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Mechanical and wear behaviour of the Al-Mg-nano ZrC composite obtained by means of the powder metallurgy method

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Abstract. This work concentrates on the impact and contribution of zirconium carbide (ZrC) and magnesium to the mechanical and tribological properties of aluminium matrix composites. Distinctive weight portions of zirconium carbide, containing fixed weight fractions of magnesium and strengthening aluminium composites, were prepared utilising the entrenched cold-press sintering technique used in powder metallurgy. The uniform powder mixture was obtained by using planetary ball milling and it was then observed by using the scanning electron microscope technique. The hardness of the hybrid composite increased along with increase in the amount of the ZrC particle. The wear losses of sintered Al-Mg-ZrC composites were explored by directing sliding tests in pin-on-disc equipment. Hybridisation of reinforcements also decreased the wear loss of the composites at high sliding load and speed. This study reveals that the hybrid aluminium composite can be considered a unique material with high strength, low weight and wear resistance that will find their application in components to be used in the automobile and aero space engineering sectors.

Key words: aluminium, magnesium, zirconium carbide, wear and hardness.

1. Introduction

Aluminium matrix composites (AMCs) are a truly fascinating material because they manifest solid consolidation of properties such as, for example, high elastic modulus, wear resistance and tensile strength [1, 2]. Some of the most successful applications of AMCs in the automotive industry include connecting rods, pistons, brake rotors and cast engine blocks [3]. Metal matrix composites are manufactured by means of an extensive variety of strategies, including stir casting [4], plasma spraying [5], hot pressing [6], hot extrusion [7] and powder metallurgy (P/M) [8]. Among these, the powder metallurgy method has its own particular benefits. This strategy shows a tendency to accomplish uniform distribution of strengthening particles in the matrix when utilising the mechanical alloying process. Particle sizes of the strengthening material play a key role in the hardness property of the composite. The specimens prepared by means of the powder metallurgy method are always porous and thus decrease the strength of the sintered samples. However, hard nano reinforcements can be employed to improve mechanical properties and also to fill in the porous regions of composite specimens prepared by means of powder metallurgy [9]. In addition, ceramic particles such as SiC, TiC, WC, B₄C and ZrC are preferentially considered as a secondary strengthening phase in the metal matrix, hybridising the composite.

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This enhances the mechanical properties as well as wear resistance. Incorporation of the ceramic particles as reinforcement during strengthening of the composite may fail to introduce high hardness due to the fact that they will agglomerate by any conventional process. To overcome this, researchers found that ball milling is an efficient way of alloying ceramic particles with the aluminium matrix without any agglomeration [10, 11]. However, an aluminium matrix reinforced with magnesium (Mg) and zirconium carbide (ZrC) for improvement of wear and mechanical properties and prepared using powder metallurgy has not been reported to date. The main objective of this work is thus to evaluate and report on the combined effect of the higher hardness material (nano ZrC: 0-8%) and another material (MWCNTs: 4%) on reinforcement in the aluminium matrix for improvement of various mechanical and wear properties of composites prepared using the powder metallurgy method for tribological application.

2. Experimental procedure

2.1. Materials. Magnesium powder with average particle size of $60~\mu m$ and 400~nm ZrC powders were used as reinforcement materials while pure aluminium powder with average particle size of $60~\mu m$ was selected as the matrix material. Reinforcement materials have a massive impact on the wear as well mechanical behaviour of composites. Micrographs (Fig. 1a–c) of both reinforcements and matrix materials were examined through the SEM. Figure 1a shows that commercially purchased aluminium particles are irregular in shape, and that magnesium

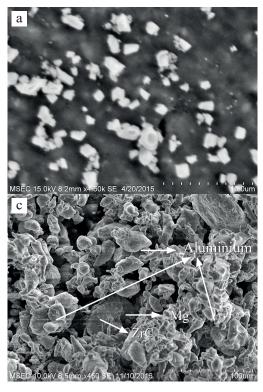
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Fig. 1 SEM micrographs of powders following addition of: a) Al powders, b) Mg, c) ZrC

particles display (Fig. 1b) curved ribbon-like morphology with smaller particle size distribution. The SEM image (Fig. 1c) of ZrC represents an agglomerated sphere structure.

- **2.2. Fabrication of composites and characterisation.** In this experiment, high-energy ball milling and cold compaction, followed by sintering processes, were all used to develop the Al-Mg-ZrC composites.
- **2.2.1. Mixing.** Figure 2a–b display SEM micrographs of ball-milled nano-ZrC particles and the average particle size of the ZrC powder after milling for 25 h oscillates around 400 nm. The most suitable technique for uniform dispersion of secondary particles (Mg and ZrC) throughout the matrix (Al) was attained using the mechanical alloying process [12].

For fabrication of the composites, precise amounts of ZrC particles were measured to attain weight percentages of 2%, 4%, 6% and 8%, respectively, and were then mixed with finegrained Al-4Mg powder in a high-energy planetary ball mill (Fritsch, Germany) containing tungsten carbide balls 10-mm in diameter (with a ball-to-powder weight ratio of 20:1) for 2 h. During ball milling of powders, argon gas and toluene were used as process control agents to prevent oxidation and contamination. The mixed powders were collected from each sample and were characterised for morphology using the scanning electron microscope (SEM). The even distribution of reinforcements is clearly observed in the SEM image. Figure 2a–d shows that the milled nano-ZrC particles appear as agglomerated sphere-like structures. Figure 2b shows the SEM micrographs of Al-4 Mg composite powder. As shown in all micrographs, Mg is evenly



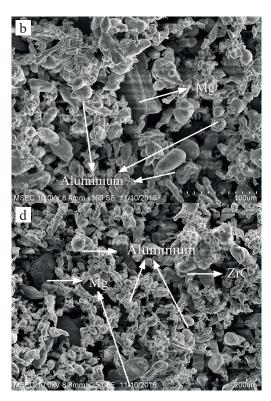


Fig. 2. SEM micrographs of: a) milled ZrC powders, b) Al-4Mg powder, c) Al-4Mg-2%ZrC, d) Al-4Mg-8%ZrC

Table 1
Details of the quantity of powder taken using the rule of mixtures

Material	Total mass (g)	Al (g)	Mg (g)	ZrC (g)
Al	4.2390	4.2390	-	-
Al-4Mg	4.1787	4.0116	0.1671	-
Al-4Mg-2ZrC	4.3053	4.0469	0.1722	0.0861
Al-4Mg-4ZrC	4.4318	4.0773	0.1772	0.1772
Al-4Mg-6ZrC	4.5583	4.1025	0.1823	0.2735
Al-4Mg-8ZrC	4.6849	4.1227	0.1873	0.3748

Table 2
Density values of the sintered powders

Material	Theoretical density	Actual density	Relative density	Porosity
	(g/cm^3)	(g/cm^3)		(%)
Al	2.7000	2.5367	0.9395	6
Al-4Mg	2.6616	2.4739	0.9294	7
Al-4Mg-2ZrC	2.7422	2.5178	0.9181	9
Al-4Mg-4ZrC	2.8228	2.6256	0.9301	7
Al-4Mg-6ZrC	2.9034	2.6819	0.9237	8
Al-4Mg-8ZrC	2.9840	2.7194	0.9113	9

dispersed in the aluminium matrix. The fine distribution of both reinforcements in the aluminium matrix resulted from the choice of appropriate mixing time (2 h) and method of mixing (planetary ball milling), as shown in Fig. 2c and Fig. 2d. It reveals not only the homogenous distribution of Mg in the aluminium matrix but also the distribution of nano-ZrC particles. After the milling process, hard reinforcements were inserted into the soft aluminium matrix as confirmed by SEM analysis.

