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Journal of Magnesium and Alloys 7 (2019) 283–290

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Full Length Article

Wear and mechanical characterization of Mg–Gr self-lubricating composite fabricated by mechanical alloying

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Received 7 November 2018; received in revised form 21 March 2019; accepted 1 April 2019
Available online 15 May 2019

Abstract

In this research, the wear and mechanical responses of pure magnesium-graphite (Mg–Gr) composite have been investigated aiming to get the optimum composition of reinforcement. The composite materials were fabricated by mechanical alloying. The percentage of graphite reinforcement was chosen as 3, 5, 7 and 10 wt.% to identify its potential for self-lubricating property under dry sliding conditions. The mechanical properties including hardness, tensile strength and flexural strength of the composites and the base material were tested. The wear tests were conducted by using a pin-on-disc tribometer. The results show that the mechanical properties decrease with increasing graphite content as compared to that of the base material. The wear rate and average coefficient of friction decrease with the addition of graphite and was found to be minimum at 5 wt.% graphite reinforcement. The addition of 5 wt.% graphite in the composite exhibits superior wear properties as compared to that of the matrix material and other compositions of the Mg–Gr composites.

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Keywords: Metal-matrix composites (MMCs); Characterization; Mechanical properties; Wear; Mechanical Alloying.

1. Introduction

Nowadays, the research scenarios are focusing on lightweight materials as weight reduction becomes an important issue for automotive and aerospace industries. In view of that, aluminium and its alloys, high strength steels and polymers were being researched and used significantly in producing lightweight components of automotive and aerospace. Besides, increasing demands on material for achieving superior overall performance has directed to widespread research and development efforts in the composite fields. It is very important to develop lightweight and high-strength materials for improving energy efficiency through the weight reduction of transportation carriers [1]. Therefore, Magnesium and its composites can be a better choice as this is the lightest metal and offer a very high specific strength among conventional engineering alloys. The density of magnesium is 1.74 g/cm^3 which is approximately two-thirds of that of aluminium and over five times lighter than steel [2]. In addition, magnesium alloys possess exceptional dimensional stability, good damping capacity and excellent recyclability which makes them a suitable candidate for lightweight application [3].

Wear is one of the most common industrial problems. The components involved in wear need to be replaced in due time. Therefore, reducing wear is one of the major challenges in the industry and to solve the problem lubricating oil and greases are commonly used in the moving components. In many industrial situations, lubricating oil and greases cannot be used in the moving parts as there is a possibility of contaminating the products or the components are very difficult to lubricate. In such cases, self-lubricating materials can be used as they need limited external lubrication or no lubrication at all [4]. Self- lubricating composite materials composed of metal and graphite particulate in which Gr particles act as the solid lubricant offer many improvements over the materials to which lubricant needs to be applied periodically. Graphite is considered as one of the widely used solid lubricant material and previous studies have shown that the addition of graphite particles to the metallic matrix significantly improves the

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https://doi.org/10.1016/j.jma.2019.04.002

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tribological behavior of the composite [5–8]. In dry sliding condition, the Gr particles form a thin lubrication layer between the contact surface and reduce the coefficient of friction, thus helps to decrease the wear rate [5,9]. The wear resistance in Al-Gr composite improves with an increase in speed as the tribolayer forms in between the worn surface and the disc. However, with the increase in load and sliding distance, the tribolayer reduces; thus, decreases the wear performance of the composite [10,11]. On contrary, researchers observed that the wear resistance in Al359 alloy-Gr composite increased for higher loading, sliding velocities and sliding distance conditions [12]. A study investigated the formation and role of tribolayer on the graphite reinforced composite and compared the behavior with non-graphite reinforced composite. They observed that graphite reinforced composite formed a stable tribolayer on the contact surfaces results in a higher transition from mild-to-severe wear at different loading conditions as compared to non-graphite composite [13]. Due to the presence of graphite particles, a thin graphite-rich tribolayer develops which helps to diminish the shear stress, reduce the plastic deformation in the subsurface area and avoid metal-to-metal contact. Thus, it reduces friction and improves the wear resistance of the composite [13]. Research also carried out on the influence of graphite weight proportion on the metallic matrix and concluded that the composite composed of below 5% Gr particles provides better wear performance [14,15]. These studies explained that beyond 5% of Gr reinforcement, agglomeration occurs which severely degrades the fracture toughness and wear resistance of the composite. Leng et al. [16] observed around 20% reduction of tool wear while using only 4% Gr in SiC/Al composites. Besides, the wear rate of Al-SiC-Gr hybrid composite at different loading condition was compared by Ames et al. [5] and they observed that 3% Gr reinforced hybrid composite displays low wear rate over entire load range. Moreover, the machining behavior of metal-Gr composite was investigated and the results were found to be good due to the fact that graphite is inherently wear resistant [17]. The particle size also plays an important role in determining the wear behavior of the metal-graphite composite. As the particle size of the reinforcement increases, wear resistance also increases due to the enhancement of fracture toughness [18]. Thus optimum particle size and volume fraction of the reinforcement are important in determining the wear behavior of the composite.

