

## Research Article

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# Relationship between $\text{Al}_2\text{O}_3$ Content and Wear Behavior of Al+2% Graphite Matrix Composites

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**Abstract:** In this study, the microstructure and wear behaviours of aluminium composites, reinforced with different amounts of (3-12%)  $\text{Al}_2\text{O}_3$  and 2% (% vol.) graphite were investigated. The  $\text{Al}_2\text{O}_3$  and graphite were added to Al matrix and mechanically alloyed for 60 minutes. Subsequently, the mechanically alloyed powders were pressed under 700 MPa pressure and sintered at 600°C for 120 minutes. The produced aluminium composites were characterized by microstructure, scanning electron microscope (SEM), X-ray diffraction (XRD), density and hardness measurements. Afterwards, wear tests were carried out on a block on-ring type wear testing device, under three different loads and four different sliding distances. As a result, the hardness and density of composites were observed to increase due to the increase in the amount of reinforcement in aluminium composites. The highest hardness and density values were obtained in composite material containing 12%  $\text{Al}_2\text{O}_3$ . The wear tests, the lowest weight loss was also obtained in composite containing 12%  $\text{Al}_2\text{O}_3$ .

**Keywords:** Al-Graphite-  $\text{Al}_2\text{O}_3$  composite; wear; mechanically alloying

## 1 Introduction

Aluminium matrix composites (AMC) are materials that are produced with reinforcement of aluminium matrix with different materials [1, 2]. Ceramic particle reinforced aluminium matrix composites have better mechanical

properties compared to unreinforced aluminium and its alloys [3]. AMCs have high strength [4], low density [5], good fatigue and wear resistance [6–9]. AMCs have also high elasticity modulus, high hardness, high electrical and thermal conductivity, and high corrosion resistance. Especially when high temperature is implemented,  $\text{Al}_2\text{O}_3$  reinforced AMCs provide good wear resistance [10]. Therefore, they are preferred particularly in aeronautics, defense, and automotive industries [11]. Studies with AMC production investigate mainly the in-situ nucleation, spray, Physical Vapor Deposition (PVD). Mostly used methods, on the other hand, are liquid state processes [12, 13] like infiltration, compression casting, blended casting, and solid-state methods like powder metallurgy [14–16] and spray deposition [17]. Powder metallurgy method, which is commonly used in the production of materials which require high-cycle fatigue properties, is seen as a good method to produce metal matrix composites among manufacturing methods. Powder metallurgy also provides a good distribution of the reinforcement particles in the alloy matrix [18]. It also gives the advantages of low production cost, high tolerance and very little need for secondary machining processes [19]. In addition, mechanical alloying (MA), a method of powder metallurgy, compared with other particle-reinforced composite production methods, can produce AMC with better mechanical properties [6]. The MA process takes place in three stages: cold welding, strain hardening, and breakage. According to the Fogagnolo model [20], at the first stage of alloying, Al particles take the shape of a flake with the high energy milling effect occurred between ball-powder-container walls. As grain coarsening takes place in the ductile matrix phase due to cold welding, diminution is observed in the brittle reinforcement phase due to fractures. Therefore, in the following stages a composite structure is obtained by embed the particles of the reinforcement phase into powder, forming the ductile matrix. In the later phase of the process, cold welded composite powders break because of strain hardening. Broken powders refine the grain size of the composite materials which may give them high hardness and good wear resistance. It is also argued that better wear resistance and good strength can be obtained by using solid

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lubricants such as; MoS<sub>2</sub>, BN, Graphite (Gr), Graphene and CaF<sub>2</sub> in AMCs due to their self-lubricating properties [21–23]. Baradeswaran and Perumal [24] reported that the hardness, tensile, bending and compressive strengths of Al7075-Al<sub>2</sub>O<sub>3</sub>-graphite hybrid composites increased with increasing weight percentage of the ceramic reinforcement elements, and the hybrid composites containing graphite exhibited excellent wear resistance. In a similar study, Alaname and Sanusi [25] reported that hybrid composite materials containing graphite had better wear resistance than composite materials without graphite, while increased graphite ratio reduced wear resistance. Also, Premnath *et al.* [26] in a similar study they conducted, they investigated the mechanical and tribological behavior of AMCs reinforced 5% by graphite and by 5%, 10% and 15% Al<sub>2</sub>O<sub>3</sub> within Al matrix. In their results, they stated that the reinforcement amount is the dominating parameter. They also reported that the abrasive wear mechanism and oxide wear mechanisms of the wear surfaces were the dominant wear mechanism. Chu *et al.* [27], determined that in the Al-C alloy system, small pieces of graphite transform into Al<sub>4</sub>C<sub>3</sub> phase in the composition while the bigger pieces of graphite stay as graphite cores in the structure. In another study by Bostan *et al.* [28], it is stated that the MA process before sintering is not so effective in the formation of Al<sub>4</sub>C<sub>3</sub> phase. In the similar studies given above, the casting method, which is the classical production method, is generally used. However, in the produce of composite materials, MA process is an important manufacturing method especially in Al-C (graphite) alloy system

