

Original Article

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# Microstructural and wear properties of the Al-B<sub>4</sub>C composite coating produced by hot-press sintering on AA-2024 alloy

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**Abstract** In this study, the surface of an AA-2024 alloy was covered with reinforced composite coating using hotpress sintering method. Al and B<sub>4</sub>C powders were synthesized through mechanical alloying technique and coated on the AA-2024 substrate at different rates. The microstructure of the intermediate transition region formed between the substrate (AA-2024 Al alloy) and the coating layer (Al/B<sub>4</sub>C MMCs), the microstructure of the Al/B<sub>4</sub>C metal matrix composites (MMCs) coating, the microhardness, and the adhesive wear resistance of the Al/B<sub>4</sub>C MMCs coating layer were investigated. It was observed that B<sub>4</sub>C powders homogeneously dispersed in the microstructure of the Al/B<sub>4</sub>C MMCs coating layer, moreover, the Al matrix and B<sub>4</sub>C reinforcement particles were bonded without a gap. It was also determined that an interface bonding occurred between Al/B<sub>4</sub>C MMCs coating layer and the AA-2024 substrate. Accordingly, it was determined that with the increase of B<sub>4</sub>C reinforcement particle ratio, the hardness of the coating layer, and the wear resistance increased.

## 1. Introduction

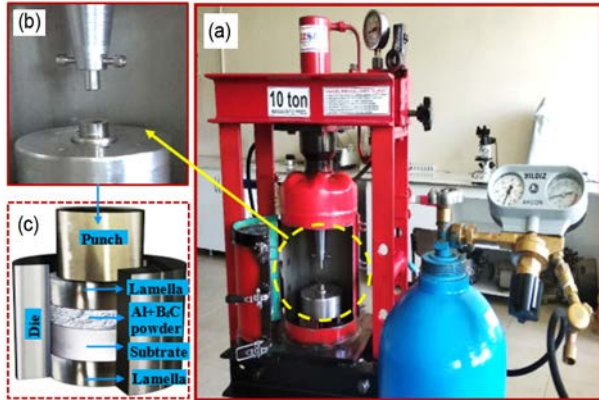
Vehicles and systems produced in recent years need to be light and high-strength to use energy efficiently. In this respect, some features such as high strength/ weight ratio, easy forming and processing, low density, good thermal and electrical conductivity, high corrosion resistance, high-toughness, and endurance in Al-based alloys must have be improved. Among aluminum alloys, AA-2024 alloy, which main alloying element is copper, comes into prominence because of its characteristics such as formability, age hardening, elastic modulus, mechanical strength [1].

Many coating techniques such as arc weld [2, 3], micro-arc oxidation [4, 5], plasma spraying [6, 7], sol-gel [8, 9], reactive magnetron sputtering [10, 11], laser [12, 13], and rarely with vacuum hotpress sintering [14-16], used by different researchers to improve material surface. Coating of a metal surface with MMCs enables different characteristics to assemble on the coating layer. Coating of Al alloys also takes an important place [8, 17-19]. Coating processes provide features, e.g., mechanical, thermal, and electrical conductivity, high damping capability, resistance to oxidation, low density, and good wear resistance [20]. In MMCs materials, due to very great hardness, wear and impact resistance, good chemical stability, use of reinforcement particles such as B<sub>4</sub>C [21-26], SiC [27-29], Al<sub>2</sub>O<sub>3</sub> [16, 30], TiC [31-33] has increased. Especially, particle-reinforced composites with aluminum metal matrix composites (AMMCs) are preferred in space, aviation, automotive, and many other sectors because of their high strength/weight ratio, good mechanical and non-magnetic features (paramagnetic substances) [34, 35]. Besides, AMMCs are preferred because they help design lighter products and significantly increase fuel efficiency to reduce material weight by more than 50 % compared to low-carbon steels [36].

In recent years, most coating and protection techniques have been used to refine the surface

Table 1. The chemical compound of AA-2024 aluminum alloy.

Fe	Si	Cu	Cr	Mn
0.5	0.5	3.8-4.9	0.1	0.3-0.9
Mg	Zn	Ti	Al	
1.2-1.8	0.25	0.15	Balanced	

Fig. 1. Unit for coating AA-2024 substrate with Al-B<sub>4</sub>C composite.

of AA-2024 alloy, which is widely used in space, aviation, and automotive technologies. However, there is no study that investigated the use of the hot press sintering method in the coating of AA-2024 substrate materials. In this study, the AA-2024 alloy surface was coated with Al-B<sub>4</sub>C MMCs, using the hot press sintering method. Surface morphology of AMMCs coating layer and interface microstructure formed on the substrate, the microhardness of coating layer, and wear resistance have been thoroughly investigated.