2.2.2. Compaction and sintering. Next, the mixed composite powders were compacted using a punch-and-die set assembly on an electrically operated compression testing machine (1000 kN). For easy ejection of green compacts, molybdenum disulphide was used as the die-wall and punch lubricant. Cylindrical green compacts of 10-mm diameter and 25-mm length were prepared. The compacted composite specimens were then sintered in an argon atmosphere at 450°C in a muffle furnace for 1 h and allowed to cool to room temperature in the furnace [13]. The sintering temperature of the composite specimens was optimised using the trial-and-error method.

2.3. Density and porosity. The actual sintered density of aluminium hybrid composite specimens was determined using the Archimedes principle, and the results are presented in Table 2. The samples were weighed using an electronic balance (Sartorius, model BS 224S) with an accuracy of 0.1 mg. Theoretical density of the aluminium-based composite specimen was calculated using the rule of mixtures [12]. Details of thus prepared powder mixture of aluminium matrix composites are provided in Table 1. Relative density of composite specimens was cal-

culated from the ratio of actual density to theoretical density. Residual porosities of aluminium-based hybrid composites were at minimum (0–10%) level.

- **2.4. Rockwell hardness.** Hardness values of the aluminium composites were determined using the Rockwell hardness method with a load of 10 kg being applied. The aluminium composite specimens were fabricated with a diameter of 10 mm and height of 10 mm. Prior to testing, each sample was cleaned with acetone and metallographically polished with abrasive paper of grades 600, 800, and 1000 and then with alumina paste as a final step.
- 2.5. Wear testing. The specific wear rate and friction coefficient were calculated from weight loss of the composite specimen and from frictional torque measured by means of a pinon-disc test machine during the experiment, respectively. The experiment was carried out as per the ASTM G99–05 standard [13] by using a pin-on-disc tribometer (Ducom, Bangalore) at room temperature in open air atmosphere. An electronic balance (Shimadzu, model: AX 200) with an accuracy of 0.1 mg was used to evaluate specimen weight before and after each test. The prismatic composite with a 10-mm diameter pin was loaded near the dead-weight loading system. The specimens and the disc were washed in acetone in preparation for wear tests under dry sliding conditions. During this test, diameter of the sliding track on the disc surface (100 mm), rotational speed (500 rpm) and sliding velocity (2.61 m/s) were all maintained as constant.

Specific wear rate
$$(mm^3/N-m) =$$

= Volume loss/(Load × Sliding distance). (1)

In the present work, two sets of experiments were carried out:

- The effect of applied load: five aluminium-based preforms from Table 1 were chosen. The applied load ranged from 5 N to 20 N.
- The effect of sliding distance: the sliding distance ranged from 500 m to 1500 m. The ZrC particle weight fraction varied from 0% to 2% in the hybrid composites containing fixed 2 wt% Mg.

In the present work, two sets of experiments were also carried out:

- The effect of applied load: six aluminium-based preforms from Table 1 were chosen. The applied load ranged from 5 N to 15 N.
- The effect of sliding distance: the sliding distance ranged from 500 m to 2000 m. The ZrC particle weight fraction varied from 2% to 8% in the hybrid composites containing fixed 4 wt% Mg.
- **2.6. Observation of worn surfaces and wear debris.** To explore the wear mechanisms of monolithic aluminium hybrid composites, the worn surface regions of wear samples tested at different loads along with wear debris were observed using the scanning electron microscope (SEM).

3. Results and discussion

3.1. Hardness. Hardness results were collected in five unique areas on every specimen to acquire a normal estimation of hardness on a cleaned surface. Figure 3 demonstrates a correlation of hardness values on the pre-prepared composites with varying weight% of ZrC. The outcomes affirm that Rockwell hardness (R_B) of the preforms increases with an upsurge in weight percentage of ZrC up to 8 wt%. The enrichment of the aluminium hybrid composite hardness is credited to the accompanying reasons: (i) high hardness of ZrC strengthening particles, and (ii) uniform scattering of ZrC. Meanwhile, composite preforms hardness increased following wear testing because of the development of hard asperities (ZrC) on the surface of the samples [12]. The mechanism behind this hardness results is known as dispersion strengthening. The Orowan mechanism (dispersion strengthening) accounts for an essential part composite strengthening, especially when the particle size of the reinforcement is within the nanometre range [14].

