



TRIBOLOGICAL INVESTIGATION OF MAGNESIUM MATRIX COMPOSITES ENHANCED BY WC NANOPARTICLE REINFORCEMENT

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ABSTRACT

Due to its remarkable unique properties, magnesium nanocomposites reinforced with ceramic reinforcements have emerged as an excellent structural material for automotive applications. The goal of this work is to investigate how well Mg-WC nanocomposites function in tribological applications. Response surface methods are used to analyze the effects of various input factors (represented as weight percentages of reinforcement, load, and speed) on the output responses (wear and coefficient of friction). Using powder processing, magnesium matrix composites supplemented with 5 weight percent WS₂ and 15-20 weight percent SiC particles were sintered. The study's primary objective was to employ a ball-on-disc tribometer to examine the wear and friction characteristics. Using an Al₂O₃ ball, the tribometer was subjected to normal room temperature stresses ranging from 1 to 4 N. The parameters include using a lubricant derived from PAO base oil, sliding at a speed of 22.5 mm/s, and a temperature of 110 °C. Optical profilometry and scanning electron microscopy were used to analyze the wear track and tribo-layer. The average friction coefficient for pure magnesium was found to be in the range of 0.16 to 0.46, while it was found to be in the range of 0.1 to 0.2 for magnesium metal matrix composites (MMCs). Magnesium Metal Matrix Composites (MMCs) showed remarkable antiwear properties and a significantly lower friction coefficient than both unreinforced magnesium and the magnesium alloy AZ31 under all testing conditions.

Keywords:

Friction, tribo-layer, and lubricant.

I. Introduction

The automotive and aerospace industries are now forced to concentrate on lowering emissions, increasing fuel efficiency, and decreasing weight due to the depletion of natural resources and sharp rise in fuel prices. The chosen material's strength, density, resistance to corrosion, and stiffness are the main considerations in transportation design. While higher payloads can be supported by materials with lower densities, stiffness and strength are factors that control performance and safety [1]. Since lightweight materials combine great strength with low weight, such as magnesium (Mg) and aluminum (Al), they have become essential solutions. Because of its high specific strength, notable stiffness, and low density relative to other structural materials, magnesium is especially useful for the automotive, aerospace, and electronics industries. Compared to aluminum, magnesium has a number of noteworthy advantages, such as a 50% improvement in machinability, a 25% to 50% boost in productivity, and an approximately 50% reduction in power consumption. As a result, a number of industries are switching from employing cast iron (CI), steel, and even aluminum (Al) to magnesium (Mg) alloys for a variety of components, including gearbox cases, housings, pistons, and power trains [2]. Furthermore, problems like as poor tribological characteristics, poor ductility, and restricted heat stability make magnesium and its alloys less useful. Because of recent developments in nanoscience, researchers have been concentrating more and more on nano-reinforcements. Enhancing several capabilities of

nanoparticles requires a high surface area. Consequently, in the magnesium matrix, researchers have employed a number of ceramic-based particles (B_4C , SiC , ZnO , Y_2O_3 , TiC , TiB_2 , BN , WC , and Al_2O_3). However, because of the difficulties that might occur from the dispersion, porosity, and agglomeration of nanoparticles—which result from their large surface area—the process of producing nanocomposites is a very complex one. Further considerations to consider while selecting the fabrication technology include the wettability issues and the cost implications of mass manufacturing. As a result, selecting a suitable synthesis method is essential to the creation of nanocomposites [3]. According to the literature, the main synthesis techniques used are powder metallurgy, the DMD method, and ultrasonic treatment associated stir-casting (UST). