DOI: https://doi.org/10.24425/amm.2025.153505

Y. NARENDRA BABU[1], D. VIJAY PRAVEEN[1*], P. UMAMAHESWARRAO[1],
B. RATNA SUNIL[2], S. KRUGON[1]

ROLE OF REINFORCEMENTS ON MECHANICAL AND WEAR BEHAVIOUR OF AZ91D HYBRID COMPOSITES

Composite materials offer a versatile and customizable solution for various industries, allowing for innovative designs and improved performance. Magnesium-based composites offer a compelling combination of lightweight, high strength, and other desirable properties, making them valuable materials for a variety of applications. The present study is focused on the microstructural, mechanical and wear characterization of the magnesium based hybrid composites which are developed with different combinations of Al_2O_3 and B_4C microparticulates through stir casting route. The microstructural study reveals the diversity in the particle phase distribution. Composite with 2% Al_2O_3 and 6% B_4C have shown higher hardness (78.5 HV0.2 and 67.3 HV0.2, respectively) and yield strength (273 MPa and 219 MPa, respectively) as compared to the other compositions. Furthermore, pin on disc test was conducted on the developed composites to study the wear and friction behavior. Test results revealed that the composite with 2% Al_2O_3 and 6% B_4C has better wear resistance due to its superior mechanical properties as compared to the other developed composites. The results demonstrated the positive role of reinforcements in the AZ91D alloy, which can be a promising material for manufacturing structures and components in the automotive sector.

Keywords: Composite materials; magnesium alloy; automobile applications; hybrid composites; wear properties

1. Introduction

In today's world, there is a burgeoning era of lightweight materials, particularly in industries like automotive where there is a significant demand for energy efficient structures. Historically, aluminum alloys have been at the forefront as the structural candidates, due to the demand for substantial weight reduction in automobiles over the past few years [1-2]. Magnesium (Mg) and its alloys, having a density one quarter that of steels and two thirds that of aluminium, exhibit higher strength-to-weight ratio and surpass both the materials [3]. Furthermore, in the context of cost effectiveness and achievable processing feasibility, Mg alloys have emerged as promising materials for numerous applications such as aviations, sports, electronics goods etc. [4]. However, one of the most significant drawbacks limiting the widespread use of Mg alloys is their poor tribological performance. To address this issue, researchers across the globe have explored various methods to enhance the wear performance of Mg alloys. Several methods such as surface coatings, treatments, microstructural modifications, and development of composites by dispersing hard ceramic particles have been widely adopted in the scientific literature to improve the mechanical and tribological properties of Mg alloys. Improving the wear performance of systems with sliding components is vital for extending their lifespan and enhancing energy efficiency [5-6]. Therefore, in order to benefit from the development of Mg based structures in the aforementioned applications, it is essential to enhance the tribological properties of Mg alloys.

Aatthisugan et al. explored the influence of B_4C particulates on mechanical and wear behaviour of AZ91D composites and demonstrated improved hardness, tensile strength and resistance to wear [7]. Gotagunaki et al. investigated the effect of reinforcement on mechanical and tribological properties of AZ91D composites by reinforcing yttrium oxide and cerium oxide at different weight percentages. Nearly 30% enhancement in the hardness and tensile strength was observed for the hybrid composites dispersed with re oxides compared with the base alloy [8]. Similar studies were carried out by Vijaykumar et al. [9] for the AZ91D composites dispersed with SiC/BN. From the experimental investigations, it was concluded that

1 DEPARTMENT OF MECHANICAL ENGINEERING, BAPATLA ENGINEERING COLLEGE, BAPATLA 522101, ANDHRA PRADESH, INDIA
2 PRINCE MOHAMMAD BIN FAHD UNIVERSITY, DEPARTMENT OF MECHANICAL ENGINEERING, AL-KHOBAR 31952, SAUDI ARABIA

* Corresponding author: vijaypraveend1982@gmail.com

the developed hybrid composites are relatively better for the applications requiring high strength and corrosion resistance due to improved performance. Aatthisugan et al. studied the mechanical and tribological behaviour of AZ91D/B4C/Gr hybrid composites developed by stir casting route. From the studies, they identified that significant improvement in the mechanical properties and resistance to wear [10]. Based on the literature reviewed, it is evident that adding different reinforcements notably enhances the mechanical properties of Mg [11-14]. However, there is limited research on understanding the influence of B_4C and Al_2O_3 effect on the mechanical behaviour of AZ91D alloy. Therefore, this study aims to develop AZ91D hybrid composite using the stir casting method with an objective to investigate the role of added reinforcements on the mechanical behaviour and wear properties of the AZ91D hybrid composites.