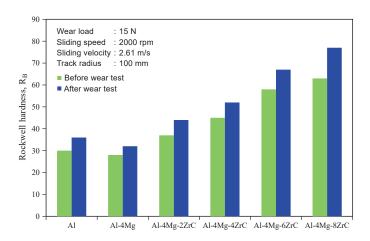


Fig. 3. Hardness values of Al hybrid composites

3.2. Friction coefficient. The friction coefficient is exhibited for hybrid composites Al-4Mg-ZrC using a pin-on-disc apparatus. Non-reinforced aluminium and reinforced hybrid composites are validated for this coefficient. The material is placed on a pin-on-disc and experiments are conducted by means of varying parameters such as load, sliding distance and sliding speed, with sliding velocity of 2.61 m/s remaining constant. Metal on metal contact takes place and contact surfaces are heated at the initial sliding stage. Their temperature is then relatively low in the steady state [15]. Heating reduced in such manner results in reduced friction coefficient values. S.C. Vettivel et al. [7] experimented with composites and found that wear and the friction coefficient have a negligible influence on the type and size of the reinforcement phase. They also reported that the friction coefficient is lower for composites than for the matrix.

The friction coefficient is different for various groups of materials as the load being applied increased within the run-in period during steady state [18], as discussed by M. Lieblich et al.

3.2.1. Effect of friction coefficient on the load. The friction coefficient for hybrid composites Al-4Mg-ZrC is observed under various loads of 5 N, 10 N and 15 N and with increases in sliding distance of 500 m, 1000 m, 1500 m and 2000 m. The track radius stands at 100 mm and sliding velocity is constant at 2.61 m/s. The friction coefficient is reduced with increases in reinforcement of the wt% fraction. In this research, zirconium carbide is the hardest material, used as reinforcement in Al-4Mg matrix composites. The Al-4Mg-2ZrC, Al-4Mg-4ZrC, Al-4Mg-6ZrC and Al-4Mg-8ZrC hybrid composites were all prepared by means of the powder metallurgy technique. Figure 4a–c represents the friction coefficient for different

