In this study, it was aimed to produce Al-matrix composite materials with B₄C-SiC and B₄C-Y₂O₃ particle reinforcement using the Powder Metallurgy (PM) method. In the composites in which the reinforcement materials were used at different particle sizes and ratios, AA2024 powders were selected as the matrix material. The powders were homogeneously mixed and compacted at room temperature under a pressure of 525 MPa. The raw specimens were sintered by keeping them at different temperatures for 45 minutes. The composite materials were subjected to wear tests, and their hardness and density values were investigated. In the study, the effects of reinforcement materials added to the matrix at different particle sizes and ratios on wear resistance, hardness, and density at different sintering temperatures were examined. As a result of the mechanical tests that were performed, wear values decreased along with B₄C reinforcement ratios that increased in parallel with density values. The highest hardness value was observed as 71.73 HB in the composite specimen with a B₄C reinforcement ratio of 20% at the sintering temperature of 595°C.

Keywords: Composite Material, Particle Size and Ratio, Powder Metallurgy, Sintering, Wear.

1. Introduction

Advanced materials have become prevalent worldwide as a consequence of advancements in science and technology, to meet the need for specialized products. The reason for this is that a single material usually cannot meet the requirements of all engineering fields. Therefore, researchers have shown great effort to use composite materials with unique properties. As a result of this, there has been an increase in the production and application fields of metal matrix composites in recent years [1]. In previous years, metal-matrix composites (MMCs) received worldwide attention with their excellent properties combining the best properties of metal matrices and reinforcements [2]. The physical and mechanical properties of MMCs can be improved by adding reinforcement material particles into the matrix. As the matrix material, light metals such as Ti, Al, and Mg that provide support for the reinforcement material are used in general. Among MMCs, the production of aluminum-matrix composites (AMCs) is becoming more prominent each day and finding various usage areas in the industry [3]. Aluminum is an excellent matrix material for developing wear-resistant AMCs [2]. Aluminum alloys are the most convenient alloys following iron alloys [4]. It is well-known that the performance of AMCs is highly dependent on the reinforcements that are added [2]. AMCs use an aluminum alloy as the matrix material and mostly include ceramics like SiC [5-8], B₄C [6,7,9], Si₃N₄ [7], Al₂O₃ [5,6,8], BN [7], Zr [8], and TiC [7] as the reinforcement material. The properties of AMCs can be changed based on their elemental and volumetric ratios [3]. AMCs reinforced with hard ceramic particles are the most promising materials for various automotive and aviation applications with their low density and high wear resistance [5,7], and they are
used in many fields in the industry, especially in critical applications such as brake calipers, gears, engine blocks, pump components, and valve components [10,11]. Several Al alloys (e.g., 2xxx, 5xxx, 6xxx, 7xxx) are industrially utilized [12]. As a reinforcement material for AMCs, \( B_4C \) has attracted special attention [13]. Boron carbide (\( B_4C \)) and silicon carbide (\( SiC \)) particles, which are the most preferred reinforcement materials for AMCs, strengthen the matrix structure and provide good wear resistance and high thermal stability [12]. \( B_4C \), which is called “black diamond”, has a low density (2.52 g/cm\(^3\)), high hardness (29.1 GPa), high modulus of elasticity (470 GPa), and high melting point (2540°C) [14]. Because \( SiC \) has high hardness values, high resistance to rupture, a high modulus of elasticity, low density, and a low thermal expansion coefficient, its use as a reinforcement material is common [12]. \( Y_2O_3 \) ceramics have critical properties such as high thermal and crystallographic stability, and they are used in various applications including protective coatings, microwave radars, solid-state lasers, superconductors, and catalytic converters [27]. In the literature, there are different methods of production including squeeze casting, stir casting, liquid metal infiltration, mechanical alloying, and powder metallurgy for MMCs [15]. The powder metallurgy (PM) method is a production method where mixed metal powders are shaped by compaction into a mold that has been prepared based on the shape and dimensions of the part to be produced and then sintered [6]. PM allows manufacturing processes that result in parts close to those with precisely shaped parts produced out of Al-based materials and MMCs with certain mechanical, thermal, and functional properties [16]. In this study, by ceramic particle reinforcement, Al-\( B_4C \), Al-\( B_4C\)-\( SiC \), and Al-\( B_4C\)-\( Y_2O_3 \) composites with an aluminum alloy matrix were produced, and the effects of the reinforcement materials on the mechanical and microstructure properties of the composites were examined.

