechanical properties and good chemical resistance [8, 10]. In contrast, B₄C has significant disadvantages such as low fracture toughness and high sintering temperature have significant disadvantages. Therefore, B₄C particles are preferred as a reinforcing element in the particle reinforced AMCs [2, 11]. In the production of the particle-reinforced AMCs, different production methods are widely used, such as die casting, non-pressurized infiltration, spray forming and powder metallurgy [12, 13]. In the production of AMCs, some problems are encountered, such as reinforcement phase wettability and non-homogeneous distribution during the methods such as casting and non-pressurized infiltration [14, 15]. Powder metallurgy method is widely used in AMC production as it offers some advantages such as homogeneous reinforcement distribution and low process temperatures, as well as not requiring a secondary treatment [16-18].

Mechanical properties of the composites can be improved depending on the amount of reinforcement phase in the matrix in AMCs. In many previous studies, it has been stated that as the amount of reinforcement in the matrix increases, the hardness and therefore the wear resistance of the composite material are increased [5, 6, 15, 19]. In addition, in a study by Pul et al. [14], it is emphasized that the addition of solid lubricants such as MoS₂, BN, Gr and CaF₂ in the particle-reinforced AMCs improves the wear resistance of the composites. Among these solid lubricants, graphite has a very low friction coefficient during sliding, since it contains weakly bonded layers even though it has strong crystalline bonds. Al-Gr alloy composites exhibit lower friction and higher wear resistance, but have high damping capacity and good processability, but Al-Gr alloy composites have reduced mechanical properties due to their graphite content [20]. In the study by Jadhav et al. [21], it has been stated that the density decreases with graphite and B₄C reinforcement in A356/B₄C/Gr composite material and the hardness increases, and there is also an improvement in the tensile strength. In another similar study, it was reported that the hardness, tensile, bending and compressive strengths of Al7075-Al₂O₃-graphite hybrid composites were increased as the weight percentage of the ceramic reinforcing element increased, while the hybrid composites containing graphite exhibited excellent wear resistance [22]. Therefore, in this study, different amounts of B₄C were added to Al-Gr alloy matrix

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composite materials were produced by mechanical alloying method which is a powder metallurgy method. It is aimed to determine the effect of addition of B_4C in different amounts in the structure of composite materials on the microstructure and wear behaviors under a constant load.

2. Materials and methods

In the experimental studies, aluminum (Al) powder (vol.%) having a size of $<50 \mu m$ and a purity of 99.5% and graphite of 2% having a size of $<40 \mu m$ were used as the matrix material. The Al matrix was reinforced with B_4C (vol.%) having a powder size of $<30 \mu m$ in three different amounts (3%, 6% and 9%). The chemical composition of the AMCs powders produced is provided in Table 1.

Table 1. The chemical composition of the AMCs powders.

	Al	Graphite (%)	B_4C (%)
Al-Gr	98	2	---
3 B_4C	95	2	3
6 B_4C	92	2	6
9 B_4C	89	2	9

The powders having the chemical composition given in Table 1 are mechanically alloyed in a planetary mill. During Mechanical alloying/Mechanical milling (MA/MM) processes, a stainless steel milling cell (400 ml), stainless steel balls having a diameter of 10 mm, a ball to powder ratio of 1:10, 1% of ethanol is used to prevent agglomeration, and a milling time of 60 minutes were used in the atmospheric environment. Mechanically alloyed Al composite powders were cold pressed (700 MPa) and green compacts of $\varnothing 10 \times 7$ mm were produced. The produced green compacts were sintered at 600 °C under argon for 120 min. and cooled to room temperature in a furnace. A Protherm HLF 50 heat treatment furnace was used for the sintering process. Standard metallographic treatments (sand-ing and polishing) were applied for the microstructure analyses and the samples prepared were etched with a solution of 2 ml HF, 3 ml HCl, 20 ml HNO_3 , 175 ml H_2O (Keller's) solution for 10-15 seconds. Microstructure analyzes of the etched samples were carried out by Carl Zeiss Ultra Plus Gemini Scanning Electron Microscope and Electron Diffraction Spectroscopy (SEM+EDS). In addition, the sintered samples were characterized by X-ray diffraction (XRD), and hardness and density measurements. For XRD analysis were recorded on a Rigaku Ultima IV X-ray diffraction spectrometer using $Cu \text{ } k\alpha$ ($k: 1.54A$) and a scanning rate of $3^\circ/\text{min}$. Diffraction patterns were obtained with 2 theta (Bragg angle) between 10° and 90° . Density measurements were made according to Archimedes' principle. Density measurements were taken on three samples and averaged. Hardness measurements were taken with using Shimadzu microhardness tester for 10 seconds by using HV2. The hardness measurements were obtained by using three different samples, at five different points and used averaged. Wear tests