2. Materials and methods

2.1. Materials used

AZ91D is one of the most widely utilized Mg alloys due to its excellent mechanical properties, corrosion resistance, and high portability. Hence, AZ91D alloy (Exclusive Magnesium, India) was chosen as matrix material and the chemical composition of is shown in TABLE 1.

TABLE 1

Chemical composition (wt.%) of the AZ91D

Elements	Al	Zn	Mn	Si	Cu	Fe	Mg
Wt.%	8.5	1	0.2	0.1	0.03	0.005	Balance

Aluminum oxide (Al_2O_3) was selected as reinforcement. Al_2O_3 exhibits high hardness, structural stability, considerable wetting property and chemical stability. Al_2O_3 particles of size ~20 µm with 99% purity were used as reinforcements in the development of composites. Boron carbide (B_4C) particulates are highly valued for their hardness, strength, lightweight properties, and resistance to wear and corrosion. These qualities make them valuable reinforcements in composite materials, suitable for a wide range of industrial and structural applications. B_4C particulates of 15 µm average size with 99% purity was used in the present work. Al_2O_3 and B_4C reinforcements were procured from M/s Vision castings and Alloys, Hyderabad, India.

2.2. Development of composites

Composites were produced by semi solid stir casting methodology, as it combine the benefits of semi-solid metal processing with the versatility of stir casting to produce advanced composite materials with tailored properties [15]. Initially, the vacuum pressure was reduced to below 150 Pa. Subsequently, the AZ91D alloy was melted at 710°C in a graphite crucible in a stirred vacuum furnace under a protective atmosphere (Ar). Then the molten alloy was cooled at 580°C to acquire the semi-solid state. Further, the preheated reinforcements were introduced into the molten melt according to the considered compositions as shown in TABLE 2. Subsequently, stirring of the molten metal was carried out for 8 minutes at a speed of 450 rpm using a three blade mechanical stirrer. Similar procedure was followed for the remaining weight percentages of the composites. The content of Al_2O_3 was selected as fixed (2%) and the content of B_4C was varied in the developed hybrid composites.

TABLE 2

Compositions of Hybrid Magnesium Composites

S. No	**AZ91D**	**Al_2O_3**	**B_4C**	**Designation**
1	100	0	0	M0
2	98	2	0	M1
3	96	2	2	M2
4	94	2	4	M3
5	92	2	6	M4
6	90	2	8	M5

2.3. Characterization

Microstructural characterization was carried out for the base alloy. The samples were polished up to mirror like surface finish by following standard polishing techniques. Particle distribution and matrix/ particle interfaces were examined by using scanning electron microscope (FEI Quanta FEG 200F, Netherlands) coupled with energy dispersive spectroscopy (EDS). Phases analysis of the materials was done by using an X-ray diffractometer (XRD, Bruker D8, Germany) with a step size of 0.02 degree/s between a scanning range of 20-90 degrees.

2.4. Density and porosity measurement

The density of a composite is a critical physical property used for its characterization, representing the compactness of the material. It quantifies the distribution of matrix and reinforcement, typically described by volume or weight fractions within the composite structure. The densities of the composite and reinforcements are determined using the Archimedes principle. This involves measuring the sample's mass in both air and in a standard fluid of known density. In the present work, the sample's volume was assessed by measuring the displacement of distilled water in by immersing in a 50 ml measuring jar. The density is then determined by dividing the mass per unit volume of the sample. The density of the developed magnesium composites was determined based on the average of three samples.

Due to the presence of pores and voids, there is typically a discrepancy between the measured density and the theoretical density values in the composites. These differences significantly impact the mechanical and wear properties of the composites. Therefore, an examination of porosity is essential in the qualitative analysis of the composites. The percentage porosity is measured using the following Eq. (1).

$$\%\,porosity = \left[1 - \frac{measured\ density}{theoretical\ density}\right] \times 100 \quad (1)$$

2.5. Microhardness measurement

Hardness indicates a material's resistance to local plastic deformation from mechanical indentation and is influenced by binder material, particle size, and porosity. It can also refer to resistance against abrasion, bending, cutting, and scratching, affecting the material's machinability. Test specimens are cleaned and polished for accurate measurement. The test sample's surface is smoothed with emery papers to allow a regular indentation shape and ensure the sample is held perpendicular to the indenter. Microhardness tests are conducted on all composites in accordance with ASTM E384 standards.

2.6. Tensile testing

The tensile test is a crucial mechanical test where a sample is subjected to controlled tension until failure. Composite specimens, prepared according to ASTM E8/E8M standards, have been undergone tensile testing at standard room temperature with a strain rate of 0.01/s (Zwick-Roell, Germany). Each composition is tested three times, and the average values are used to study the tensile strength of the prepared composites. The results from the tensile test are crucial for designing machine elements and understanding the material's behavior under different loads.

2.7. Wear testing

In most wear characterization studies of composites, dry friction was analyzed to assess the impact of wear parameters. In the present work, wear tests were conducted on pin-on-disc tribometer (DUCOM TR-20 LE, Bangalore). The specimens, each measuring 10 mm in diameter and 25 mm in length, were polished sequentially with emery paper of grades 600, 800, and 1000. Prior to testing, the surfaces of the samples were cleaned using a soft paper soaked in acetone. Experiments are conducted according to ASTM G-99 standards at room temperature in the laboratory. From the thorough literature it is noticed that load and sliding distances are found to be significant parameters. Hence, in the present study, all the tests were performed at various loads (5 N, 10 N, 15 N, 20 N, 25 N and 30 N) and sliding distances 1200 m and sliding speed of 1 m /s to study the wear and friction behavior.

3. Results and discussion

3.1. Microstructural characterization

The characterization of AZ91 Mg alloy was done by scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS) analysis. Fig. 1 presents the typical SEM image and EDS analysis of the base alloy. Since, AZ91D Mg alloy contains higher Al content which is not dissolved in Mg lattice at the room temperature, higher Al content (more than 1%) eventually forms another intermetallic β – phase ($Mg_{17}Al_{12}$) as indicated in Fig. 1(a).

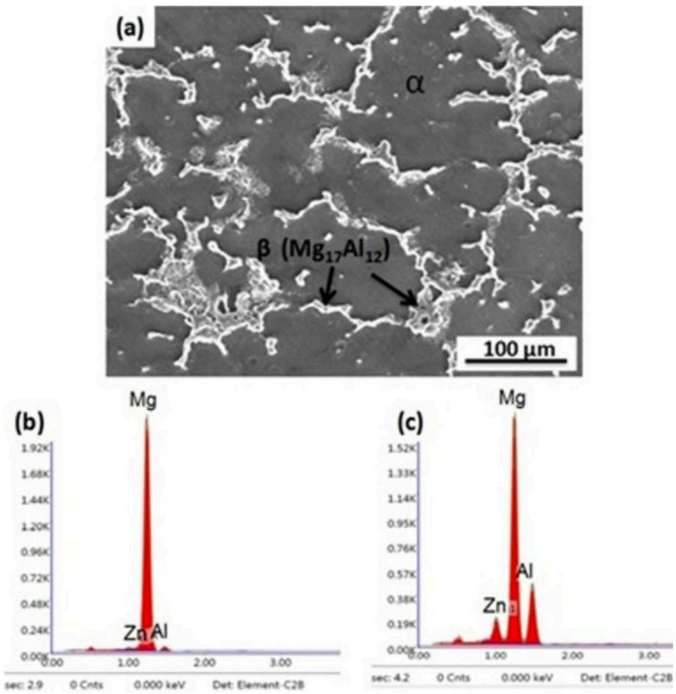

Fig. 1. (a) SEM of AZ91D Mg alloy, (b) EDS at α – phase and (c) EDS at β – phase

The solid solution grains (α – phase) of Mg and Al contain lower amounts of Al (less than 1%) at the room temperature. Therefore, AZ91D Mg alloy usually exhibit the presence of both the phases, α and β as represented in Fig. 1(a). The corresponding EDS analysis was carried out in the solid solution grains and at the grain boundaries clearly differentiate the chemical compositions. The elemental composition of solid solution grains (α – phase) indicates lower amount of the alloying elements (Fig. 1(b)). On the other hand, the EDS analysis was carried out at the grain boundaries (Fig. 1(c)), confirms the higher amounts of Al and Zn compared with the grain interior.

Fig. 2 and Fig. 3 shows the XRD of the AZ91D magnesium alloy, 2 wt.% of Al_2O_3 reinforced AZ91D magnesium composite and 2 wt.% of Al_2O_3 + 2 wt.% of B_4C reinforced AZ91D magnesium composite. The results show the different phases of composed alloys of α – Mg, $Mg_{17}Al_{12}$ along with the reinforcements. All the peaks were identified and indexed. The presence of α phase along with significant peaks corresponding

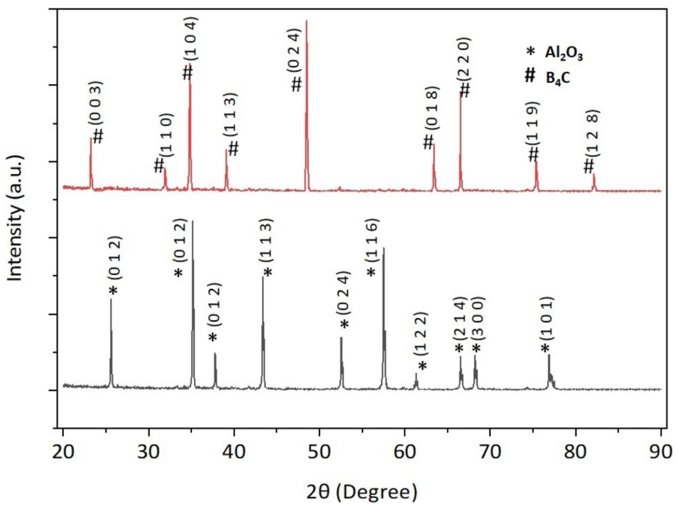

Fig. 2. XRD of the reinforcements

to β-phase can be seen from the XRD data. No other peaks were identified which confirms that the alloy and the composites are free from any undesired phases. The XRD data of AZ91D Mg alloy supports the microstructural observations carried out by using SEM and EDS analysis.

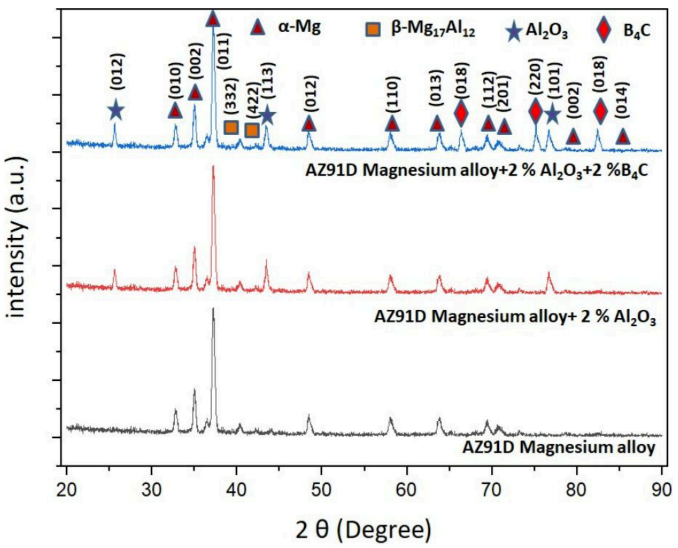

Fig. 3. XRD of AZ91D magnesium alloy and composites

3.2. Density and porosity

The densities of the developed composites measured from the experimental observations are presented in the TABLE 3. From the results, density of the composites was observed to be increased with the increase in the weight percentage of the reinforcements in the developed composites. It is also noticed that porosity of the composites is decreased with the increase of the reinforcements. Incorporating secondary phase particulates in a semi-solid state and controlling the stirring process ensured uniform dispersion of the particulates in the molten melt. This resulted due to the increased viscosity and reduced fluidity of the melt. Moreover, the entrapment of gas bubbles in the molten melt led to increased porosity, particularly noticeable for the composition M5 [16].

TABLE 3

Density and percentage porosity of the composites

S. No.	Designation	Theoretical Density	Experimental Density	% Porosity
1	M0	1.8100	1.7544	3.07
2	M1	1.8536	1.8006	2.86
3	M2	1.8678	1.8181	2.66
4	M3	1.882	1.8325	2.63
5	M4	1.8962	1.8499	2.44
6	M5	1.9104	1.8591	2.68

3.3. Assessment of microhardness

The test specimens were polished on both sides using emery papers to ensure compatibility with the testing equipment. The average of three measurements was recorded, and the test results for the composites are presented in Fig. 4.

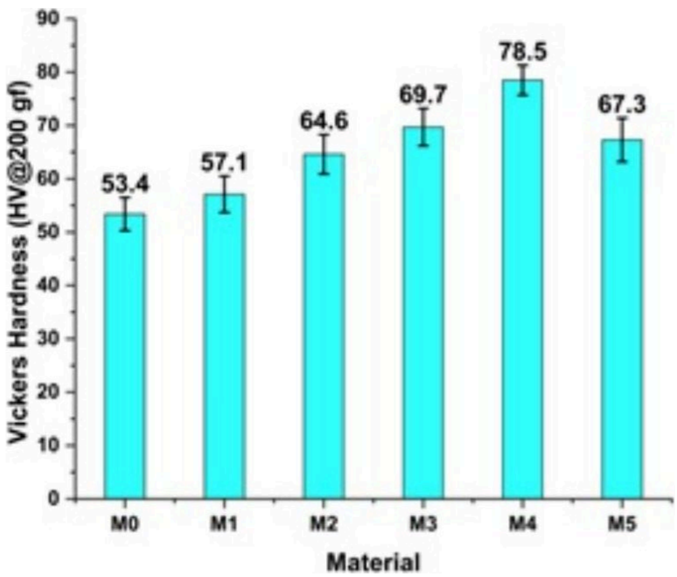

Fig. 4. Microhardness Vs AZ91D composites

The findings indicate a notable enhancement in microhardness (78.5 HV0.2) of the developed composites up to M4 composition. This increased hardness could be due to the presence of the Al_2O_3 as well as B_4C reinforcements restricted the localized matrix deformation indentation and also the load bearing capacity of the composite. Further, microhardness was measured as decreased for M5 sample (67.3 HV0.2). The reduction in the microhardness is due to the prevailing weak bonding between reinforcement as the agglomeration is increased with the higher amount of reinforcement. This further reduces the effective load transfer through the matrix leading to lower measured hardness [17].

3.4. Evaluation of tensile properties

Tensile test measures the material ability to withstand the load before material failure under uniaxial tensile force. Usually incorporation of the hard particulates increases the tensile strength due to dispersion strengthening mechanism. Under an external tensile load, materials can develop significant internal stress, leading to localized damage if the local stress exceeds the material strength. The variation of the ultimate tensile strength vs material is presented in the Fig. 5. In the present work, the results revealed that incorporation of secondary phase particulates into the base material has increased the ultimate tensile strength up to material M4 (273 MPa). This could be due to homogeneous dispersion of the introduced particulates into the AZ91D magnesium alloy, which makes delay in the formation of the local damage. Hence, higher tensile strength is observed at material M4. Further, decrease in the tensile strength was noticed for M5 sample (219 MPa). When the weight percentage of reinforcement in magnesium alloy with Al_2O_3/B_4C particles reaches a certain level, it induces the formation of transition layers at the interfaces of the components. Although these layers facilitate bonding between the particulates and the magnesium alloy, they lack sufficient toughness to bear loads effectively, often resulting in decreased tensile properties of the composites [18].

3.5. Effect of reinforcement on wear and friction behavior

The wear rate of the composites is estimated by computing the mass loss of the specimen before testing and after testing. Specimens were cleaned after each experiment to avoid entrapment of the wear debris. Initially wear test was performed on the developed composite specimens to study the influence of the reinforcement on wear and the results are shown in Fig. 6. The results demonstrated the wear resistance of the composites is increased with the increase of the weight percentage of the reinforcements. It is also observed that wear resistance is higher at the compositions M4 when compared with the base alloy. This behavior could be due to increased hardness due to dispersed reinforcement, increased ability to bear applied loads [19]. Further addition of reinforcement slightly decreased the wear resistance due to weak bonding between particulates and matrix material. In addition, the increased content of the hard particulates acts as abrasives themselves which led to decrease in the wear resistance i.e. at M5 composition by promoting abrasive wear. When the reinforcing particles were pulled out from the matrix, in addition to the wear debris, these reinforcements from the matrix also act as abrasive particles and increases the wear rate as observed for M5 sample. Variation of the coefficient of friction (COF) and testing conditions are illustrated in the Fig. 7. From the plots, it is observed that with the increase in the weight percentage of reinforcements, (COF) is decreased. As the weight percentage of the reinforcement increased, the surface roughness of the composite typically decreased.

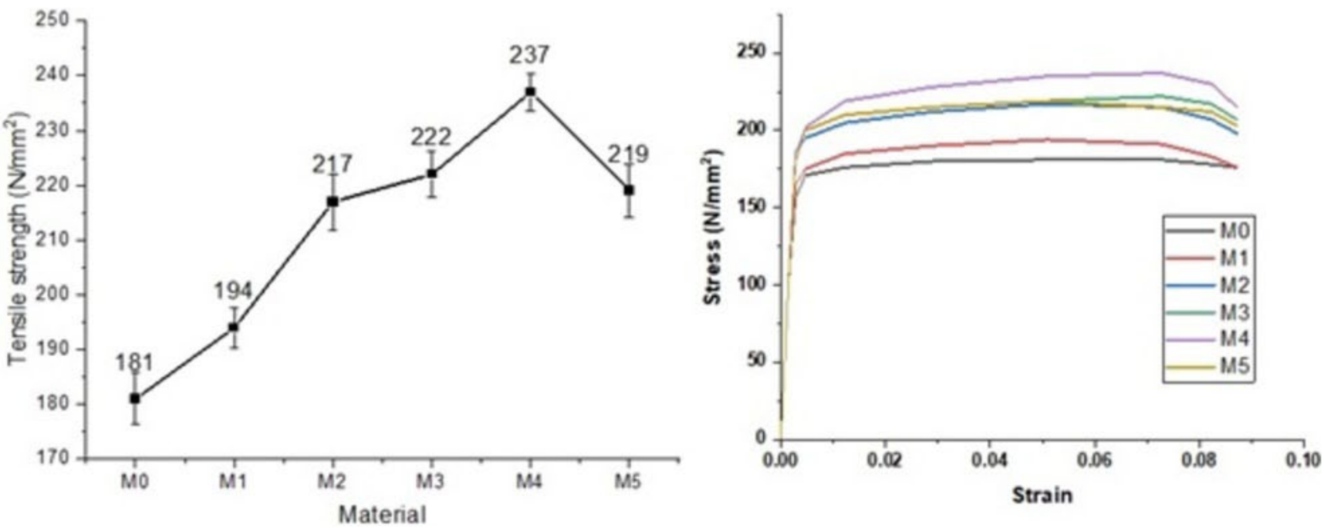

Fig. 5. Tensile test observations

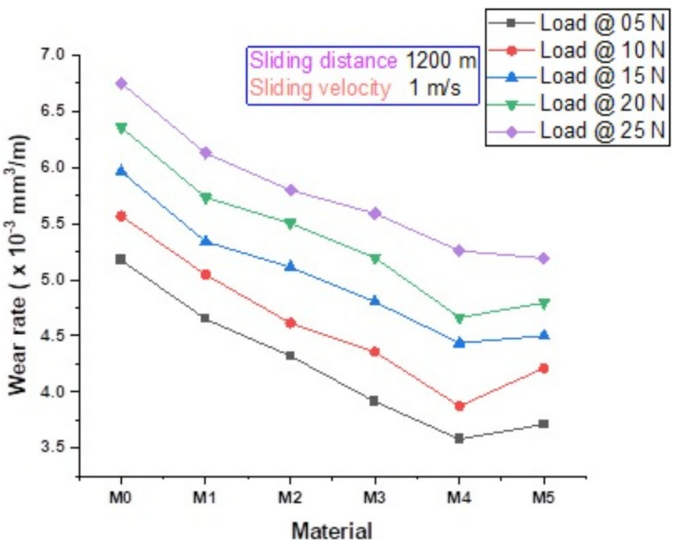

Fig. 6. Variation of wear rate vs AZ91D composites

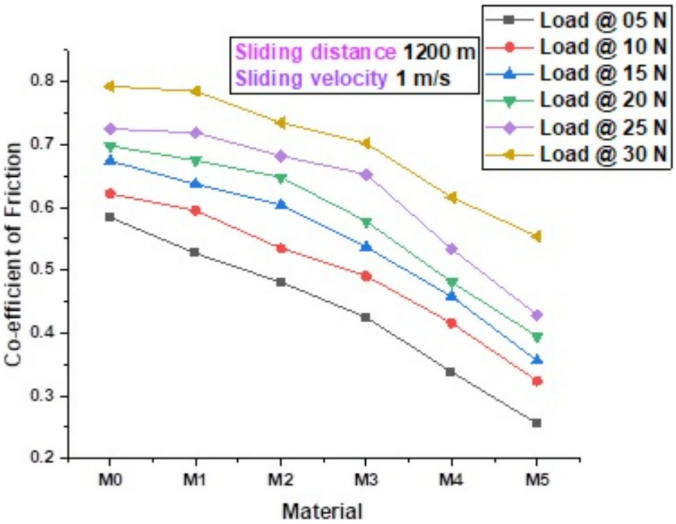

Fig. 7. Variation of COF vs AZ91D composites

In general, smoother surface exhibits lower COF because there are fewer asperities and contact points to resist the relative motion. In addition to this, the introduced particulates, both Al_2O_3 and B_4C particulates minimize the surface contact and also facilitating the smoother surface contact due to their hardness and wear characteristics. Hence, the developed composites offered superior wear and frictional resistance compared to base alloys because of dispersoids [20-21]. These dispersoids restrict the plastic deformation of the matrix alloy and improve its high-temperature strength. By shielding the matrix from contact surfaces, the introduced hard particulates decrease wear rates and lower the coefficient of friction.

For the developed hybrid composites, the pin-on-disc test revealed several wear mechanisms based on load and speed. At lower loads, oxidative wear became evident, marked by a smooth oxide layer forming on the worn surface. However, as the load increased, deep grooves were observed due to abrasive wear resulting from the exposure of the hard reinforcement particles at the surface. Additionally, adhesive wear was observed at high temperatures, indicated by material transfer patches between the composite and the disc counter face.

4. Conclusion

The current research work explores the characterization of AZ91D magnesium alloy reinforced with B_4C and Al_2O_3 with different weight percentages developed through semi stir casting technique. From the experimental investigation the following conclusions are drawn.

- XRD analysis confirms the phases used in the present work and the produced composites are free from undesired phases.
- Higher hardness and tensile strength are identified at 2 wt.% of Al_2O_3 and 6 wt.% of B_4C reinforced AZ91D magnesium composite due to strengthening effect produced by uniformly dispersed particulates.
- Better resistance to wear observed due to increased bond between particulate and the matrix at 2 wt.% of Al_2O_3 and 6 wt.% of B_4C reinforced AZ91D composite.
- Coefficient of friction is decreased with the increase in the weight percentages of the reinforcement in to AZ91D magnesium alloy due to smoother surface contact provided by the secondary particulates.

Hence, the present work demonstrate the potential of incorporating Al_2O_3 and B_4C with appropriate fractions with improved mechanical and wear properties for structural applications.

REFERENCES

[1] A. Das, S.P. Harimkar, Effect of graphene nanoplate and silicon carbide nanoparticle reinforcement on mechanical and tribological properties of spark plasma sintered magnesium matrix composites. Journal of Materials Science and Technology **30** (11), 1059-1070 (2014).

[2] J.B. Ferguson, F. Sheykh-Jaberi, C.S. Kim, P.K. Rohatgi, K. Cho, On the strength and strain to failure in particle-reinforced magnesium metal-matrix nanocomposites (Mg MMNCs). Materials Science and Engineering: A **58**, 193-204 (2012).

[3] P. Umamaheswarrao, B.R. Sankar, M. Pardhasaradhi, K. Rajasekharababu, Experimental study on drilling force during drilling of AZ91D magnesium alloy. Materials Today: Proceedings **44**, 305-308 (2021).

[4] S.K. Baral, M.M. Thawre, B.R. Sunil, R. Dumpala, A review on developing high-performance ZE41 magnesium alloy by using bulk deformation and surface modification methods. Journal of Magnesium and Alloys **11** (3), 776-800 (2023).

[5] K. Xiu, H.Y. Wang, H.L. Sui, Y. Wang, C.L. Xu, J.G. Wang, Q.C. Jiang, The sliding wear behavior of TiC p/AZ91 magnesium matrix composites. Journal of Materials Science **41**, 7052-7058 (2006).

[6] P. Umamaheswarrao, B.R. Sankar, T. Nancharaiah, Experimental investigations on machining force in turning of AZ91D magnesium alloy using PCBN tools. Materials Today: Proceedings **39**, 1206-1209 (2021).

[7] I. Aatthisugan, R. Murugesan, T. Rao, Influence of boron carbide content on dry sliding wear performances of AZ91D magnesium alloy. Proceedings of the Institution of Mechanical Engineers, Part J: Journal of Engineering Tribology **237** (4), 746-756 (2023).

[8] S. Gotagunaki, V.S. Mudakappanavar, R. Suresh, C.D. Prasad, Studies on the mechanical properties and wear behavior of an AZ91D magnesium metal matrix composite utilizing the stir casting method. Metallography, Microstructure, and Analysis **12** (6), 986-998 (2023).

[9] P. Vijayakumar, K. Pazhanivel, N. Ramadoss, A. Ganeshkumar, K. Muruganantham, M. Arivanandhan, Synthesis and characterization of AZ91D/SiC/BN hybrid magnesium metal matrix composites. Silicon **14** (16), 10861-10871 (2022).

[10] I. Aatthisugan, A.R. Rose, D.S. Jebadurai, Mechanical and wear behaviour of AZ91D magnesium matrix hybrid composite rein-

forced with boron carbide and graphite. Journal of Magnesium and Alloys **5** (1), 20-25 (2017).

[11] P. Bharathi, T. Sampath Kumar, Latest research and developments of ceramic reinforced magnesium matrix composites – A comprehensive review. Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering **237** (3), 1014-1035 (2023).

[12] S.K. Khatkar, Hybrid magnesium matrix composites: A review of reinforcement philosophies, mechanical and tribological characteristics. Reviews on Advanced Materials Science **62** (1), 20220294 (2023).

[13] S.S. Kumar, V. Mohanavel, An overview assessment on magnesium metal matrix composites. Materials Today: Proceedings **59**, 1357-1361 (2022).

[14] A.K. Matta, N.S. Koka, S.K. Devarakonda, Recent studies on particulate reinforced AZ91 magnesium composites fabricated by stir casting – a review. Journal of Mechanical and Energy Engineering **4** (2), 115-126 (2020).

[15] C.D. Yim, K.S. Shin, Semi-solid processing of magnesium alloys. Materials Transactions **44** (4), 558-561 (2003).

[16] J. Hashim, L. Looney, M.S.J. Hashmi, Metal matrix composites: production by the stir casting method. Journal of Materials Processing Technology **92**, 1-7 (1999).

[17] D. Kumar, L. Thakur, Investigation on mechanical and wear performance of Ultrasonic-assisted stir cast AZ91D/Al2O3 magnesium matrix composites. Metals and Materials International **29** (9), 2767-2781 (2023).

[18] R. Schaller, Metal matrix composites, a smart choice for high damping materials. Journal of Alloys and Compounds **355** (1-2), 131-135 (2003).

[19] S. Gali, P. Prattipati, Microstructure, mechanical performance and wear behavior of AZ91D-B_4C-ZrO_2 hybrid composites. Composites **10**, 11 (2022).

[20] Y.T. Yao, L. Jiang, G.F. Fu, L.Q. Chen, Wear behavior and mechanism of B4C reinforced Mg-matrix composites fabricated by metal-assisted pressure less infiltration technique. Transactions of Nonferrous Metals Society of China **25** (8), 2543-2548 (2015).

[21] D. Dash, R. Singh, S. Samanta, R.N. Rai, Influence of TiC on microstructure, mechanical and wear properties of magnesium alloy (AZ91D) matrix composites. Journal of Scientific and Industrial Research **79** (2), 164-169 (2022).