2. Materials and Methods

In this study, where reinforcement materials were used at different ratios and particle sizes, AA2024 aluminum alloy powder was used as the matrix component, and \( B_4C \), \( SiC \), and \( Y_2O_3 \) powders were used as the reinforcement components at different ratios and particle sizes. Aluminum alloy at a particle size of 100 \( \mu m \) was used as the matrix, while the reinforcement materials included boron carbide (\( B_4C \)) at ratios of 7, 10, 15, and 20% and particle sizes of 10 and 22-59 \( \mu m \), 20% \( B_4C \) + silicon carbide (\( SiC \)) at ratios of 3-10% and a particle size of 10 \( \mu m \), and 20% \( B_4C \) + yttrium oxide (\( Y_2O_3 \)) at ratios of 3-10% and a particle size of 10 \( \mu m \). The chemical composition of the AA2024 alloy that was used as the matrix material is shown in Table 1.

2.1 Specimen Preparation

The powders were weighed using a RADWAG PS 1000/C/2 brand precision scale with a sensitivity of 0.001 g and homogeneously mixed at different weight ratios. Composite materials were produced by applying a pressure of 525 MPa onto a mold prepared with the dimensions of 10x10x55 mm using a 100-ton uniaxial hydraulic press branded Hidrokar. Polyethylene glycol (1% wt.) was added to the powder mixtures as a binder. The specimens were sintered at 550, 575, and 595°C for 45 minutes using a Protherm brand furnace.

2.2 Hardness Test

The hardness measurements were made with specimens that were brought to the dimensions of 10x10x10 mm using an EMCO-TEST DuraVision Brinell hardness tester device (Load: 15.625 kg, Ball Diameter: 2.5 mm, Time: 10 s). The effects of the \( B_4C \) at a particle size of 10 \( \mu m \) that was added to the AA2024 matrix on hardness are shown in Figure 1. It was seen that at all sintering temperatures, as the ratio of the \( B_4C \) reinforcement increased, hardness values also increased. This was an expected result because of the higher harness value of the \( B_4C \) that was added to the matrix compared to the matrix material. The highest hardness value was found as 71.73 HB in the composite specimen produced using 20% \( B_4C \) and sintered at 595°C. Similarly, in the study conducted by Karakoc H. et al. using Al6061 as the matrix material and \( B_4C \) and \( SiC \) powders as the reinforcement materials, it was found that the composite materials including reinforcements were harder than the unreinforced Al6061 alloy, where the unreinforced alloy had a mean hardness of 50 HB, and the 12% \( B_4C \) composite had a mean hardness of 76 HB [12]. Likewise, in their hardness tests on \( SiC \)- and \( B_4C \)-reinforced Al7071-matrix composite materials they produced with the PM method, Pul M. and Baydaroglu V. observed that as the reinforcement particle ratio in the structure increased, hardness values also increased [6]. Hasirci H. and Gil F. also produced AMCs with \( B_4C \) reinforcement and examined their mechanical properties. They found higher hardness values in the composite material produced with \( B_4C \) reinforcement addition to the Al [17]. Additionally, Ergul E, Kurt H. I., Civc C. and Eycic G. mixed 50% MgO and 50% carbon nanotubes (CNT) by weight with the die casting method, added these reinforcement materials to the Al2024 matrix material at ratios of 0.2, 0.5, 1, and 2% by weight.
produced MgO/CNT-reinforced Al2024 composite materials, and investigated hardness results. They observed that the hardness values of the specimens increased by reinforcement addition [18]. Şenel M. C., Gürbüz M. and Koç E. produced composite materials with the PM method by adding Si₃N₄ reinforcement to the aluminum matrix at ratios varying in the range of 0-12% and examined the effects of the reinforcement ratio on the mechanical properties of the composites. According to the results of their experiments, while the hardness value of the pure aluminum was approximately 28 HV, hardness could be increased to 58 HV by adding 9% Si₃N₄ by weight [19].

Table 1. Chemical composition of the AA2024 matrix material (%). [20]

<table>
<thead>
<tr>
<th>Matrix Material Chemical Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA2024</td>
</tr>
<tr>
<td>Fe</td>
</tr>
<tr>
<td>0.50</td>
</tr>
</tbody>
</table>

Figure 1. Changes in the hardness values of AA2024 + 10 μm B₄C depending on temperatures and reinforcement ratios.

Figure 2 shows the effects of the B₄C+SiC added into the AA2024 matrix on hardness. Accordingly, in all specimens sintered at different temperatures, as the ratio of SiC increased, hardness values also increased. The reason for this result was the fact that the hardness values of the B₄C and SiC reinforcement materials were higher than that of the matrix material. The highest hardness value was found as 91.82 HB in the specimen that was produced by adding 20% B₄C + 10% SiC and sintered at 595°C. Likewise, Gündoğan K. and Özsari A. R. B. investigated the mechanical properties of composite materials produced with an AA2024 or AA6061 matrix and reinforced with B₄C and SiC particles using the pressure infiltration technique. As the pressure values of the composite materials increased, the authors observed an increase in their hardness and strength values. After they compared the reinforcement components, they concluded that the composites with the B₄C reinforcement had better mechanical properties than those with the SiC reinforcement [21].
Figure 2. Changes in the hardness values of AA2024+B₄C+SiC depending on temperatures and reinforcement ratios.