were carried out at four different (between 250-1000 m) sliding distances at a sliding speed of 0.5 ms^{-1} under a load of 20 N on a pin on disc wear test device according to ASTM G99 standard. Before each wear test, the surface of samples and disks were cleaned with acetone. In the wear tests, three different samples were tested for each parameter and the results were averaged. Wear rates were calculated by the equation given in Equation 1 using the weight loss results obtained.

$$W_a = \Delta m / (M \cdot s \cdot \rho) \quad (1)$$

Wherein, W_a is the wear rate, Δm is the weight loss (g) obtained after the wear test, M is the load (N) used in the tests, s is the sliding distance (m), and ρ is the density (g/cm^3) of the wear sample. After the wear test, SEM images of the worn surface were examined and the active wear mechanisms were tried to be determined.

3. Results and discussion

The microstructure of the SEM images and large area EDS results of the different amounts of B_4C reinforced AMCs are given in Figure 1.

When the microstructure SEM images of AMCs produced by the addition of different amounts of B_4C in the Al-Gr matrix were given in Figure 1. In Figure 1.a, seen that there were undissolved graphite particles in the Al matrix. This situation was described with formation of the large Gr particles in the Al matrix with mechanical alloying, and so the graphite particles were not completely dissolved in the structure. In a previous study, it was stated that in the Al-C system, the smaller graphite particles were completely dissolved in the structure and the larger graphite particles remained as graphite particles in the structure [23]. Sinan et al. [24] described the graphite powders could be distributed homogeneously in the Al matrix with the long mechanical alloying time at room temperature. It is clear that B_4C added to the Al-Gr matrix is homogeneously distributed. It is seen that the B_4C added as reinforcement is located at the grain boundaries. However, it is understood on the SEM images that there is an oxidation in the structure (white points). In the large area EDS results given in Figure 1.e, this is supported by the presence of some O with matrix Al, B and C. In addition, as the amount of reinforcement in the structure increases, micro-voids occur especially at the grain boundaries. The XRD analysis result of the composite material in which 9% of B_4C are added to the Al-Gr matrix is given in Figure 2.

In Figure 2, XRD analysis results were given. In Figure 2 seen that Al_4C_3 compound which is expected to occur in the structure together with matrix Al, C (graphite) and B_4C is formed. In the study of Bostan et al. [25], it is stated that Al_4C_3 compound is formed in the nanoscale in the alloy by sintering in Al-C system produced by the mechanical alloying method. It is also