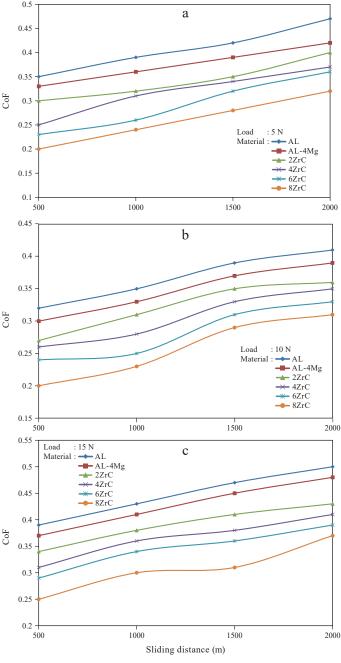


Fig. 4. Variations of the friction coefficient as a function of sliding distance

compositions of zirconium carbide as a function of load at various sliding distances. The experiment is conducted to reduce the friction coefficient under normal load and magnesium from the hybrid composites was pressed out to the contact surface. The friction coefficient is raised under higher loads, with a reduced fraction of zirconium carbide to the Al matrix. The aluminium hybrid composite experienced various friction coefficient values for various wt% fractions. The primary reinforcement magnesium has 4 wt% and secondary reinforcement zirconium carbide presents a different wt%. Magnesium is reinforced to enhance its tribological properties and carbide is reinforced to attain mechanical properties for tribological applications. From the graphs, one can observe that reduction in the load applied and sliding distance for high wt% fractions of carbide material to the matrix results in a lower friction coefficient. The curve in the graph exhibits the friction coefficient value as 0.3 to 0.42, with applied load of 5 N, for various sliding distance from 500 m to 2000 m for the Al matrix. The primary reinforcement, 4 Mg, as added to the Al matrix shows 0.28 to 0.37 for the above-mentioned load and sliding distance. The zirconium carbide reinforced into the aluminium matrix as 2 wt% ZrC, 4 wt% ZrC, 6 wt% ZrC and 8 wt% ZrC exhibits 0.25 to 0.15 at 500 m sliding distance and 5 N load. By increasing the sliding distance to 2000 m, hybrid composite results amount to 0.35 to 0.27. At an applied load of 15 N, the Al matrix has a high FC value of 0.39 at a sliding distance of 500 m, whereas Al-4Mg-8ZrC has a reduced FC value of 0.25. By increasing sliding distance to 2000 m, with applied load of 15 N, the FC is increased for all compositions. Finally, one can deduce from the observations that contribution of primary and secondary reinforcement, i.e. 4 Mg and wt% ZrC, respectively, into the Al matrix provides for a reduced friction coefficient at the applied load of 5 N, and for the FC increasing gradually with increases in the load up to 15 N at sliding velocity of 2.61 m/s.

3.2.2. Effect of friction coefficient of on sliding distance.

During the test, metal to metal contact is carried out, thereby squeezing the hardest zirconium carbide particles out of the aluminium matrix. There might be formation of an adherent film during tribo contact, which exhibits aluminium matrix transformation into harder counter surface due to increased sliding velocity. The friction coefficient is decreased with the help of even distribution of magnesium and nano ZrC without agglomeration. Due to fragmentation of the aluminium matrix, loose wear debris is generated on the counter face, showing an increase in the friction coefficient. To reduce the FC, sliding distance is varied with the track radius and sliding velocity remaining constant.

Figure 4 shows friction coefficient values for the aluminium hybrid composite for various wt% of ZrC with varying sliding distance at different loads (N). Sliding velocity and track radius, at 2.61 m/s and 100 mm, respectively, were kept constant. Initially, the sliding distance is 500 m and the curve in the graph indicates the friction coefficient value of 0.18 for Al-4Mg-8ZrC. Gradual increase in sliding distance from 1000 m to 2000 m results in a higher friction coefficient

being obtained. The experiment is then repeated and various friction coefficient values for various wt% of ZrC are found. From Fig. 4a, it is observed that the friction coefficient is low for sliding distance of 500 m, even following the addition of reinforcement. Figure 4b shows that at a 10 N load, the friction coefficient value (0.32) is low for the Al matrix and it becomes even lower (0.2) following the addition of reinforcement in the form of 8 wt% ZrC. But for sliding distance of 2000 m at the applied load of 10 N and 15 N, Al-4Mg-8ZrC exhibits higher friction coefficient of about 0.31 and 0.37, respectively, as shown in Fig. 4b and Fig. 4c. Finally, when comparing all the compositions, a reduced friction coefficient is found for 4 wt% primary (Mg) and 8 wt% secondary (ZrC) reinforcements.

3.3. Wear loss. The specific wear rate for the nanostructured aluminium matrix and commercial coarse-grained aluminium matrix was studied by Alireza et al. [19], revealing that a lowered wear rate for the nano-grained Al matrix. Researchers have also studied the effect of pure metals and alloys on wear behaviour, and investigated the reduced wear loss/wear rate for finer grain size matrix materials. The wear loss/wear rate oscillates according to its contributing factors such as sintering temperature, applied load and sliding velocity. In general, composite hardness and wear loss/wear rate of the material are inversely proportional to each other. The Al matrix loses its mechanical strength, which leads to severe wear, when heated to sintering temperature. Gradual increase in the load applied resulted in micro cracks generation in the material at the softer surface and in the increase in sliding distance results along with the increase in weight loss (wear). The addition of nano ceramics to the Al matrix results in reduction of weight loss under normal sliding conditions against an EN 31 steel disc. Weight loss of the hybrid composite Al-4Mg-ZrC varied with load applied at various sliding distances with constant sliding velocity and track radius of 2.61 m/s and 100 m, respectively. When the normal load is increased, the pin-and-disc apparatus reduces its contact with each other. It also increases the specimen surface that has ZrC particles added.