For reason, aluminium composites are produced by adding Al<sub>2</sub>O<sub>3</sub> reinforcement material to the Al-graphite matrix at different amounts by using mechanical alloying method. Besides, it is aimed to form Al<sub>4</sub>C<sub>3</sub> (in-situ) phase through Al-C reaction during the sintering process in Al<sub>2</sub>O<sub>3</sub> reinforced (ex-situ) Al-Graphite composites. This study also investigates the effects of both Al<sub>4</sub>C<sub>3</sub> phase which is formed through chemical reaction and the amounts of Al<sub>2</sub>O<sub>3</sub> added ex-situ on the microstructure, hardness and wear behaviours.

## 2 Materials and Method

In the experimental studies, aluminum powder of a size about 50 µm and 99.5% (vol.) purity was used as the matrix material. The matrix was also reinforced with %2 Graphite (Gr) and Al<sub>2</sub>O<sub>3</sub> (% vol.) of about 30 µm powder size at four different ratios (3%, 6%, 9% and 12%). Chemical composi-

tion of composite powders that were produced are given in Table 1.

**Table 1:** Chemical compositions of produced AMC powders

Element	Al (%vol.)	Graphite (%vol.)	Al <sub>2</sub> O <sub>3</sub> (%vol.)
Al+2Gr	98	2	—
+3Al <sub>2</sub> O <sub>3</sub>	95	2	3
+6Al <sub>2</sub> O <sub>3</sub>	92	2	6
+9Al <sub>2</sub> O <sub>3</sub>	89	2	9
+12Al <sub>2</sub> O <sub>3</sub>	86	2	12

Powders with the chemical composition given in Table 1 were mechanically alloyed in a planetary mill. Stainless steel milling cell was used in the MA process. In the MA operations, a 10 mm ball, 1:10 ball powder ratio, 1% ethanol as process control chemical and 60 minutes of process time were chosen. MA'ed aluminum composite powders cold pressed (700 MPa) to produce  $\varnothing 10 \times 7$  mm green compacts. The green compacts produced were sintered at 600°C for 120 min and cooled to room temperature in the furnace. Protherm HLF 50 brand heat treatment furnace was used for sintering process. Samples, prepared for micro-structural examination according to the standard metallographic procedures, were etched for 10-15 seconds in 2 ml HF, 3 ml HCl, 20 ml HNO<sub>3</sub>, 175 ml H<sub>2</sub>O (Keller's) solution. Microstructural studies of etched composite materials were conducted by Carl Zeiss Ultra Plus Gemini Fesem brand scanning electrode microscope (SEM+EDS). Also, produced composite materials were characterized by measuring X-ray diffraction (XRD), hardness, and density. For the XRD examinations, Rigaku Ultima IV brand X-Ray Diffraction Spectrometer is used. Density measurement was done with Archimedes' principle. The mean value of density measurements from three samples was calculated. Hardness measurements were made for 10 seconds as HV2 in Shimadzu brand micro hardness measurement device. Hardness measurements was calculated by using three different samples from five different points. Wear tests were done according to ASTM G77 standard in the block-on-ring wear test machine. Tests were carried out at 0.2 ms<sup>-1</sup> sliding speed, under three different loads (10, 20, 30 N) and four different sliding distances (300-1200 m). Before the wear tests, surfaces of the sample and ring were cleaned with acetone. The wear tests were done for each parameter with three different samples, and the average weight loss and friction coefficient were calculated. Wear ratios were calculated by using the weight loss results obtained

in Equation 1.

$$Wa = \frac{\Delta m}{(M.s.\rho)} \quad (1)$$

where  $Wa$  is the wear ratio,  $\Delta m$  is the weight loss determined after the wear test (g),  $M$  is the load used in the tests (N),  $s$  is the sliding distance (m) and  $\rho$  is the density of the worn sample ( $\text{g}/\text{cm}^3$ ). After the wear tests, active wear mechanisms were tried to be determined with examining the SEM images of the worn surfaces.

