

# Investigation of Microstructure and Tribological behavior of WE43/nano B<sub>4</sub>C Composites Produced by Spark Plasma Sintering

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Keywords	Abstract
Spark Plasma Sintering	In this study, composite samples of WE43 (Mg-4Y-3RE-Zr) reinforced with nano-B4C particles in
WE43	different ratios (0.5 and 2 wt.%) were prepared by spark plasma sintering (SPS). The powders were mixed in a 3-dimensional ball mill at 300 rpm. The mixed powders were then hot pressed under 35 MPa
B4C	pressure at 525 °C for 6 min. XRD and FESEM-EDS instruments were used to characterize the
Tribology Behavior	composite samples. Microhardness and wear tests were performed to designate the mechanical properties. It was found that the highest hardness occurred in the composite sample with 2% nano-B4C
Mechanical Properties	composites. It was also found that tribological properties improved with the increase of nano-B <sub>4</sub> C content.

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## **1. INTRODUCTION**

Metal matrix composites (MMC) exhibit excellent mechanical properties compared to their conventional, nonreinforced counterparts. In particular, magnesium (Mg)-based MMCs occupy an important position due to their lightweight advantage (Nimityongskul et al., 2010). By reinforcing the Mg matrix with a hard ceramic phase, properties for example high hardness, high strength, and low wear resistance can be improved. Therefore, Mg, which has the lowest density among metals, is increasingly used in aerospace, automotive, and medical applications (Ghasali et al., 2017a). Among Mg alloys, WE43 alloy, which have stand out the attention of researchers in recent years, are preferred because of their superior mechanical and microstructural properties compared to other Mg alloys, as they contain rare earth elements. Despite neodymium, yttrium and other rare earth elements exhibit properties such as good mechanical performance at high temperatures, flame retardancy and high temperature creep resistance, studies are continuing to improve the problems in wear properties (Banijamali et al., 2022).

Studies have long been conducted to reinforce Mg-based alloys with ceramic particles to increase their strength. It is well known that various ceramics such as SiC, Al<sub>2</sub>O<sub>3</sub>, ZrB<sub>2</sub>, TiB<sub>2</sub>, and B<sub>4</sub>C are dispersed in the matrix phase as reinforcing materials during the fabrication of MMC. B<sub>4</sub>C is a very hard ceramic with low density, high chemical resistance and abrasion resistance. It is well known that B<sub>4</sub>C reinforcement of the Mg matrix increases the interfacial bonding strength, hardness and wear resistance of the composite (Taşcı et al., 2013). Ceramic reinforced metal matrix composites (MMCs) can show very good mechanical and wear properties when compared to non-reinforced metals (Yadav et al., 2022). It is extremely important that the composites have a defect-free microstructure and maintain their mechanical properties. For this purpose, it is necessary to distribute the reinforced ceramics homogeneously in the matrix phase. It is very difficult to

achieve such homogeneous distribution, especially in the casting process. Therefore, it is possible to produce a fine and non-porous microstructure using the powder metallurgy (P/M) technique (Ghasali et al., 2017b). Spark plasma sintering (SPS), which has occupied an important place in the P/M technique in the last decade, makes it possible to produce metal alloys and MMCs under vacuum, at very low pressing pressure and in a very short time (Saheb et al., 2012).

In this study, nano- $B_4C$  ( $nB_4C$ ) was added to the WE43 alloy in amounts of 0.5% and 2% using the SPS technique to provide homogeneous mixing of the powders. Microstructure characterization and mechanical properties of these produced composites focused on improving their wear performance.

## 2. MATERIAL AND METHOD

#### 2.1. Production Method of Composite Specimens

The matrix WE43 powder with an average diameter of 45  $\mu$ m was supplied by Magnesium Elektron<sup>TM</sup> ltd. The nB<sub>4</sub>C (55 nm) was provided by Nanografi Nanotechnology<sup>TM</sup> and used for 0.5 and 2% nB<sub>4</sub>C to prepare the composite powders. In order to mix the starting powders homogeneously, the mixing process was carried out for 4 hours at 300 rpm in a 3D ball mill with an atmospherically controlled chamber. Table 1 shows the chemical composition of WE43.