3.3.1. Effect of wear loss on load. The aluminium matrix, with its reinforcement zirconium carbide material, shows reduced wear loss as determined from Fig. 5. The primary reinforcement (4 Mg) added to the Al matrix shows slight increase in wear loss of 0.19 g, as shown in Fig. 5a, for the applied load of 5 N at the sliding distance of 500 m. The wear loss is increased to 0.43 g for the Al-4Mg composite at the applied load of 15 N and sliding distance of 2000 m, as shown in Fig. 5c. From Fig. 5a-c one can observe that there is gradual rise in wear loss when the load and sliding distance are increased. The wear loss value rose from 0.19 g to 0.43 g when 2 wt% ZrC with 4 wt% Mg were being added to the Al matrix. When the wt% of ZrC was further increased, Al-4 Mg exhibited the wear loss value of 0.3 g for the applied load of 15 N, whereas the wear loss value was reduced again, to 0.28 g, for a 10 N load applied with the sliding distance of 500 m.

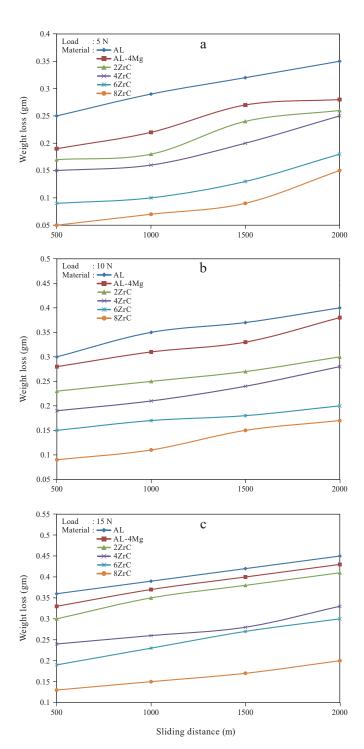


Fig. 5. Variations of wear loss as a function of sliding distance

3.3.2. Effect of wear loss on sliding distance. Figure 5 shows wear loss of hybrid composites prepared by means of the powder metallurgy method as a function of load at various sliding distances of 500 m, 1000 m and 1500 m. The curve from the graph indicates reduced wear loss at sliding distance of 500 m when the load applied is 10 N. Further increase in sliding distance from 1000 m to 1500 m with load at 10 N results in an increase of wear loss from 0.31 g to 0.33 g for Al-4Mg composites. The reduction of wear loss also varies when the

composite is enriched with zirconium carbide at various wt% fractions such as Al-4Mg-2ZrC, Al-4Mg-4ZrC, Al-4Mg-6ZrC and Al-4Mg-8ZrC. Thus, increasing the weight fraction of the hardest material to the matrix helps to generate good wear resistance behaviour, which implies that slight changes in the curve show in the wear loss.

4. Conclusions

This work investigated the wear resistance and mechanical behaviours of aluminium hybrid composites fabricated using the powder metallurgy method. The overall results for P/M-prepared aluminium nano-hybrid composites can be summarised as follows.

- Scanning electron micrographs of the prepared composite powders showed an even distribution of both primary (Mg) and secondary (ZrC) reinforcements in the matrix.
- The hardness and density of the hybrid composites increased with an increased ZrC content.
- The wear study reveals that the prepared composites have exhibited higher wear resistance when compared to the matrix material.
- Among the six different composites, wear resistance was found to be superior for an Al-4 wt% Mg-8 wt% ZrC-reinforced aluminium composite.
- The superior wear resistance of the composite is attributed to the refinement of grain size (reinforcements) and filling of pores, which enhances the bonding strength between the matrix and the reinforcements.
- Increasing the amount of nano-ZrC particles led to a reduced friction coefficient and wear loss.

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