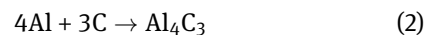
### 3 Results and Discussion

SEM images of AMCs with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement are given in Figure 1.

Microstructural SEM images of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement given in the Figure 1 clearly show the dispersed graphite in the structure. The first one is the agglomeration, which is likely to occur during the MA processes, and the second is the cold welding of the powders caused by the continuous high energy collision of balls to each other and to the walls of the container during the process. Microstructure SEM images (Figure 1 b, c, d and e) show that there are pores in the structure of the composites produced. Formation of these pores in the structure is related to the AMC production method. Because, it is very difficult to achieve full density in materials produced by powder metallurgy. This was also reported in some previous studies [29]. The reinforcing elements ( $\text{Al}_2\text{O}_3$ ) used in the production of particle reinforced composites can also be seen in the SEM images given in Figure 1. In the structure of aluminium composites, it is understood that  $\text{Al}_2\text{O}_3$  ceramic reinforcement elements are generally in the grain boundaries. Ozyurek *et al.* [6] determined that with the effect of alloying, composite powders which were mechanically alloyed for 120 minutes were deformed enough, and hard  $\text{Al}_2\text{O}_3$  particles were embedded into the matrix. However, in this study, it is seen that 60 minutes mechanically alloyed composite powders adhere to aluminium grains during alloying but are not embedded enough and are located at the grain boundaries. It can also be said that  $\text{Al}_2\text{O}_3$  particles are dispersed more evenly in the structure due to their good embedding in Al particles. XRD result of the composite material produced with 12%  $\text{Al}_2\text{O}_3$  reinforcement is given in Figure 2.

XRD result of the composite material produced with 12%  $\text{Al}_2\text{O}_3$  reinforcement given in Figure 2 show that there is  $\text{Al}_4\text{C}_3$  compound in the composition of composite materials which was expected to develop in-situ along with  $\text{Al}_2\text{O}_3$  which was added into the mechanically alloyed alu-

minium composite as reinforcement. Formation of this compound in the structure is an expected situation. Formation reaction of  $\text{Al}_4\text{C}_3$  and reaction temperature are given in Equation 2 and Equation 3.