## 2. Experimental procedure

Fig. 1 shows the experimental setup designed for coating the AA-2024 substrate with Al-B<sub>4</sub>C. In this study, firstly, the powders were mixed, and then the substrate used in experimental studies was prepared in  $\varnothing 12 \times 5$  mm by measure. Table 1 lists the chemical compositions and their corresponding ratio used in the AA-2024 alloy substrate. The substrate was mechanically cleaned to remove the oxide layer and residual material from the surface, then washed with alcohol and dried.

For the coating layer, Al powder with size of 44  $\mu\text{m}$  and purity of 99.5 % was used, also B<sub>4</sub>C powder used in this study had 99 % purity with average particle size of 10  $\mu\text{m}$ . The volumetric powder ratio used on the coating layer and production parameters can be seen in Table 2. To ensure the homogenization of the coating powders, balls with a diameter of 10 and 5 mm and with ratio of 1:1, and ball powder with ratio of 15:1 were mixed in 3-axis turbula mixer 3 at 350 rpm for about 25 minutes.

To form the AMMCs coating layer, firstly the die was prepared, and a HSS lamella with dimension of  $\varnothing 12 \times 5$  mm was placed at the bottom. Then, it was placed after the cleaned coating surface of AA-2024 alloy substrate. Al/B<sub>4</sub>C powder

Table 2. Production parameters of AA-2024 substrate coating with Al-B<sub>4</sub>C.

Production parameters	Samples			
	S1	S2	S3	S4
B <sub>4</sub> C particle reinforcement ratio % vol	0	2.5	5	8
Weighting pressure MPa	110			
Sintering temperature °C	575			
Shielding gas	99 % argon gas			

mixture was poured over an AA-2024 alloy pad in the die.

Later, an upper piston lamella made of HSS material was put on Al/B<sub>4</sub>C powder mixture to use as upper-pressure piston. The matrix was heated up to 575 °C in the Core Mf 129 oven operating in an argon gas atmosphere and kept at this temperature for 20 minutes. Then, the sample at 575 °C was pressed under a pressure of 110 MPa on a specially designed (in argon gas environment) press bench and was kept under pressure for 5 minutes to cool down in that environment.

For XRD examinations, the coating surface of Al/B<sub>4</sub>C MMCs of the samples was mechanically cleaned, washed with alcohol, and dried. XRD was taken with the help of Rigaku Miniflex 600 diffractometer, which scanned for the range of 20-90° and with 0.005° step range, also CuK $\alpha$  radiation ( $\lambda = 0.15406 \text{ \AA}$ ) was utilized. Samples, prepared for microstructure examinations, (2 ml HF + 3 ml HCl + 5 ml HNO<sub>3</sub> + 190 ml water) were etched with keller solution for about 30 seconds. To analyze the distribution of microstructure and rigid phases in the structure, Nikon Eclipse MA200 brand optical microscope (OM) with Clemex Software and Tescan Mira3 equipment for SEM-EDS were used. Microhardness measurements were taken in Emcotest Durascan 20 device by applying a load of 50 g for 10 seconds. Microhardness measurements were evaluated by taking the arithmetic mean of the values taken from 10 different points in the cross-section of the samples. The coating surface was cleaned and dried for wear samples. Linear reciprocating wear tests were carried out under ASTM G133 standards in CSM/Anton Paar Instrument Tribometer device using a ball-on-disc mechanism. These tests were carried out under 1 N force, 250 m sliding distance, and ASTM A276 against material conditions.

## 3. Results and discussion

### 3.1 XRD analysis

Fig. 2 reveals the XRD pattern obtained for the S1 and S4 samples. The XRD graphics were taken from the surface of the MMCs coating layer, where the AA-2024 alloy is coated with Al-B<sub>4</sub>C powders. The XRD provides sharp peaks indicated Al and B<sub>4</sub>C phases [23, 37-39]. The Al phase peaks are the prominent peaks compared with those of B<sub>4</sub>C. The reason could be the lower volume fraction of the B<sub>4</sub>C phase in the structure [40]. The peaks of the B<sub>4</sub>C phase are given in Fig. 2(c). The Al main matrix peaks forming the coating layer can be seen at 38, 45, 65, and 78°, and the reflection planes are