Previous studies have reported that the homogeneous dispersion of the reinforcement particles in the metallic matrix improves the mechanical and wear properties of the metal matrix composites [19–21]. However, in manufacturing MMCs, the homogeneous dispersion of the reinforcement particles is a major challenge. In a conventional casting process, the reinforcing ceramic particles are added in molten metal. During solidification, the molten metal shrinks the reinforcing particles close to each other and the cluster of reinforcing particles are formed in different places on the composite surface. Therefore, it is quite impossible to obtain a uniform mixture of metal matrix and non-metal reinforcement particles by traditional casting or stir-casting methods. These particle clustering regions are highly vulnerable to produce crack during conventional loading process [22]. Therefore, composites produced by these conventional process usually show low mechanical and wear properties in service condition [23,24]. To overcome the situation, mechanical alloying is considered to be an effective alternative process. The mechanical alloying which involve mechanical milling, compaction and sintering process is a very advantageous powder processing technique which offers uniform dispersion of the reinforcing particles over matrix material thus ensures excellent mechanical and wear properties.

Table 1
Composition of magnesium and graphite.

Sample	% of Mg	% of Gr
S1	100	0
S2	97	3
S3	95	5
S4	93	7
S5	90	10

The above studies discussed was largely related to the aluminium matrix composite reinforced with graphite particles. The mechanical and wear behavior of graphite reinforced magnesium composite was scarcely investigated. Atthisugan [13] studied the mechanical and wear behavior of stir-cast AZ91D Mg matrix composite reinforced with B4C and Gr. They revealed a low wear loss in the composite as compared to the unreinforced Mg alloy. Moreover, Narayanasamy [25] investigated the influence of Gr and MoS_2 on the mechanical and wear properties of Mg and found MoS_2 reinforced Mg provides better wear behavior as compared to Gr reinforced Mg. These findings indicate that the results found so far on the mechanical and wear behavior on the Mg–Gr composite are not conclusive and further research is needed to investigate the optimum composition of the matrix and reinforcement materials. Therefore, an attempt has been made to investigate the mechanical and wear properties of Mg–Gr composite. The wear rate and friction coefficient under dry sliding conditions have been investigated and the optimum amount of graphite addition in Mg has been evaluated.

2. Materials and methods

The composites were produced using the powder metallurgy route. Pure magnesium (99% purity) was used as the matrix material and graphite powder (99% purity) was added in different weight percentage values as a reinforcement to fabricate the self-lubricating composite. The different compositions of magnesium and graphite are shown in Table 1. The average particle size of magnesium and graphite was 100 µm and 40 µm respectively. Both the powders were mixed according to the designed percentage as mentioned in Table 1 in a planetary ball mill (type RETSCH PM 100). A process control agent, Polyvinyl Alcohol (PVA) was added to the powder mixture to avoid agglomeration of the powder and to prevent the deposition of the powder on the walls of the vial and

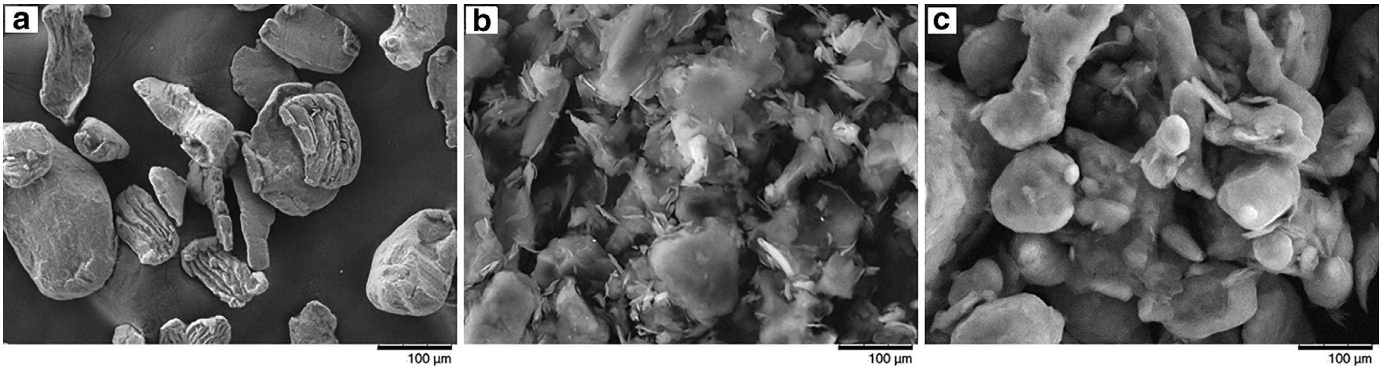

Fig. 1. SEM images of (a) Mg powder, (b) Gr powder and (c) Mg/Gr mixture.