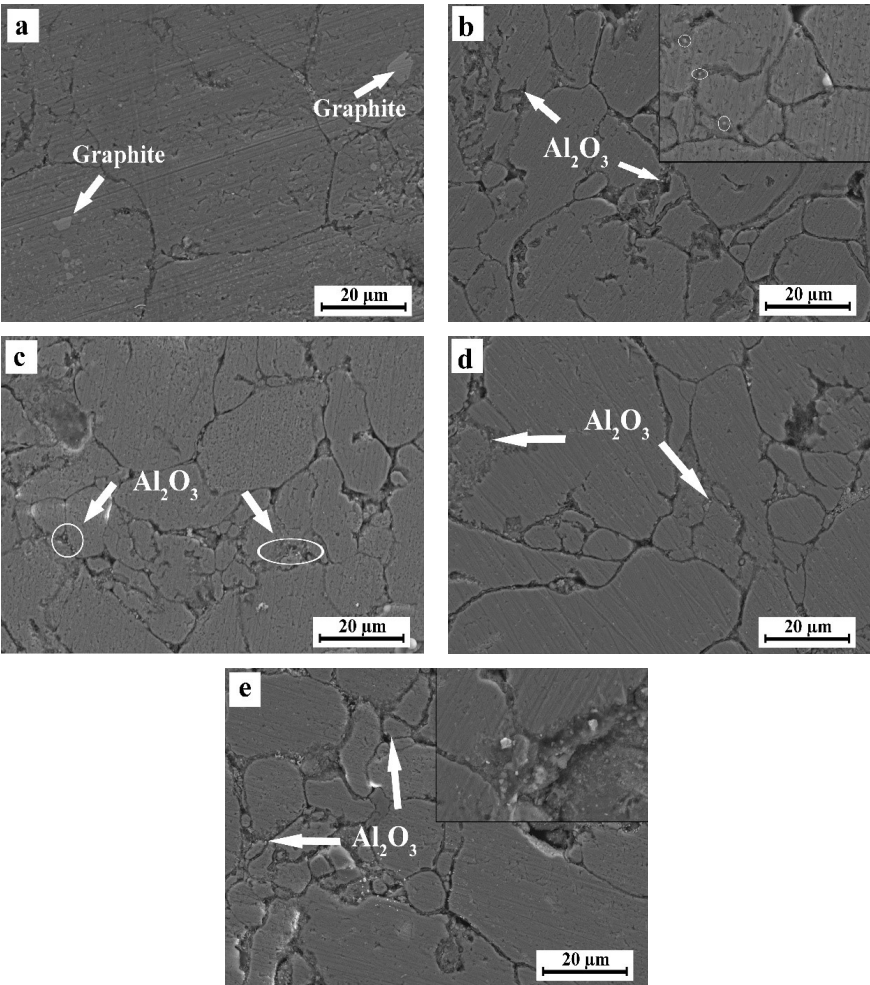


$$G^\circ = -56600 + 10T(\text{cal}) \quad (3)$$

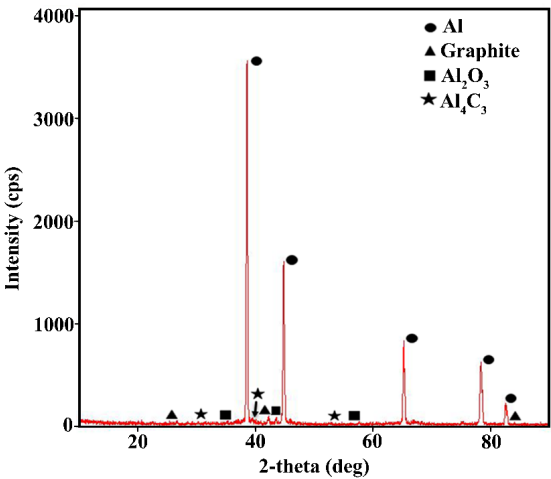
where,  $\Delta G^\circ$  is Gibbs free energy, and  $T$  is reaction temperature in Kelvin. According to Equation 2, Gibbs free energy at the sintering temperature ( $600^\circ\text{C}$ ) is approximately  $-47870$  cal. Therefore, it is an indication of the energy that is enough for formation of in-situ reactions at the temperature of the sintering process. Bostan *et al.* [28], stated that in the Al-C system produced with mechanical alloying, nano sized  $\text{Al}_4\text{C}_3$  compound is formed in the structure of the alloy with sintering. Besides, Chu *et al.* [27], determined that small graphite pieces turn completely into  $\text{Al}_4\text{C}_3$  phase, and bigger graphite pieces are seen in the structure as graphite cores that have not transformed. This situation also explains the graphite particles seen in Figure 1a. The changes in hardness and density of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement are given in Figure 3.

Density measurement results of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement given in Figure 3. Indicate that density increases by the amount of reinforcement which has a higher density value. The lowest density was measured as  $2.628 \text{ g}/\text{cm}^3$  in non-reinforced material (Al+2Gr), while the highest density was obtained as  $2.735 \text{ g}/\text{cm}^3$  in the sample with 12%  $\text{Al}_2\text{O}_3$ . Similar results were obtained in a previous study [14]. Similarly, hardness results given in Figure 3 shows that hardness increases with increasing ratio of reinforcement. The lowest hardness is obtained as 687 HV from the Al+2Gr alloy without reinforcement, and the highest hardness is obtained as 750 HV from the composite material with 12%  $\text{Al}_2\text{O}_3$  reinforcement. Hard oxide ( $\text{Al}_2\text{O}_3$ ) particles improve the hardness of the matrix which is relatively ductile. This condition indicates that the reinforcement material improves the resistance of the matrix against the plastic deformation and contributes to the hardness of the composite material [30]. Weight losses and wear rates of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement are given in Figure 4.

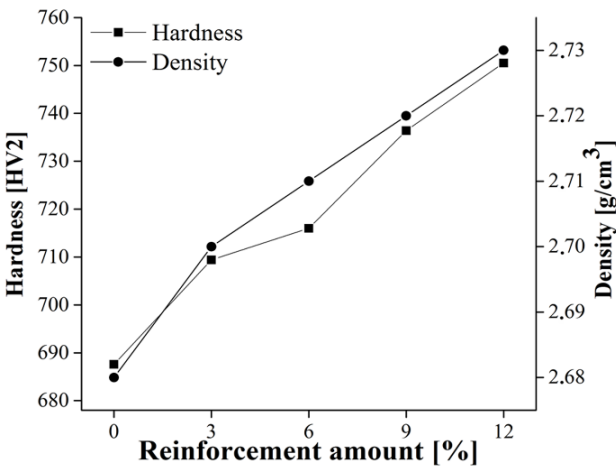
Weight loss results in Figure 4 show a decrease in weight loss by increasing amount of  $\text{Al}_2\text{O}_3$  reinforced to the Al+2Gr matrix. Weight loss results supports the hardness test results given in Figure 3. While the highest weight loss result is obtained with the sample having the lowest



**Figure 1:** Microstructural SEM images of composite materials with different amounts of Al<sub>2</sub>O<sub>3</sub> reinforcement; Al+2Gr (a), 3% Al<sub>2</sub>O<sub>3</sub> (b), 6% Al<sub>2</sub>O<sub>3</sub> (c), 9% Al<sub>2</sub>O<sub>3</sub> (d) and 12% Al<sub>2</sub>O<sub>3</sub> (e).