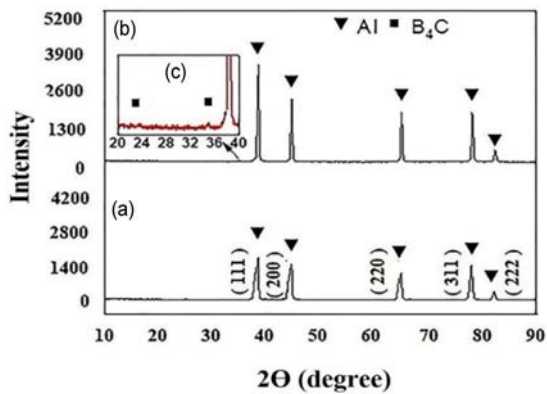


Fig. 2. The XRD pattern of samples: (a) S1; (b) S4 sample; (c) the inset of the XRD pattern magnified some selected region of the S4 sample.

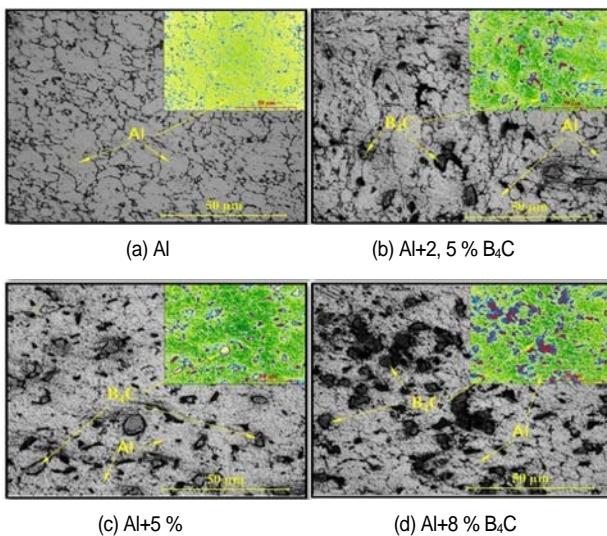


Fig. 3. OM images of produced AMMCs coating layer.

(111), (200), (220), (311), and (222), respectively [23]. The peaks of  $B_4C$  reinforcement particles were determined at  $23.35^\circ$ .

### 3.2 Micro-structural analysis

Fig. 3 shows the OM images of the coated layer for both samples. It can be seen that Al and  $B_4C$  powders, which formed MMCs coating layer are well mixed up and  $B_4C$  particles have been broken down through mechanical mixing process. In Fig. 3(a), reveals the OM image of AA-2024 coated with Al powder. Besides, Figs. 3(b)-(d) show the OM images of Al- $B_4C$  samples with different compositions that were hot-pressed on the substrate. The  $B_4C$  particles in S2, S3, and S4 samples are homogeneously dispersed and there is no coagulation in the structure. As the reinforcement material ratio on the coating layer increased,  $B_4C$  particles were clearly seen in the OM images.

In Fig. 4, SEM images of coating layer interfaces taken from cross-section of the samples are given. In Fig. 4(a), a certain

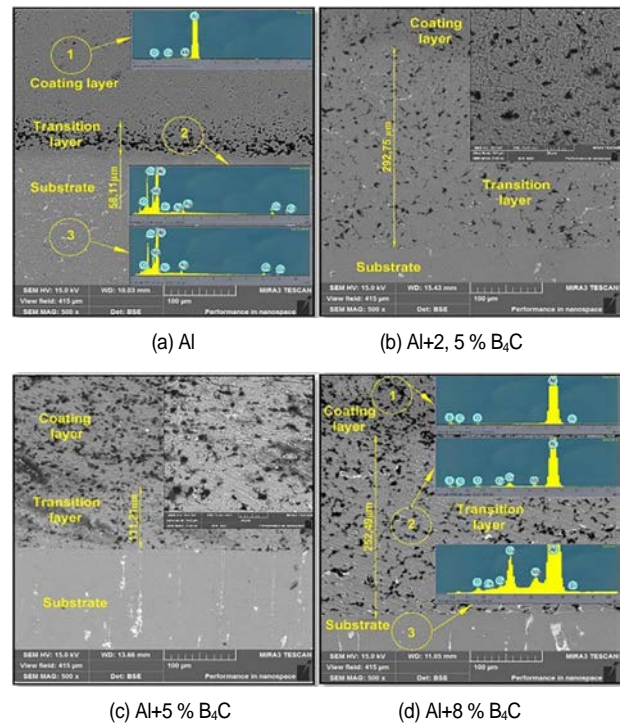


Fig. 4. SEM images and EDS analyses of interface microstructure of samples.