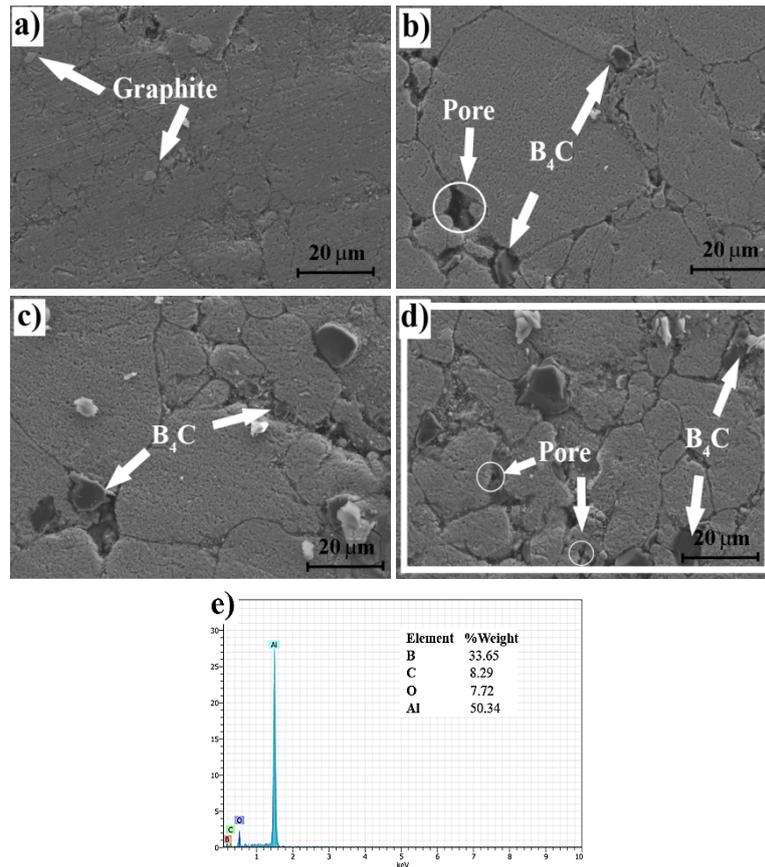


Figure 1. The microstructure of SEM images and EDS results of AMCs, produced with the addition of different B_4C amounts in the Al-Gr matrix, Al-Gr + (a) 3% B_4C (b), 6% B_4C (c), 9% B_4C (d) and wide area EDS results of 9% B_4C (e).

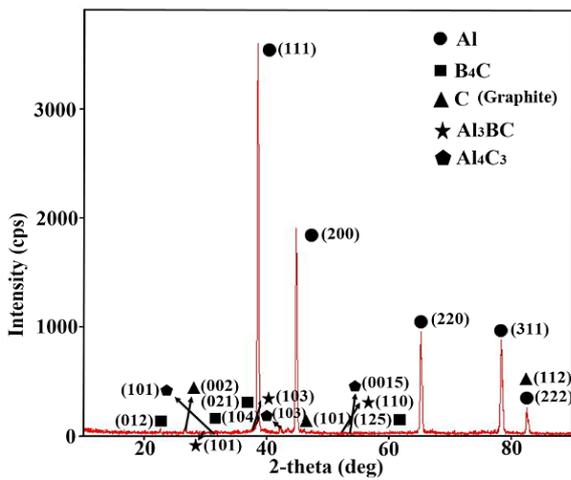


Figure 2. XRD analysis result of the Al-Gr + 9% B_4C composite material.

understood that Al_3BC is formed in the structure. In the study of Mohanty et al. [26], it was stated that Al-B-C compounds were formed along the grain boundaries in the structure with B_4C added at a ratio of 1% and 2%, and as the amount of B_4C increased (3%-25%), Al-B, boron carbide and Al-boron carbide were formed instead of Al-B-C triple system. However, in the same study, it was stated that the microstructure became more complex with increasing amount of B_4C in the structure and the phase accumulation increased along the grain boundaries. The changes in the hardness

and density of AMC materials produced by adding different amounts of B_4C to the Al-Gr matrix are given in Figure 3.

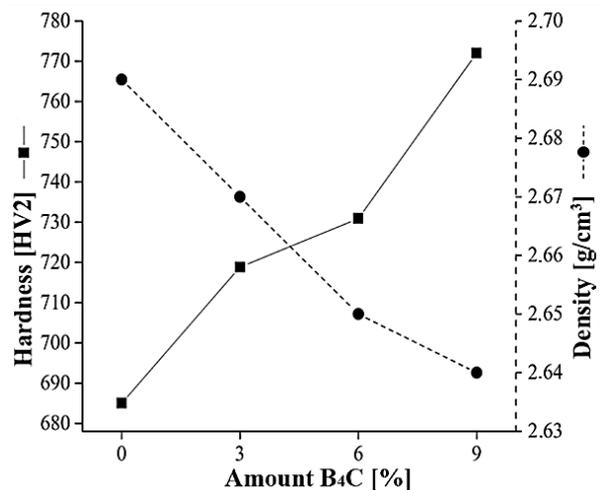


Figure 3. Hardness and density results of the Al-Gr + B_4C AMC materials, produced by the addition of different amount of the B_4C .