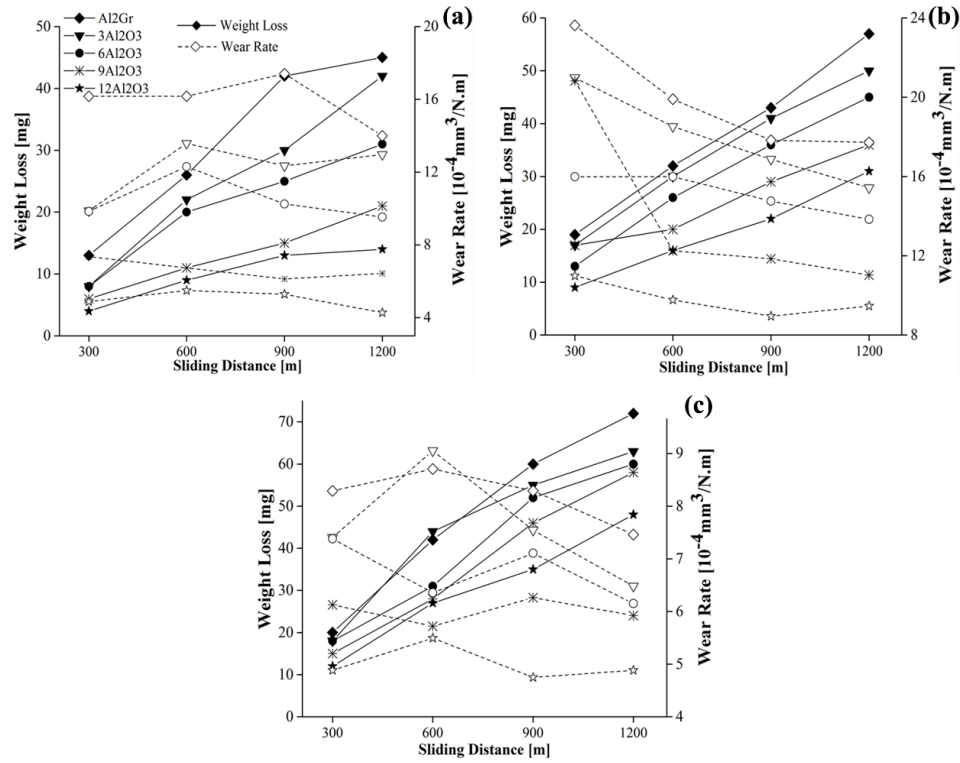


**Figure 2:** XRD result of the composite material produced with 12% Al<sub>2</sub>O<sub>3</sub> reinforcement.

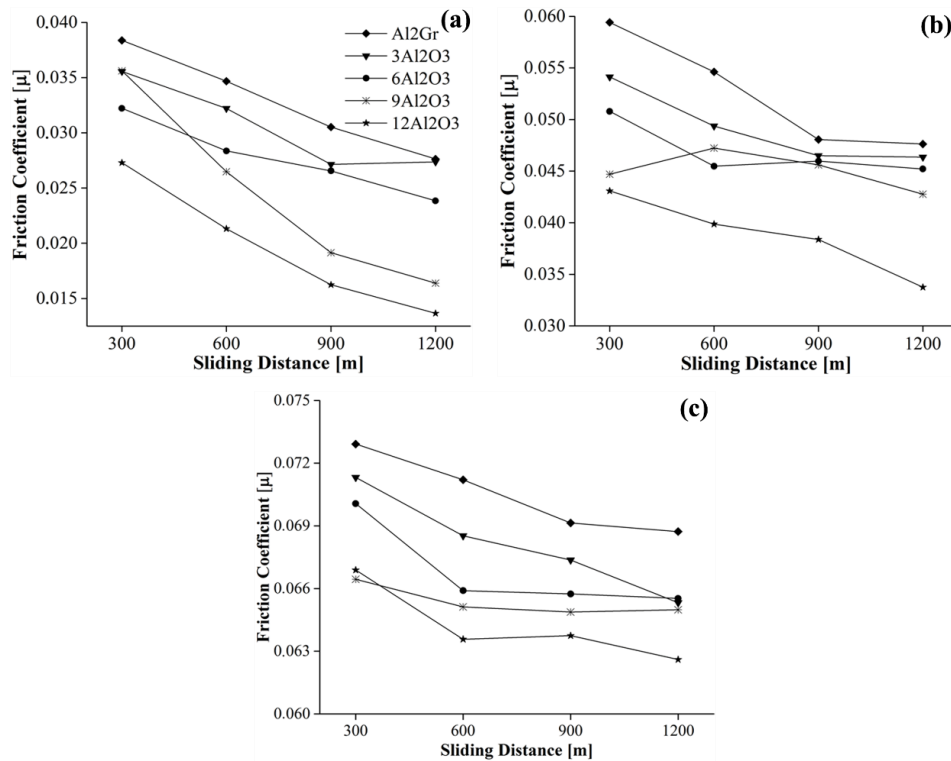


**Figure 3:** Changes in hardness and density of composite materials with