balls. The milling was conducted for 2 h with the rotational speed of 150 rpm to ensure the homogeneity of the mixture. A microstructural analysis of the mixed powders was conducted by using Scanning Electron Microscopy SEM (JOEL model 6390). The SEM images of the as-received magnesium and graphite powder and the mixture of the magnesium-graphite is shown in Fig. 1. The SEM images show that most of the Mg particles were spherical and ellipsoidal shape (Fig. 1a), whereas the Gr particles were in the shape of flakes (Fig. 1b). A uniform distribution of the Gr particles in the Mg matrix was achieved during the milling process as shown in Fig. 1c. An X-ray diffraction (XRD) was carried out to study the phase evaluation of the mixed powders. The XRD was performed using a high-resolution X-ray diffractometer (Shimadzu XRD 6000). The powder samples were cleaned with acetone and dried in air prior to the X-ray diffraction studies. The XRD patterns of the ball-milled powders are shown in Fig. 2. The obtained Bragg angles were matched with standard values for Mg and Gr. The XRD pattern shows high-intensity Mg and carbon peaks indicates the presence of Mg and Gr in the powder mixture (Fig. 2c). After the mixing process, the mixture of the elemental powders was heated in an oven up to 100 °C for 1 h to evaporate the volatile matter in the mixture. The mixture was then compacted in a single-die uniaxial hydraulic press (TOYO: Model TL30, capacity: 300 kN) and a green compacts having a diameter of 30 mm were obtained. The compaction was carried out at room temperature with a nominal force of 250 kN. Zinc stearate was used in the die wall as a lubricant. The green compacts were then sintered in a muffle furnace at 550 °C for 1 h with a continuous supply of argon gas and gradually cooled to room temperature within the furnace. The photographs of the developed Mg–Gr composite samples are shown in Fig. 3. To study the distribution of the reinforcement graphite particles on the Mg matrix, microstructural characterization of the composites was conducted. The ends of the developed composite samples were polished sequentially by using different grades of abrasive papers. The final polishing was performed using 1 µm diamond particles to achieve mirror polish. Microstructural characterization was carried out using a metallurgical microscope (OLYMPUS BX51M, Made in Japan). The density of the pure Mg and the composite samples were measured using Archimedes technique according to ASTM B962-08. The density was recorded using a high precision digital electronic weighing balance with an accuracy of 0.0001 g. All of the samples were tested three times to more precisely determine the density. The micro-hardness of composites was determined using Vickers hardness tester (Wilson Hardness: Model 402 MVD, Made in USA). A normal load of 15 g was applied to all the specimen for 15 s. The test was conducted at the room temperature along the longitudinal axis of the specimens. For each specimen, ten measurements were taken with an interval of 1 mm to avoid any effect by the neighboring indentations and the mean value was taken as the Vickers hardness (VHN) number. The tensile properties of the Mg–Gr composites were evaluated by uniaxial tension tests at room temperature in a universal testing machine (INSTRON 3369) as per the ASTM 08–8 standard. The tests were performed using a 100 kN load cell and 0.016 mm/s displacement rate. Moreover, the flexural strength of the specimens was determined by using the conventional 3-point bending test to find the maximum load withstanding ability of the composites.

The wear properties of the developed composite samples were investigated using a Pin-on-disc tribometer. The tests were performed at room temperature with applied loads of 10, 20 and 30 N and sliding speeds of 0.5, 0.8 and 1 m/s followed by the guidelines of ASTM G99-95 standard. Pins with 50 mm length and 6 mm diameter were used for the wear test. The pin surface was polished and was rotated against an OHNS (Oil Hardened Nickel Steel) disc. The specimen and counter disc were cleaned with ethanol before and after each run to remove trace contaminants. A weight loss method was used to determine the wear rate. The weight of each specimen was measured using an electronic weighing balance with a resolution of ±0.1 mg. Each test was repeated three times and the average of the results was taken. Besides, the morphology of the worn surfaces was analyzed by SEM.

3. Results and discussion

The optical micrographs of the Mg–Gr composite samples with different wt.% of Gr are shown in Fig. 4. These micrographs show the microstructure of the sintered samples. Fig. 4 illustrates that there is no agglomeration of the