When the hardness changes of the AMCs produced by adding different amounts of B_4C given in Figure 3 are examined, it is seen that this parameter increases with increasing amount of reinforcement as compared to the reference material (Al-Gr). The lowest hardness value was obtained as 687 HV in the matrix material

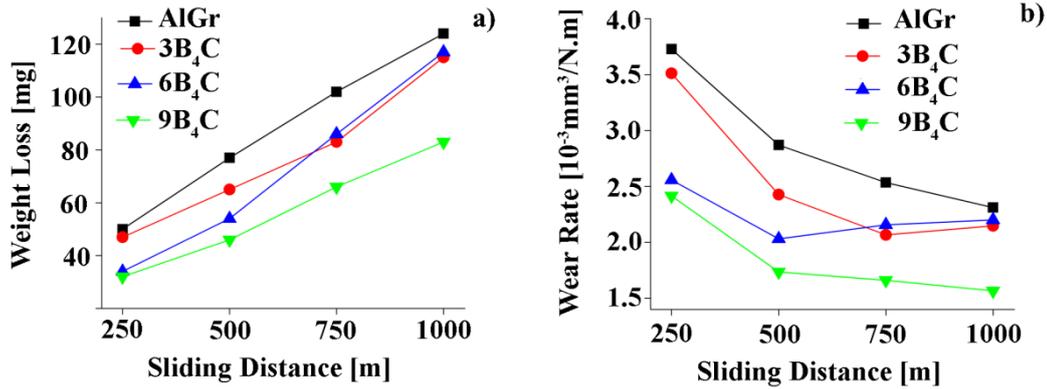


Figure 4. Weight loss (a) and wear rate results (b) of the composite materials produced by the addition of different amounts of B₄C in the Al-Gr matrix, obtained by using constant load.

(Al-Gr) without B₄C, whereas the highest hardness was obtained as 788 HV in the composite material with 9% of B₄C. This can be explained by the mixture law given in Equation 2 [27].

$$H_c = H_m \cdot F_m + H_t \cdot F_t \quad (2)$$

H_c, H_m and H_t given in the equation are the hardness of the composite, matrix and reinforcement material respectively, F_m and F_t are the fraction of the matrix and reinforcement material respectively. According to Equation 1, the hardness increases due to the increased hard reinforcement ratio in the matrix. Similar results were obtained in the previous studies [11, 18, 28, 29]. The hardness increases could be described with the increasing of the reinforcement ratios in the AMC system [30]. Also, the density changes were given in Figure 3. The decreases of the density ratios in Figure 3 could be described with the increasing of B₄C reinforcement ratios in the AMC parts [31]. The highest density value was obtained in the matrix material (Al-Gr) as 2.68 g/cm³, whereas the lowest density value was obtained in the material with 9% of B₄C as 2.64 g/cm³. This decrease in AMC densities could be described with the B₄C particles having lower density ratios than main matrix. Also the main matrix wettability capacity in the AMCs - B₄C composite system [29, 31]. In addition, micro voids in the structure occurred as an inevitable consequence of the powder metallurgy method are another factor affecting the density. The increased amount of reinforcement and the micro voids in the structure are another reason for decreasing the density of B₄C reinforced composites [8, 11]. The weight loss and wear rate results of AMC materials produced by addition of the different amounts of B₄C in the Al-Gr matrix. The weight loss and wear rate results were given in Figure 4.

When the weight loss results of composite materials in which different amounts of B₄C were added given in Figure 4.a. Also, it could be seen that the weight loss increases with increasing sliding distance as expected. The highest weight loss was obtained in the Al-Gr matrix alloy, whereas the lowest weight loss was obtained in the composite material with 9% of B₄C. This

situation was described with the increasing of reinforcement ratios in the matrix, increased the hardness values. Also, the hardness values were main determinant for determining the wear resistance of the AMC systems [32]. In the study of İpek [33], it was stated that the wear resistance of the Al/B₄C composite material increased significantly with the increasing of the B₄C reinforcement ratios in the Al matrix alloy. Also, in Figure 4, it was seen that there was unexpected increase in weight loss at the 500 m sliding distance for 6% B₄C AMC composite structure. The unexpected increase in the weight loss can be explained with falling of the large B₄C particles' during the friction. Also, when the wear rate results were examined in Figure 4, it could be seen that they were compatible with the results of weight loss. In addition, the wear rate was decreased as the sliding distance and the amount of reinforcement in the matrix are increased [34]. However, the presence of graphite in the composition of composite materials was another reason for the decreasing of the wear rate. In the graphite-containing Al hybrid composites, the graphite between the pin/disc reduces the wear by facilitating the sliding over the contact surface [35, 36]. In Figure 5 seen the friction coefficients of the AMC materials produced by the addition of different B₄C amounts in the Al-Gr matrix under a constant load.