different amounts of Al<sub>2</sub>O<sub>3</sub> reinforcement.

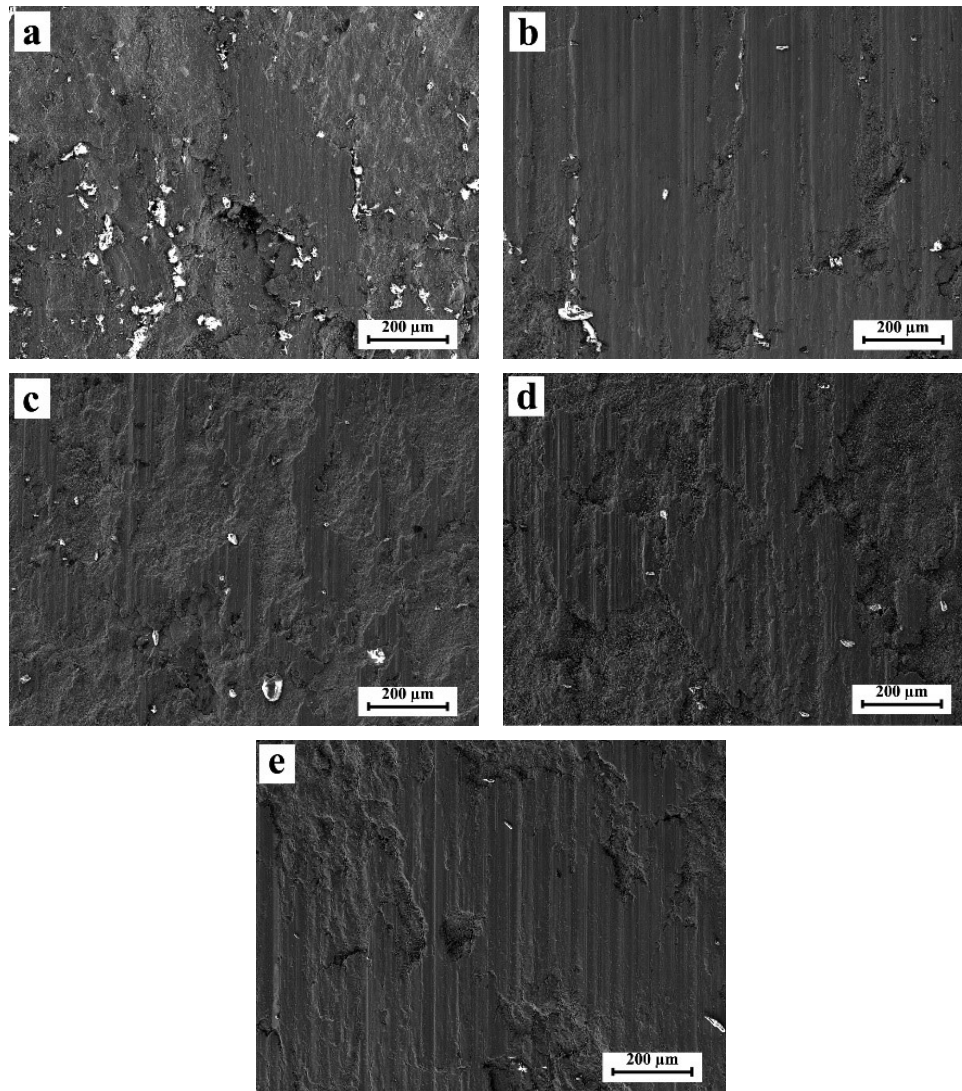




**Figure 4:** Weight losses and wear rates under different loads of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforced; 10 N (a), 20 N (b), and 30 N (c).