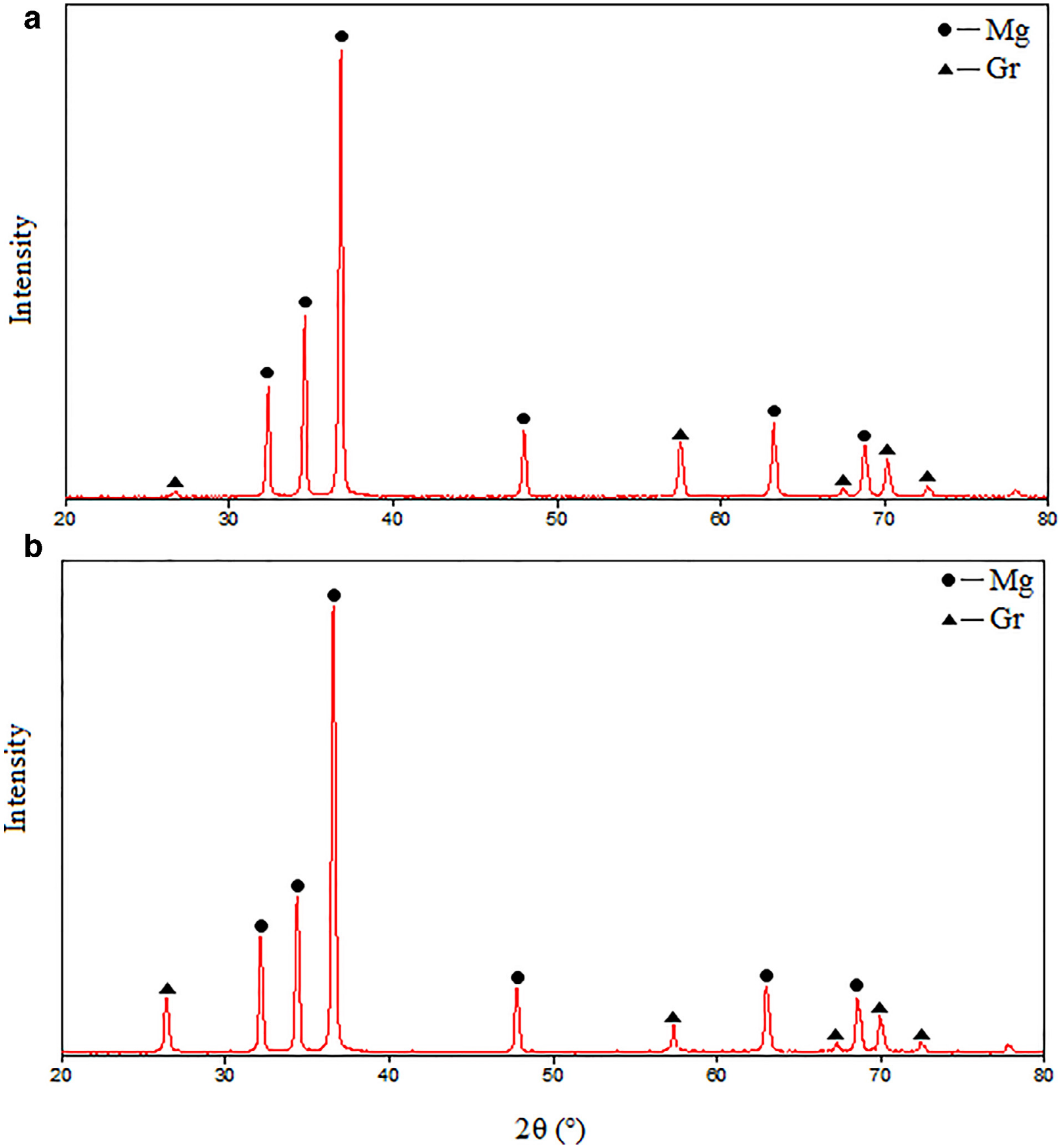

Fig. 2. XRD pattern of powder mixture (a) Mg + 3%Gr, (b) Mg + 10%Gr.

reinforcing particles in the composite samples rather a homogeneous distribution of Gr particles is achieved in all the samples. Besides, no pores and surface cracks were observed on the surface of the samples indicates a strong bonding has been achieved between the Mg matrix and Gr reinforcement during the sintering process. The homogeneous distribution of the reinforcing particles and insignificant surface cracks contributes to improved mechanical and wear properties of the composites. Besides, Fig. 5 represents the density result of pure Mg and the Mg–Gr composite with varying wt.% of Gr. The density of the pure Mg is measured 1.68 g/cm^3, very close to the theoretical density of the Mg (1.73 g/cm^3). It can be noticed that the densities of the composites increased with the addition of the reinforcement Gr particles. In the composite samples, the lowest density, 1.72 g/cm^3 is observed in the sample S2 which is composed of 3% Gr particles and the highest density 1.96 g/cm^3 is observed in the sample S5 which is composed of 10% Gr particles. This shows that the higher the wt.% of the Gr the higher the density of the composite. The graphite particles have a theoretical density of 2.2 g/cm^3, slightly higher as compared to the density of pure Mg. Therefore, once Gr particles are added to the Mg matrix, the overall density increases. The results of the hardness tests of the pure Mg and Mg−Gr composites are presented

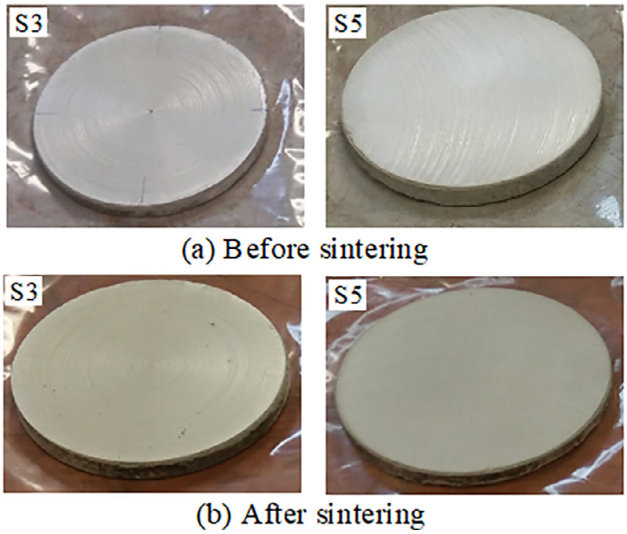

Fig. 3. Green compacts and sintered samples of Mg–Gr composite.