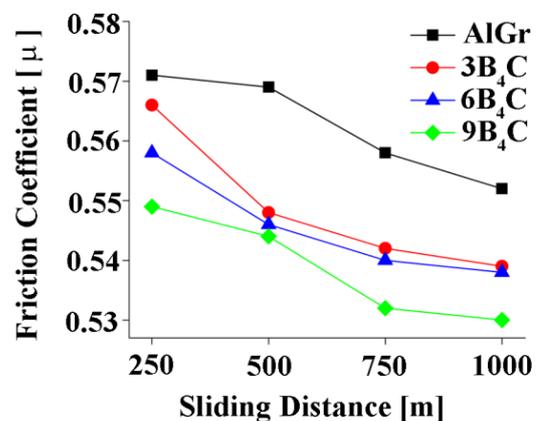


Figure 5. Friction coefficients of the composite materials produced by the addition of different amounts of B₄C, obtained by using constant load.

In Figure 5, the friction coefficients of the composite materials with different amounts of B_4C were given. In Figure 5 could be seen that the friction coefficient decreases with the amount of reinforcement and sliding distance. The oxide layer formed by the effect of heat caused by friction between the pin-disc is effective in reducing the friction coefficient. Similar results were obtained in a study by Özyürek et al. [5]. In addition, the

graphite in the composition of the composite materials production was another important factor in the reduction of friction coefficient. In the study of Baradeswaran and Perumal [22], they were stated that the graphite in the structure reduces the friction coefficient by about 50%. In Figure 6 given the SEM images of the worn surface images of the AMC materials produced by the addition of the different B_4C amounts in the Al-Gr matrix.

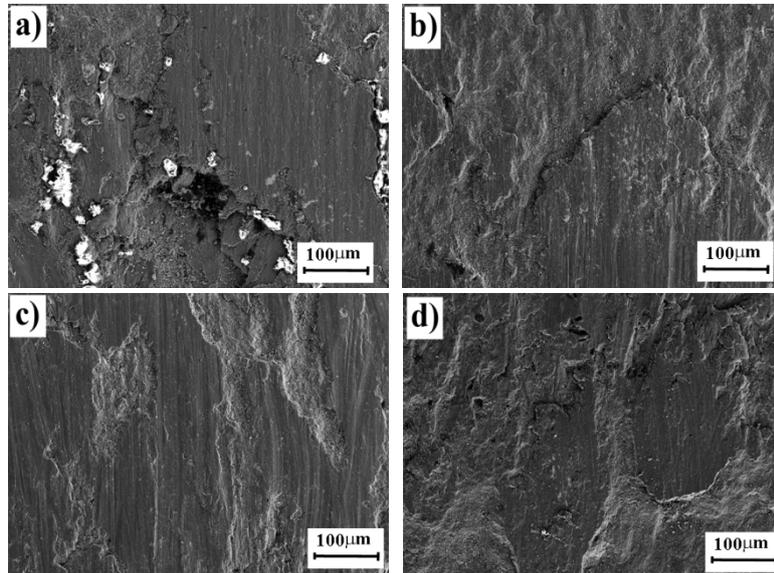


Figure 6. SEM images of the worn surface of the AMC materials produced by the addition of different amounts of B_4C , Al-Gr (a), Al-Gr + 3% B_4C (b), 6% B_4C (c), 9% B_4C (d).