**Figure 5:** Friction coefficients under different loads of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforced; 10 N (a), 20 N (b), and 30 N (c).

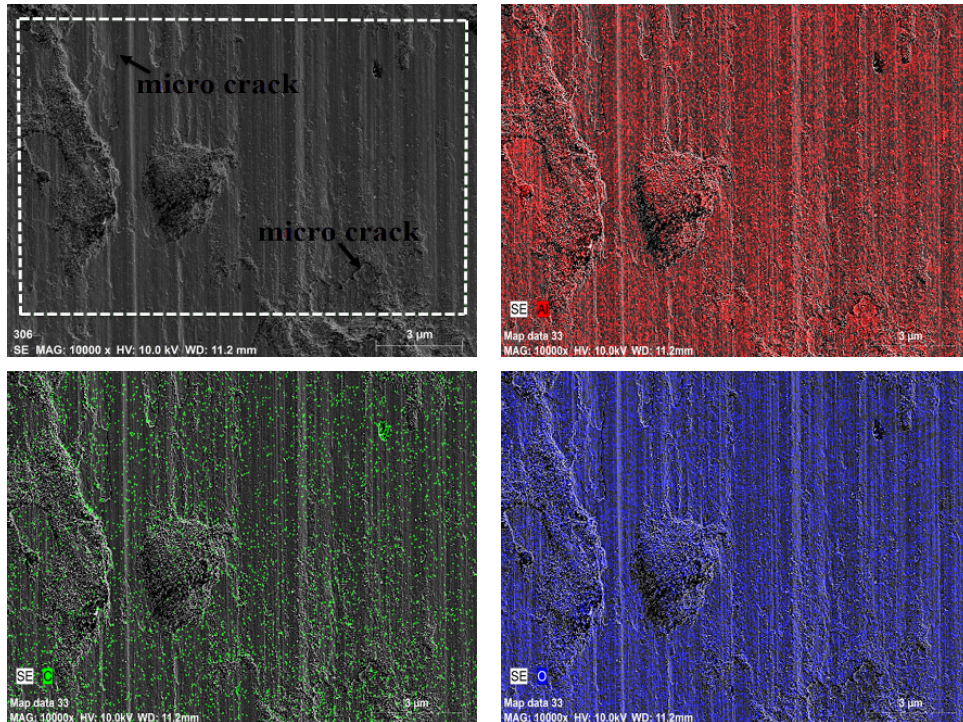


**Figure 6:** SEM images of worn surfaces of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement under 30 N loads; Al+2Gr (a), 3%  $\text{Al}_2\text{O}_3$  (b), 6%  $\text{Al}_2\text{O}_3$  (c), 9%  $\text{Al}_2\text{O}_3$  (d) and 12%  $\text{Al}_2\text{O}_3$  (e).

hardness value (Al+2Gr), and the lowest weight loss is measured for the sample with the highest hardness (12%  $\text{Al}_2\text{O}_3$  reinforced composite material). Wear rate results given in Figure 4 indicate that although there is a fluctuating variation, a general tendency of decrease is observed. The increasing amount of  $\text{Al}_2\text{O}_3$  in the matrix and the sliding distance are a dominant factor in the reduction of wear rate. The decrease in the wear rate due to the increase in the amount of the reinforcement element results from the contribution of the reinforcing element to the strength of the AMCs. Purohit *et al.* [31], stated that wear rate decreases when the ratio of hard reinforcement in aluminium matrix increases. In addition, the presence of 2% graphite in the composition of the composite material produced can be seen as a cause of this reduction in wear rate. Reduc-

tion of wear rates in aluminium hybrid composites containing graphite may be the cause reducing wear by facilitating sliding on the contact surface [32, 33]. Again, fluctuations seen in wear rate results can be explained by the large pieces that broke off the material during the wear process. When the weight loss results obtained under load of 30 N are examined, the sudden increase in weight loss of 3%  $\text{Al}_2\text{O}_3$  reinforced composite material at a sliding distance of 600 m is a clear indication of this situation. These results are supported by the results of some previous studies [6, 34]. Friction coefficients of composite materials with  $\text{Al}_2\text{O}_3$  reinforcement at different amount are given in Figure 5.