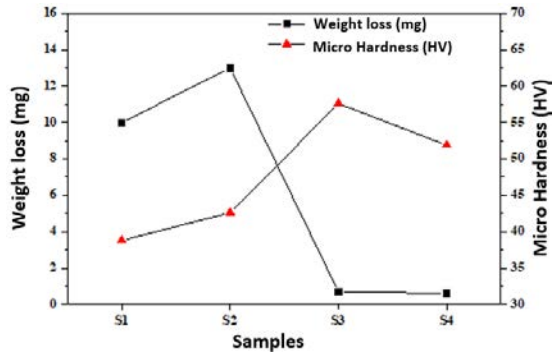
area between AA-2024 substrate and a coating is formed that indicates a metallurgical bond with a good dampening, moreover, there are no pores and cracks. It was determined that there is porous space in this area and the porosity decreases as it approaches the coating layer. The growth of pores in the diffusion region can be explained in terms of the Kirkendall effect. This is because Al tends to diffuse from the AA-2024 substrate surface towards the coating layer during the sintering process, moreover, when elements transit from the surface, i.e., from alloy to the coating layer, they leave pores on the aluminum powder coating due to the difference in diffusion velocity. Due to the difference in mobility of alloying elements, it is expected that they leave pores behind during the cooling process [37-39]. In Figs. 4(b)-(d), it is seen that  $B_4C$  particles homogeneously disperse in the Al matrix. There isn't any coagulation, while a diffusion region between substrate and coating layer can be seen. EDS analyses taken from these transition regions reveal a diffusion from substrate towards coating layer. At the X2000 magnified photo taken from the coating layer of S2 and S3 samples, it is found that the Al matrix surrounds  $B_4C$  reinforcement particles and there is no gap (Figs. 4(b) and (c)).

### 3.3 Micro-hardness and wear analysis

Hardness is a significant mechanical property to investigate the protective layer on the surface of materials. Because the hardness of the coating layer affects the wear resistance of the material [3], measurements of HV microhardness and weight loss from wear have been taken from the coating layer of the

Table 3. Microhardness and weight loss as a function of  $B_4C$  volume fraction.

Sample	Weight loss (mg)	Microhardness (HV)
S1	10	38,8
S2	13	42,6
S3	0.7	57,6
S4	0.6	51,9

Fig. 5. Microhardness and weight loss in relation to  $B_4C$  volume ratio of samples.

samples. The results acquired are given in Table 3 and Fig. 5. When these results are examined, it is found that the hardness value increases with the increase of  $B_4C$  ratio in the coating layer, while the weight loss decreases [40, 41].

By analyzing the results given in Table 3, Figs. 5 and 6, it is stated that the microhardness of the S1 sample coated with pure Al powder is low and it has a high weight loss. The increase of the  $B_4C$  ratio, which is known as a hard particle, enhances wear resistance and decreases weight loss due to the decline in contact area on the wear surface [24]. Although the  $B_4C$  ratio in S2 is 2.5 % and microhardness is higher than S1, the sample lost more weight due to the delamination wear mechanism caused by deposits during wear process. As can be seen from the OM images in Fig. 3(b), high weight loss has occurred in S2 due to the stress induced around  $B_4C$  particles, therefore it indicates that Al matrix and  $B_4C$  reinforcement particles well mixed and thus breaking off in a layer during wear. When loading is applied to composite materials produced by powder metallurgy, stress is induced around the hard reinforced particles. In Fig. 6(b), the crack observed just below the worn surface shows that high plastic stress emerges in the matrix at high temperatures and matrix is in direct contact with the material. Strain gradients that created beneath the contact surface cause subsurface crack propagation and the wear progresses through subsurface delamination [42]. In general, the microhardness increases with the rise of the  $B_4C$  ratio in the coating layer [43]. However, the hardness of the S4 is slightly less than the hardness of the S3. Also, the reducing weight loss of S3 and S4 is due to increasing microhardness (Figs. 6(c) and (d)). Besides, some researchers [44-47] have