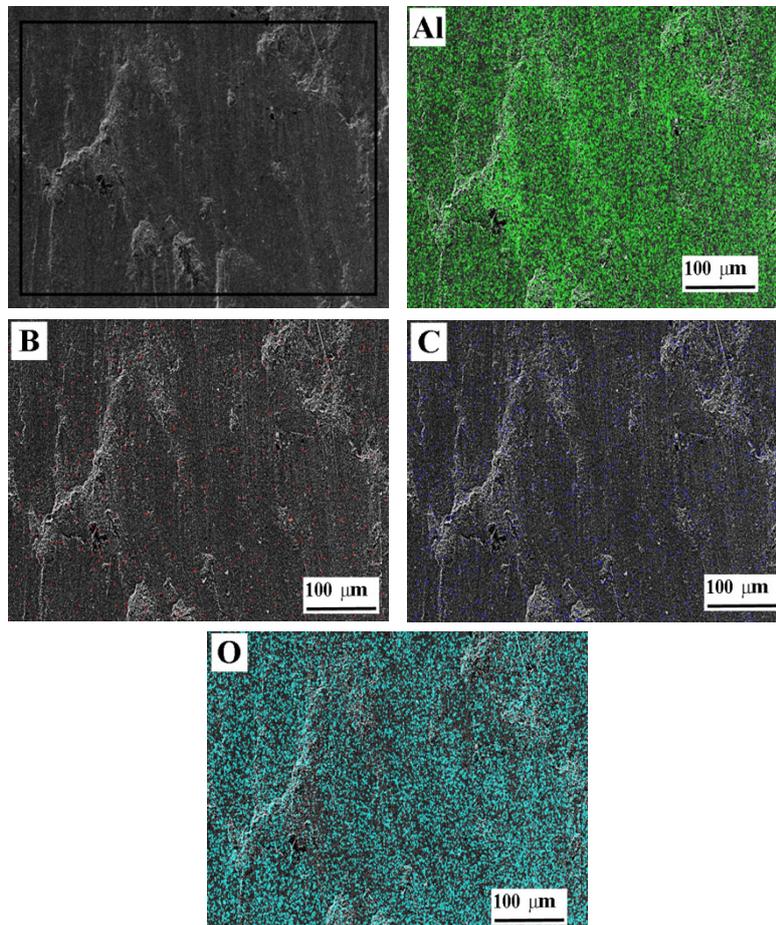


Figure 7. SEM and MAPPING images of the worn surface of the Al-Gr + 9% of B_4C composite material.

In Figure 6, the SEM images of the worn surface of the composite materials were given and in the images determined that with the increasing of the B_4C ratios the damages on the surfaces decreased. Especially in the Al-Gr alloy used as a matrix (Figure 6.a), the oxides (white areas) formed with stripping were seen more clearly. It was understood that the stripping on the sample surfaces decreases depending on increasing amount of B_4C . It was clearly seen that the stripping was having minimal on the worn surface of the composite material containing 9% of B_4C as compared to the other composites. In addition, the friction coefficient results given in Figure 6 supported this situation. Increased surface roughness due to spilling on the surface causes friction coefficient to increase. MAPPING results of the worn surface of AMC materials produced by adding different amounts of B_4C to the Al-Gr matrix were given in Figure 7.

In the MAPPING images of the AMCs reinforced with different amounts of B_4C (Figure 7) were clear determined that an oxide layer was formed on the surface (due to pin-ring friction) as a result of the wear tests. The heat released between the disc and the sample contact surfaces during wear tests causes the formation of oxide layers on the surface of the Al matrix. This oxide layer on the surface was an important factor affecting the tribological parameters such as weight loss, friction coefficient and wear rate. Because, this oxide layer on the surface was protected the surface with its solid lubricant effect [22]. The graphite (C), which exhibits a homogeneous distribution in the matrix as well as the oxide layer formed on the worn surface, forms a thin film in the metal-metal contact region during friction and this film prevents the oxide particles from breaking [36]. Therefore, both the graphite in the structure and the oxide layer formed during the wear test serve as a solid lubricant together, which makes a significant contribution to the wear resistance of the Al composite. In previous similar studies, it was stated that the graphite addition in the main matrix has formed a film layer that acts as a solid lubricant on the contact surface. Also, this film was increased the wear resistance of composite materials. [37, 38].

4. Conclusions

The results of the wear behavior of the composite materials were given as below results, obtained after the addition of different B_4C amounts in the Al-Gr matrix under a constant load.

- It was determined that B_4C reinforcement was distributed homogenously in the microstructure of the Al/Gr/ B_4C composites produced using powder metallurgy method, as expected and that they usually located at the grain boundaries. Microvoids were formed as the amount of reinforcement increases.
- It was determined in the Al-Graphite-9% B_4C structures XRD analysis results that, the Al_4C_3 and Al_3BC

intensities formation were determined in the produced parts which were formed in the main phase structure.

- The hardness of Al-Graphite- B_4C AMCs was increased with increasing amount of B_4C in the matrix.
- The density of Al-Graphite- B_4C AMCs was decreased with increasing amount of B_4C in the matrix.
- As a result of the wear tests, the highest weight loss was obtained in the non-reinforced Al-Graphite sample. It was determined that weight loss decreased with increasing B_4C in the matrix.
- As a result of the wear tests, the highest friction coefficient was obtained with the non-reinforced Al-Graphite sample. It could be described with the friction coefficient of the composites, which was decreased depending on the increasing of the amount of B_4C addition in the matrix (Al-Gr).

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