When the friction coefficients of different amount  $\text{Al}_2\text{O}_3$  reinforced composite materials given in Figure 5 are



**Figure 7:** Worn surface EDS (Mapping) results under 30 N loads of 12%  $\text{Al}_2\text{O}_3$  reinforced composite materials.

examined, it appears that the friction coefficients are decrease with the increased sliding distance in all loads. Oxide layer formed by increasing slide distance and heat from the ring-sample friction, decreases the friction coefficient. Graphs also indicate a decrease in friction coefficient as the ratio of the reinforcement increases. Ozyurek *et al.* [6], also obtained similar results. Graphite in the AMCs causes the friction coefficient to decrease, too which serves as a solid lubricant. Baradeswaran and Perumal [35] report that graphite in the structure decreases the friction coefficient about 51%. Under 20 N load, the coefficient of friction of the reinforced aluminium composite with 9%  $\text{Al}_2\text{O}_3$  increases at a slip distance of 600 m, while it is seen that the sliding distances are reduced. SEM images of worn surfaces of composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement under 30 N loads are given in Figure 6.

SEM images of worn surfaces given in Figure 6 clearly show the obvious deformation marks on the sample surface. The wear on the sample without  $\text{Al}_2\text{O}_3$  (Figure 6a) is understood be more than the other samples. Oxidation (white areas) on the surface can also be seen clearly on the same sample. In all samples, it is seen that some of the separated particles are re-adhered to the surface and chipping is observed on the surfaces of the composites added with 6% and 9%  $\text{Al}_2\text{O}_3$ . The friction coefficient under the 30 N load given in Figure 5 supports this situation. Pieces that

are broken off the surface increase the surface roughness and cause friction coefficient to increase. Figure 7 gives the EDS (mapping) results of 12%  $\text{Al}_2\text{O}_3$  reinforced composite materials worn under 30 N loads.

The EDS (mapping) of 12%  $\text{Al}_2\text{O}_3$  reinforced aluminium composite given in Figure 7 shows the plastic flow zones, micro cracks and local chipping on the surface after the wear tests. Plastic flow region (smooth region) begins with the first stages of the wear tests and increases with the sliding distance [36]. Micro cracks caused by the stresses developed just beneath the surface during the wear tests. Growing micro cracks by the increasing sliding distance causes detachment from the surface. On the other hand, oxide layer can be clearly seen on the wear surface (due to pin-ring friction). The heat produced between the ring and the sample contact surface causes oxide layer on the aluminium matrix surface. This oxide layer developed on the surface is an important factor affecting tribological parameters such as: weight loss, friction coefficient and wear ratio. Because, this oxide layer on formed on the surface serves as a solid lubricant and protects the surface [37]. Besides the oxide layer on the worn surface, graphite (C) which is homogenously distributed in the matrix forms a thin film layer at the metal-to-metal contact interface and prevents the oxide particles from detaching [37]. Both solid greases (existing graphite and  $\text{Al}_2\text{O}_3$  layer formed during the wear test) significantly increases the wear resis-



tance of the aluminium composite. Ravindran *et al.* [38], and Akhlaghi and Zare-Bidaki [39], reported that graphite added to the matrix up to 5% forms a good solid grease film and improves the wear resistance of the aluminium composites.

## 4 Conclusion

Results obtained from the study that investigates the wear behaviour of mechanically alloyed composite materials with different amounts of  $\text{Al}_2\text{O}_3$  reinforcement to Al-2Graphite matrix are given below:

- It is determined that the  $\text{Al}_2\text{O}_3$  reinforcement to the Al+Graphite matrix in Al-Graphite- $\text{Al}_2\text{O}_3$  composite materials does not display a homogeneous distribution in the structure. It concentrated mainly at the grain boundary.
- XRD analysis results of Al-Graphite-12%  $\text{Al}_2\text{O}_3$  composite materials showed that, Al and Graphite forms  $\text{Al}_4\text{C}_3$  compound in the composition.
- Al-Grafit- $\text{Al}_2\text{O}_3$  AMC's hardness increases by the amount of  $\text{Al}_2\text{O}_3$  in the structure.
- Al-Grafit- $\text{Al}_2\text{O}_3$  AMC's density increases by the amount of  $\text{Al}_2\text{O}_3$  in the structure.
- According to the wear test results, the highest weight loss is obtained from the Al-Graphite sample without reinforcement. It was determined that increasing amount of  $\text{Al}_2\text{O}_3$  reinforcement decreases the weight loss.
- According the wear test results, the highest friction coefficient is obtained from the Al-Graphite sample without reinforcement. Friction coefficient of the composites decreases by the amount of  $\text{Al}_2\text{O}_3$  added to the composites.

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