in Fig. 6. It is found that the Vicker's micro-hardness value (VHN) decreases with an increase in the Gr reinforcement in the Mg–Gr composites. The sample S5 (90%Mg + 10%Gr) shows almost 41% reduction of hardness as compared to the sample S1 (Pure Mg). Usually, the Gr particles are soft in nature and bear low hardness value (25.5 VHN) as compared to that of pure Mg which has a hardness of 34 VHN. Therefore, the addition of Gr to the magnesium matrix leads to a reduction in the hardness of the composites. The same tendency of hardness was also observed by Narayanasamy and Selvakumar [25]. The stress-strain diagrams of all the tested materials are shown in Fig. 7. It can be observed in the figure that pure Mg samples possess high yield and tensile strength as compared to that of the composites. The addition of Gr to pure Mg reduces both the yield strength and tensile strength and these values continue to decline as more Gr is added to the composite samples. The tensile strength drops from 121 MPa at 0% addition of Gr particles to a minimum of 93 MPa at 10% addition of Gr particles. This indicates that increasing the Gr reinforcement significantly deteriorates the tensile properties of the composites. This reduction of tensile properties in the Gr rich composites can be attributed to cracking at the particle-matrix interface. Due to the increased percentage of Gr in the composite, the brittleness of the material increases and thus increases the possibility of crack nucleation in the composite which results in low tensile strength. However, the samples with 3% and 5% Gr show reasonably high tensile value as compared to the other two compositions. Moreover, the percentage of elongation of the pure Mg and the Mg–Gr composites are shown in Fig. 8. It can be observed in Fig. 8 that the elongation percentage declines with the increment of Gr particles in the composites. Graphite particles are soft but brittle in nature, thus the addition of Gr particles in the ductile Mg matrix enhances the brittleness of the composite. Thus, the percentage of elongation decreases. Moreover, the flexural strengths of the composites are shown in Fig. 9. The flexural strength of the composites also reduces with increasing graphite percentage. The incorporation of Gr increases the tendency of crack initiation at the reinforcement-matrix interface due to the mismatch of elastic behavior of the Gr reinforcement and Mg matrix, which in turn weakens material properties and reduces strength.

The wear rate of the pure Mg and Mg–Gr composites at different load is presented in Fig. 10. It is observed that the wear rate increases in all the materials with increasing normal load. However, it is seen from the figure that the wear rate of the base material decreases once graphite is added to it and the lowest wear rate was found in the composite composed with 5 wt.% Gr. Once graphite is added to the pure Mg material, it forms a thin lubricating layer between the contact surface which reduces metal to metal contact, results in low wear rate. In addition, the variation of wear rate with different sliding speed is presented in Fig. 11. The wear rate increases in all the materials with increasing sliding speed. The composite materials show a low wear rate as compared to that of pure Mg and in this case also the 5 wt.% Gr reinforced composite exhibits the lowest wear rate. Therefore, it can be concluded that 5 wt.% Gr is the optimum value which provides the best wear performance to the magnesium matrix composite. The variation of friction coefficients of the pure Mg and the composites with varying Gr content are shown in Fig. 12. The composite materials show low coefficient of friction as compared to that of the base material. The lowest average coefficient of friction value under dry sliding con-

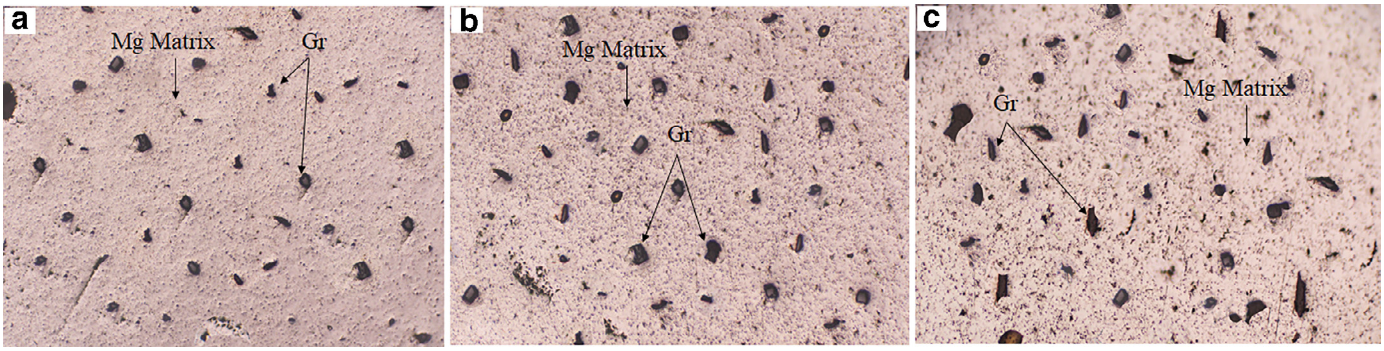

Fig. 4. Optical micrograph of Mg–Gr composite (a) Mg + 5 wt.% Gr, (b) Mg + 7 wt.% Gr and (c) Mg + 10 wt.% Gr.