Table 1.	WE43	Chemical	Composition
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Element	Mg	Y	Nd	Gd	Z
WE43	Bal	3.72	2.13	1.11	0.41

Powders with different ratios of  $nB_4C$  in the WE43 alloy were prepared by SPS. A custom-made graphite mold was used to press the powders. The powders placed in the mold were first recompressed at a pressure of 5 MPa at room temperature. The mixed composite powders were then pressed under a load of 35 MPa at 525 °C for 6 minutes.

## 3. RESULTS AND DISCUSSION

## **3.1. Examination of Powders**

The images of the initial WE43, nB<sub>4</sub>C, and mixed WE43/nB<sub>4</sub>C composite powders used in the study are shown in Figure 1. Mixing of the composite powders was performed in a 3D high energy mixer. Figure 1a shows that the WE43 powder used as matrix alloy has a spherical shape. In Figure 1b, the nB<sub>4</sub>C particles were found to have a complex shape with sharp corners. By examining the images, a series of grooves and micro ploughing, plastic deformations parallel to the sliding direction, debris and significant crater areas were visible on the worn surface of the unreinforced WE43 matrix alloy. It can also be seen that the morphology of the matrix powder does not change and remains spherical during the mixing process.

## 3.2. Microstructure Characterization

The XRD patterns of the WE43/%2 nB<sub>4</sub>C composites produced by SPS are shown in Figure 2. Examination of the XRD peaks shows that the produced composites contain  $Mg_{41}Nd_5$  intermetallic phase and  $\alpha$  Mg matrix phase. It was found that the rare earth elements yttrium and neodymium form compounds with the matrix phase Mg to different extents. Since the crystal structures of these phases are compatible with the matrix, they are believed to increase the obstacle of the composite by preventing dislocation movement. The matrix alloy in the XRD patterns was found to increase slightly with the addition of nB<sub>4</sub>C particles, along with additional peaks corresponding to nB<sub>4</sub>C (Tang et al., 2022).



*Figure 1. a)* WE43 powder morphology, *b*) *nB*<sub>4</sub>*C* ceramic reinforcement powder, *c*) WE43/*nB*<sub>4</sub>*C* composite powder



Figure 2. XRD analysis of WE43 alloy and WE43/nB<sub>4</sub>C composites

After the spherical powders were subjected to 35 MPa pressing in the SPS device and sintered at 525 °C, the surface images of all samples were examined using FESEM to characterise the microstructure. Figure 3a shows the unreinforced WE43 alloy, Figure 3b indicates the 0.5% nB<sub>4</sub>C composite, and Figure 3c reveals the microstructure images of these samples fabricated with 2% nB<sub>4</sub>C ceramic reinforcement. When the images were examined, no significant difference was found in the grain sizes of these unreinforced and composite specimens. This is because the driving force required for grain size growth and the absence of deformation in the powder morphologies during the mixing process play a certain role (Shuai et al., 2021). It is desirable that the grain sizes are close to each other during the sintering process. It was found that nB<sub>4</sub>C reinforcement ceramics are generally formed at grain boundaries. It was found that the phases that appear as white dots in the grains are phases such as Mg<sub>41</sub>Nd<sub>5</sub>, Y<sub>2</sub>O<sub>3</sub>. It is therefore suspected that the WE43 alloy contains a rare earth element that affects the growth kinetics of the intermetallics.



*Figure 3. a)* WE43 alloy, *b)* WE43/0.5% nB<sub>4</sub>C composite, *c)* WE43/2% nB<sub>4</sub>C

By studying the microstructure of WE43 by FESEM/EDS (Figure 4), it was found that the precipitates at the grain boundaries are rich in neodymium and yttrium, which are determined and homogeneously distributed in the matrix structure of the  $nB_4C$  ceramic particles.