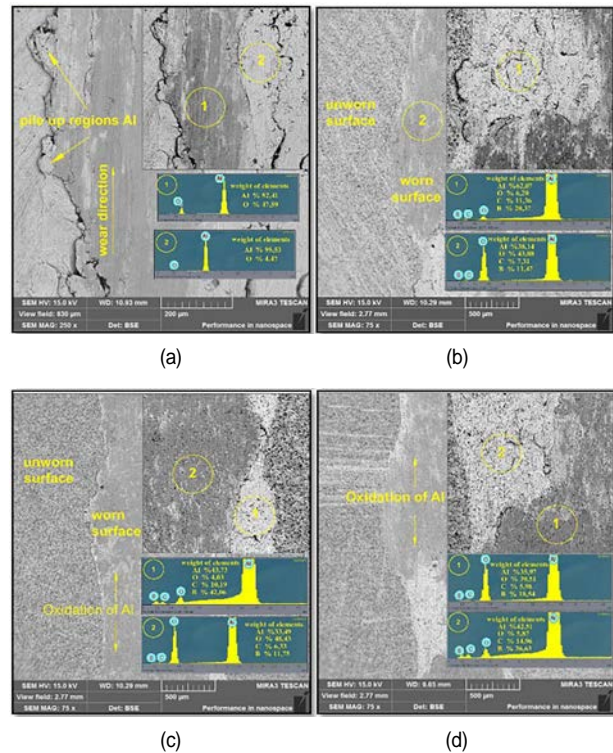


Fig. 6. SEM images and EDS analyses of interface microstructure of the samples.

suggested that the formation of aluminum oxide and boron oxide during wear diminishes the friction coefficient, and thus reduces wear loss. The EDS analysis taken from region #1 of worn samples, displays that oxygen level is low while it is detected with a higher ratio in region #2. Also in region #2 a layer formed that resisted to wear.

Fig. 7 shows the friction coefficient of samples coated with Al/ $B_4C$  MMCs, depending on sliding distance during adhesive wear. The friction coefficient is important evidence for the understanding of wear and friction performance of the materials [48]. From Fig. 7 it can be seen that friction coefficient curves in adhesive wear increase rapidly until they reach the highest value and then gradually decrease and finally it reaches a plateau. The friction coefficient curves of S1 increase to 1.20 as the highest value, then reached a constant value around 0.38, and finally gradually declined, depending on the sliding distance.

The friction coefficient showed a high fluctuation in S2 at the first stage, depending on the sliding distance. It reached the highest value at 0.96 and the stable value at 0.5. The reason for the high fluctuation in S2 is due to the local surface fracture and the accumulation of debris formed during wearing. On the other hand, its steady-state shows that the fractures are moving away from the surface [49]. S3 has shown a more stable fluctuation in friction coefficient than other examples. Also, it is seen that it changes around 0.72 and 0.54. In S4, while there is a fluctuation in the first case, it becomes stable in the course of

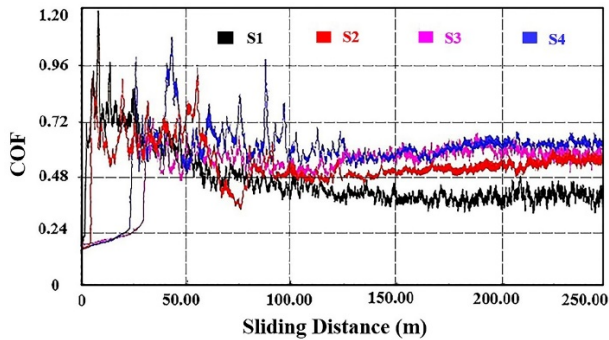


Fig. 7. The friction coefficient of produced AMMCs coating layers.

time, and the friction coefficient changes between 1.1 and 0.6. The fluctuation of friction coefficient in the first 100 m of wear distance is related to the accumulation of debris on the surface and their removal from the surface. While friction coefficient increases with the accumulation of debris, it decreases with their removal from the surface. Low fluctuation in coefficient is a sign of oxidation wear or small deposits [50].

#### 4. Conclusions

In this study, Al/B<sub>4</sub>C AMMCs were coated through the AA-2024 substrate with hotpress sintering technique. Different proportions of B<sub>4</sub>C particles were used in AMMCs coating layer. The microstructural properties of the layer and substrate interface, microhardness, and adhesive wear resistance of the coating layer formed were examined. It was seen that Al and B<sub>4</sub>C powders were homogeneously dispersed in the structure. According to microhardness results, it was determined that the hardness value on the coating layer enhanced with the increase of B<sub>4</sub>C reinforcement particle volume rate. It is also found that with the increase of B<sub>4</sub>C reinforcement particle ratio, wear resistance enhanced as well.

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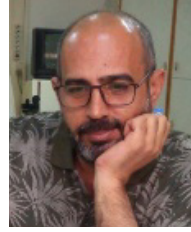
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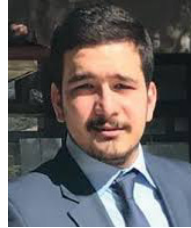
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