Figure 3 shows the effects of B₄C+Y₂O₃ added into the AA2024 matrix on hardness. It is seen that as the ratio of Y₂O₃ increased, hardness values also increased. The reason for this result was the fact that the hardness values of the B₄C and Y₂O₃ reinforcement materials were higher than that of the matrix material. The highest hardness value among the AA2024-matrix B₄C+Y₂O₃-reinforced composite materials was found as 85.33 HB in the specimen that was produced by adding 20% B₄C + 10% Y₂O₃ and sintered at 595 °C.

Figure 3. Changes in the hardness values of AA2024+B₄C+Y₂O₃ depending on temperatures and reinforcement ratios.

In the composites that were produced, in general, hardness values were observed to increase along with increased reinforcement ratios and sintering temperatures.

2.3 Density

The density measurements of the composite materials were made using a RADWAG AS 220/C/2 brand density measurement device based on the Archimedes principle. Figure 4 presents the effects of B₄C at a particle size of 10 μm that was added into the AA2024 matrix on density and porosity. Accordingly, in all specimens sintered at different temperatures, as the ratio of B₄C reinforcement increased, density decreased, and porosity increased. The density values of the examined specimens were compatible with those reported in the literature, and as the B₄C reinforcement ratio increased, density decreased. The main reason for this was the increased porosity that was observed and the fact that B₄C is a material that has a low density.

Similarly, in the Al2024-matrix B₄C- and TiB₂-reinforced composite materials produced by Pul M., as the ratio of reinforcement increased, the ratio of pores between TiB₂ and B₄C particles and the
aluminum powder increased, and thus, density values decreased [22]. Topcu İ., Dikici M. and İpek C. also investigated the density and wear resistance of aluminum-matrix B$_4$C-reinforced composite materials. The lowest density value was observed in the Al/B$_4$C composite with the highest B$_4$C ratio by weight (15%). It was seen that the density values of the specimens decreased along with increased B$_4$C ratios, but the values of the specimens with the same B$_4$C reinforcement ratios increased along with increased sintering temperatures [23].

The effects of B$_4$C+SiC added into the AA2024 matrix on density are shown in Table 2. Accordingly, as the ratio of SiC that was added increased, the ratio of porosity also increased. The effects of B$_4$C and Y$_2$O$_3$ added into the AA2024 matrix on density are shown in Table 3. Accordingly, in all specimens sintered at different temperatures, as the ratio of Y$_2$O$_3$ that was added increased, both density and porosity increased.

Table 2. Changes in the density values of AA2024 + B$_4$C+SiC depending on temperatures and reinforcement ratios.

<table>
<thead>
<tr>
<th>Sequence No</th>
<th>AA2024</th>
<th>$%$ B$_4$C</th>
<th>$%$ SiC</th>
<th>AA2024 particle size (μm)</th>
<th>$%$ B$_4$C particle size (μm)</th>
<th>$%$ SiC particle size (μm)</th>
<th>Sintering temperature (°C)</th>
<th>Theoretical density value (g/cm$^3$)</th>
<th>Experimental density value (g/cm$^3$)</th>
<th>Pore ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>77</td>
<td>20</td>
<td>3</td>
<td>100-150</td>
<td>22-59</td>
<td></td>
<td>550</td>
<td>2,470</td>
<td>2,495</td>
<td>10,31</td>
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<tr>
<td></td>
<td></td>
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<td>2</td>
<td>70</td>
<td>20</td>
<td>10</td>
<td>100-150</td>
<td>22-59</td>
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<td>550</td>
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<td>2,483</td>
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<td>10,77</td>
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<td>595</td>
<td>2,499</td>
<td>2,499</td>
<td>10,20</td>
</tr>
</tbody>
</table>
Table 3. Changes in the density values of AA2024 + B4C+Y2O3 depending on temperatures and reinforcement ratios.

<table>
<thead>
<tr>
<th>Sequence No</th>
<th>% AA2024</th>
<th>% B4C</th>
<th>% Y2O3</th>
<th>Particle size (μm)</th>
<th>B4C particle size (μm)</th>
<th>Y2O3 particle size (μm)</th>
<th>Sintering temperature (°C)</th>
<th>Theoretical density value (g/cm³)</th>
<th>Experimental density value (g/cm³)</th>
<th>Pore Ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>77</td>
<td>20</td>
<td>3</td>
<td>100-150</td>
<td>22-59</td>
<td>10</td>
<td>550</td>
<td>2,501</td>
<td>10,93</td>
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<td></td>
<td></td>
<td></td>
<td>575</td>
<td>2,519</td>
<td>10,29</td>
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<td>595</td>
<td>2,523</td>
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<td>11,94</td>
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<td></td>
<td>595</td>
<td>2,635</td>
<td>11,06</td>
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</table>

2.4 Wear Test

The wear tests were carried out with the Tribometer pin-on-disk wear testing device shown in Figure 5 under a 2N load for 15 minutes (3.516 mm track diameter, 20000 mm sliding distance, and 2000 cm/s linear velocity). Like the density test results, as the ratio of B4C reinforcement increased, wear values decreased. As seen in Figure 6, in all specimens sintered at different temperatures, wear values decreased along with increasing reinforcement ratios. These results were similar to the results of other studies conducted with composite materials. Hasircı H. and Gül F. produced Al-matrix B4C-reinforced composite materials and investigated their wear behaviors. They reported that the hardness of the composite increased due to the B4C reinforcement particles added into the matrix, the pure Al material had the lowest hardness values, and in general, wear amounts decreased in the composites in comparison to the pure Al material as the ratio of reinforcement increased [17]. Likewise, Karakoç H. observed lower wear amounts based on weight loss as a result of B4C and Si3N4 reinforcement addition [24]. In another study by Karakoç H., Al7075/B4C and Al7075/B4C/Si3N4 composite materials were produced using the powder metallurgy method, and their wear behaviors were analyzed. The researcher determined a decrease depending on increased ratios of B4C and Si3N4 by weight and found the lowest weight loss value in the Al7075/15%B4C/2%Si3N4 material [24]. Furthermore, Ark H. studied the effects of SiC as a reinforcement component on the wear properties of aluminum-matrix composite materials. The author used PM as the production method for Al-SiC composite materials and the pin-on-disk method in the wear test. It was observed that the weight loss value decreased by 48.4% to 79.6% when SiC was added at 10% by weight to the matrix [25]. Aksöz S. and Bostan B. produced 10/20% B4C-reinforced AA2014-matrix composite materials using the casting method and sintering at 600°C for 2 hours after casting and examined the effects of reinforcement ratios on wear behaviors. They found lower wear-related weight loss as a result of adding increased ratios of B4C reinforcement and sintering. The lowest weight loss was observed in the composite material containing 20% B4C that was sintered at 600°C for 2 hours [26].
Figure 5. Wear tests of the composite specimens (a: Weighing before the test; b: Wear test; c: Weighing after the test).

Figure 6. Changes in the weight loss values of AA2024 + 10 μm B₄C depending on temperatures and reinforcement ratios.
Figure 7 presents the changes in the weight loss values of AA2024+(B₄C+SiC) and AA2024+(B₄C+Y₂O₃) along with increased sintering temperatures. It is seen that with the increase in the sintering temperature, wear values decreased, and the AA2024+(B₄C+SiC) composite had higher wear resistance values in comparison to the AA2024+(B₄C+Y₂O₃) composite.

2.5 SEM

The microstructures of the specimens were examined using a JEOL JSM-6060 brand SEM device.

Figure 8. SEM image of 85%AA2024+15%B₄C (10 μm) sintered at 575°C (x1000).

Figure 9. SEM image of 77%AA2024+20%B₄C (22-49 μm) +3%SiC sintered at 575°C (x500).

It was observed in the microstructure of the 77%AA2024+20% B₄C (22-59 μm) +3% SiC composite shown in Figure 9 that the distributions of B₄C and SiC were generally homogeneous but occasionally non-homogeneous, and the structure was non-porous in general.

As seen in Figures 8 and 9, although both materials were sintered at the same temperature, the 77%AA2024+20% B₄C (22-59 μm) +3%SiC composite had a lower porosity value because a more compacted structure was obtained with the addition of SiC (10 μm).
Figure 10 shows the microstructure of the composite that had lower porosity due to the more compacted structure that was obtained with the addition of 10 μm Y₂O₃.

3. Results

The conclusions made based on the results of the experiments that were conducted in this study are given below.

1- As the reinforcement particle ratio by weight increased, the hardness values of the composites increased, and in comparison to the unreinforced AA2024 matrix material, the hardness values of the composites were approximately 2.5 times higher.

2- The density values of the 10 μm B₄C-reinforced composites decreased along with increasing reinforcement ratios.

3- With the increase in the reinforcement particle ratio by weight, there was a decrease in the wear-related weight loss values of the composites.

4- The SEM examinations (Figures 9 and 10) showed lower porosity values depending on the addition of SiC (10 μm) and Y₂O₃ (10 μm) due to the more compacted structure that was obtained.

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Author Contributions

Halit Doğan: He planned, designed and performed the analysis.

Yılmaz Mutlu: He designed the study, made the statistical analyzes and wrote the article.

Ethics

There are no ethical issues after the publication of this manuscript.

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