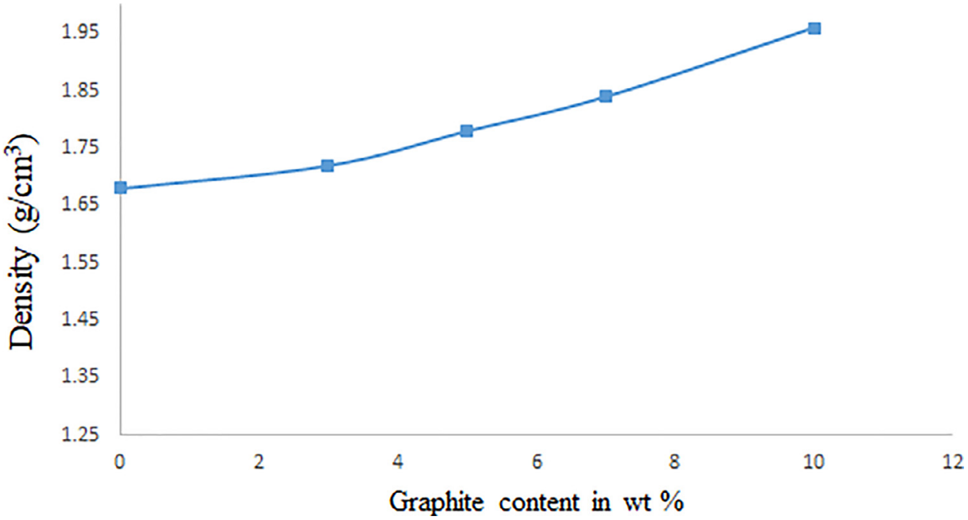

Fig. 5. Density variation of pure Mg and Mg–Gr composite with different wt.% of Gr.

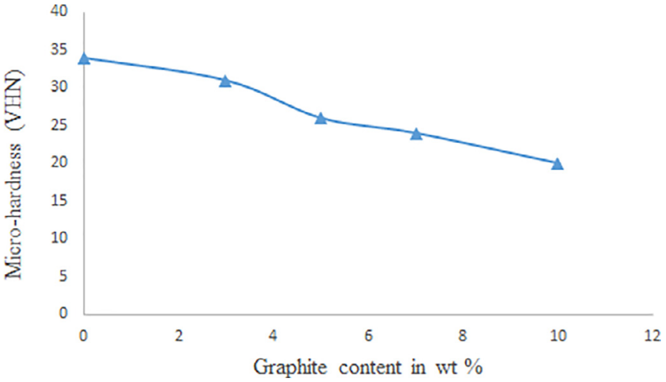

Fig. 6. Micro-hardness of pure Mg and Mg–Gr composite with different wt.% of Gr.

dition is found 0.3 in the composite composed of 5 wt.% Gr. This value increases when the percentage of graphite increases in the composite. The presence of Gr in the metallic matrix creates a smeared Gr film on top of the surface which performs as a solid lubricant and restricts the friction. Once the percentage of Gr increases in the metallic matrix, the film thickness also increases which helps to keep the friction coefficient at a lower level. However, if the tribolayer becomes more graphite-rich, then there is a possibility to form porosity and cracks on the tribosurface which eventually reduces the wear properties and increases the friction coefficient of the composites. The composites with the higher percentage of Gr reinforcement may have experienced a surface deterioration with the formation of porosity and crack, thus increases the wear rate and friction coefficient. To investigate the wear

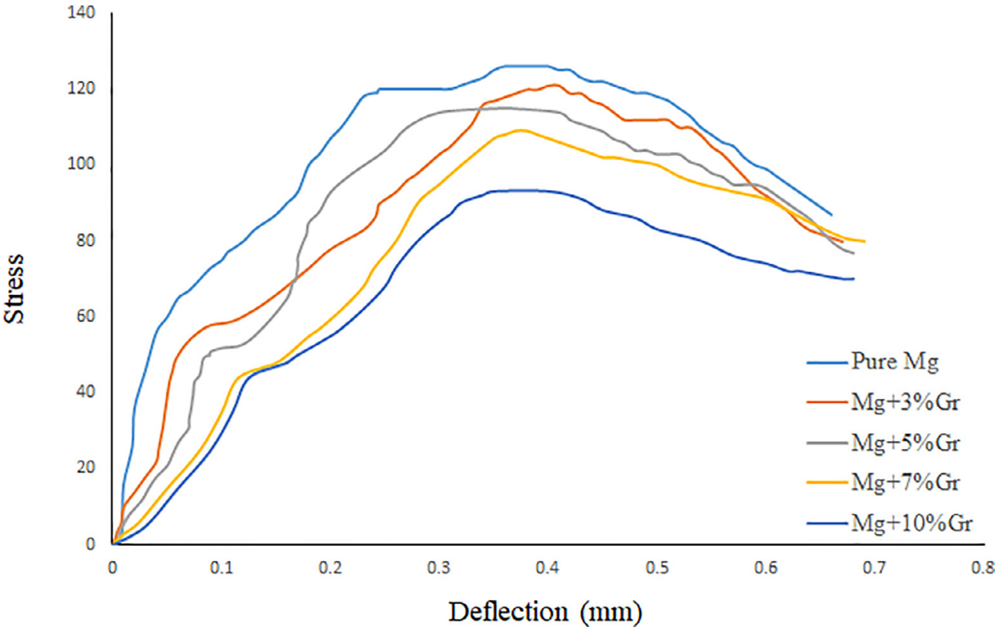

Fig. 7. Stress-strain diagram of pure Mg and Mg–Gr composite samples.