Figure 4. FESEM/EDS analysis result of WE43/2% nB<sub>4</sub>C composite material

## **3.3 Mechanical Properties**

By examining the density graph (Figure 5a) of WE43 alloy and WE43/ nB<sub>4</sub>C composites, it was seen that the highest density occurred in WE43 alloy (99.00%) and the lowest density in WE43/2 nB<sub>4</sub>C composite (97.8%). It is known that composites reinforced with ceramic particles have lower density compared to unreinforced matrix specimens. Also, by looking at the hardness graphs in Figure 5b, it can be seen that the sample with the highest hardness is WE43/2 nB<sub>4</sub>C and the sample with the lowest hardness is the unreinforced WE43 alloy. The B<sub>4</sub>Cs in the matrix phase, which is soft along with the ceramic particle reinforcement, accumulate at the grain boundaries and prevent dislocation movement. Therefore, an increase in the strength values of the composites is observed. Comparing the hardness value with that of the unreinforced WE43 alloy, it is found that it increases by about 5% at 0.5% nB<sub>4</sub>C. Similarly, when comparing the hardness values of the unreinforced WE43 alloy and the composite reinforced with 2% nB<sub>4</sub>C, it was found that there was an increase of about 12%. The reinforcement by nB<sub>4</sub>C ceramic particles was homogeneously distributed in the structure and increased the strength.



Figure 5. a) Density, b) Hardness

## 3.4. Tribological Behavior

## 3.4.1. Weight loss

Figure 6 shows the weight loss of the unreinforced WE43 matrix alloy and the WE43/nB<sub>4</sub>C composites with different reinforcements under 15 N load. The weight loss decreases with increasing reinforcement ratio compared to the matrix material. It is known that various properties such as grain size, porosity in the structure, strength and hardness affect the wear behavior of materials. It is believed that the reinforcement particles act as a coercive factor at the grain boundaries, leading to the development of wear resistance (Moheimani et al., 2021).



Figure 6. Weight loss

## 3.4.2. Wear mechanisms

FESEM analysis was performed to investigate the wear mechanisms of WE43 alloy and WE43/  $nB_4C$  composites during wear testing and the effects of the presence of  $nB_4C$  particles in different proportions on wear behavior. Figure 7. SEM shows images of the worn surfaces of the WE43 alloy and WE43/ $nB_4C$  composites subjected to the wear test under 15N load. By examining the images, a series of grooves parallel to the sliding direction, debris, plastic deformations and significant crater areas were visible on the worn surface of the unreinforced WE43 matrix alloy. It is assumed that this situation exhibits typical characteristics of abrasive wear.

## 4. CONCLUSION

In this study, powder-metal composites were produced by the SPS method after homogeneously mixing WE43 alloy and WE43/nano  $B_4C$  powder in a 3D ball mixer. The microstructure, hardness and wear properties of the composites were investigated. The following conclusions can be drawn from the results.

The mixing process in an atmospherically controlled 3D ball mill showed that the  $nB_4C$  particles were homogeneously distributed at the grain boundaries. While the highest density value was observed in the

unreinforced WE43 alloy, the density values decreased with increasing  $nB_4C$  particle addition. The lowest density value was found in the WE43/2%  $nB_4C$  composite. While the lowest hardness occurred in the unreinforced WE43 alloy, the highest hardness was observed in the WE43/ 2%  $nB_4C$  composite. It was observed that the hardness values increased with increasing  $nB_4C$  ratio. The addition of  $nB_4C$  particles improved wear resistance and minimized weight loss.



Figure 7. Worn surface SEM pictures a) WE43 alloy, b) WE43/0.5% nB<sub>4</sub>C composite, c) WE43/2% nB<sub>4</sub>C

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## **CONFLICT OF INTEREST**

The authors declare no conflict of interest.

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