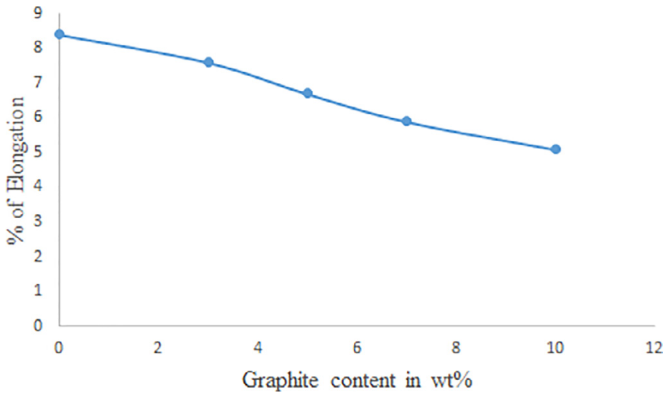

Fig. 8. Percentage of elongation of the Mg–Gr composite samples.

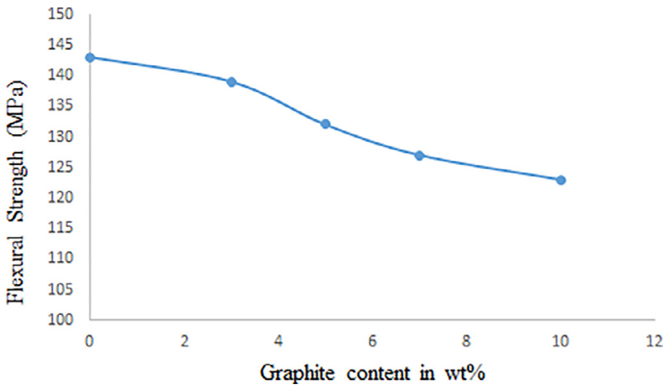

Fig. 9. Flexural strength of pure Mg and Mg–Gr composite with varying wt.% of Gr.

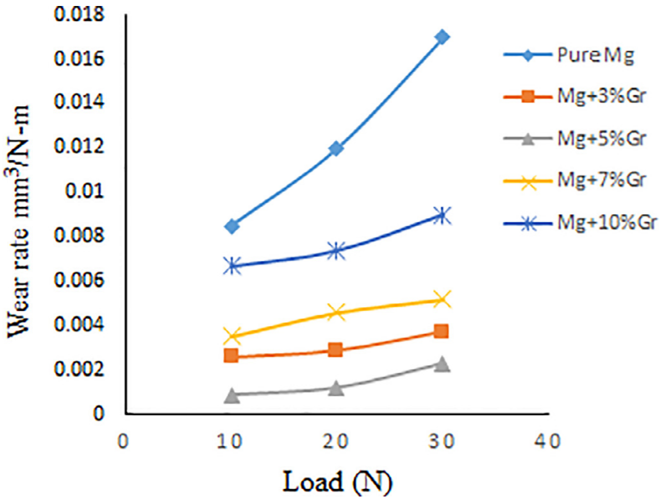

Fig. 10. Variation of wear rate with varying applied load.

mechanism, the worn surfaces of the Mg–Gr composite samples with 5 wt.% Gr and 10 wt.% Gr are characterized by SEM and the results are presented in Fig. 13. The abrasion and delamination wear mechanisms are observed in both the composites. The SEM image shows large grooves along the sliding direction in both the composites indicates plastic deformation which occurs due to the abrasion. Debris is also found in many places indicates the delamination mechanism. Besides, pores are observed in many places in both the composites, however, it is observed that the number of pores in the worn surface of the 10 wt.% Gr reinforced composite are higher (Fig 13d) as compared to that of the 5 wt.% Gr reinforced composite. The existence of a large number of pores in the tribosurface of the 10 wt.% Gr reinforced composite clearly indicates the surface deterioration which occurs due to the presence of a higher percentage of Gr. Therefore, the wear rate and friction coefficient of the composite with higher reinforcement percentage increases. In the worn surface of 5% Gr reinforced composite, a graphite lubricating layer is observed which uniformly covers the entire worn surface of the composite (Fig 13b). This prevents direct contact between the pin and disc and effectively reduce the friction coefficient. Hence, from the above results, it can be concluded that the addition of graphite to the base material significantly improves the wear behavior of the composite and 5% addition of graphite to the Mg matrix exhibits superior wear properties.

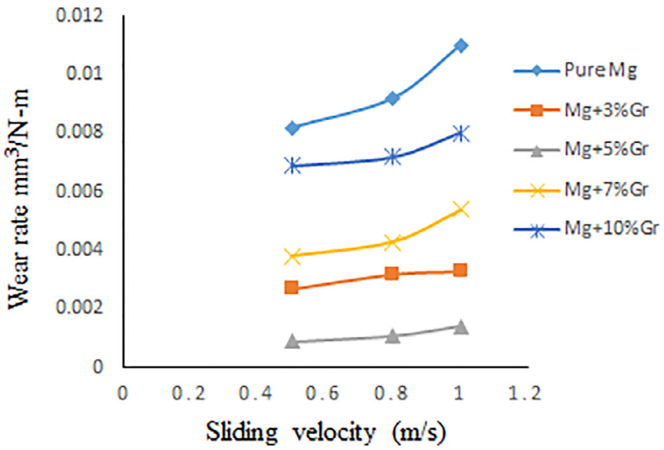

Fig. 11. Variation of wear rate with varying sliding speed.

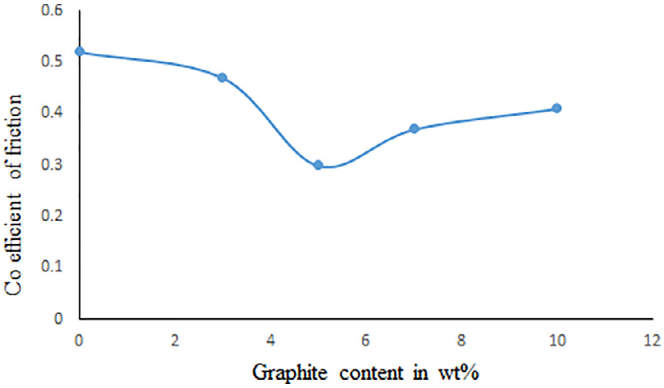

Fig. 12. Coefficient of friction of pure Mg and Mg–Gr composite samples.

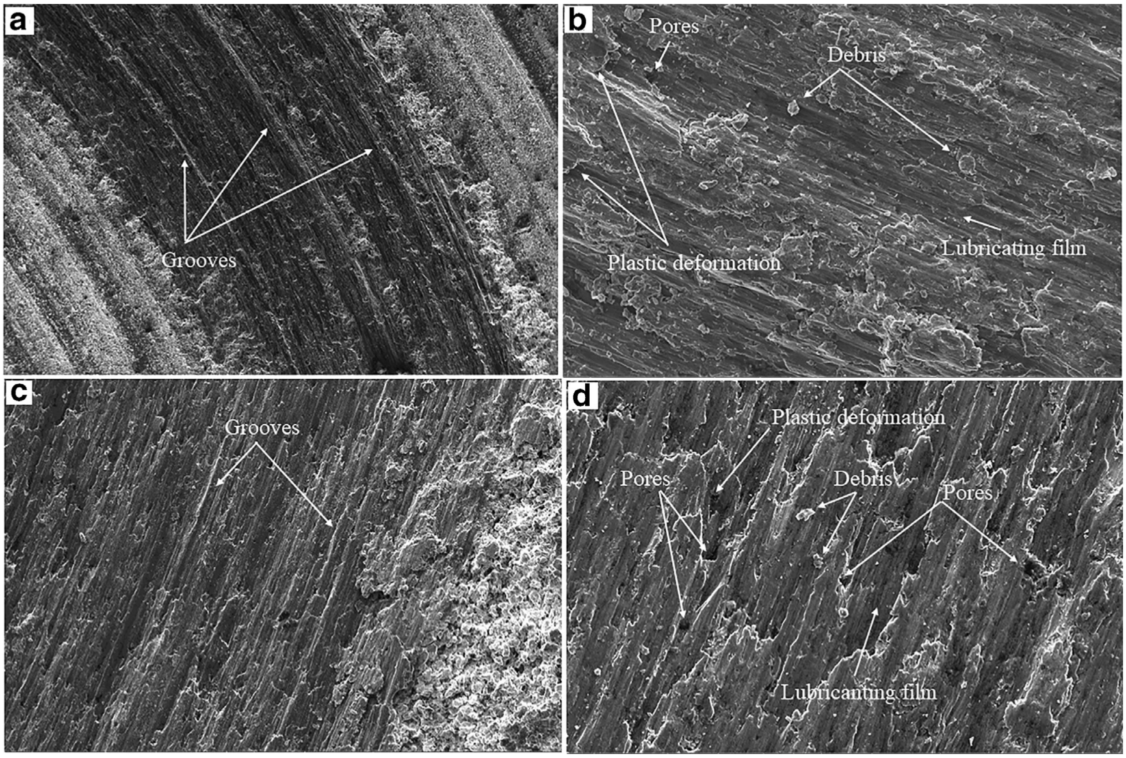

Fig. 13. SEM micrograph of the worn surface of the composite (a) (b) Mg+5%Gr (c) (d) Mg+10%Gr.

4. Conclusions

The effects of graphite (Gr) content on the mechanical and wear behavior of Mg–Gr composite synthesized by mechanical alloying is investigated. It is demonstrated that the increased graphite results in reduced mechanical properties including hardness, tensile strength and flexural strength of the Mg–Gr composite. The Mg–Gr composite as compared to pure Mg possessed lower wear rate and lower friction coefficient and these were minimum at 5 wt.% graphite reinforced composite. However, when the percentage of graphite exceeded 5%, both the wear loss and friction coefficient of Mg-Gr composites increased due to the formation of porosity caused by graphite particles. The SEM micrograph confirmed the presence of a smooth graphite layer on the entire worn surface of 5% graphite reinforced composite and this possessed the superior wear properties than that of other compositions.

Acknowledgement

The authors gratefully acknowledge University Malaysia Pahang (UMP) for providing financial support under project no RDU 160371 during this research work.

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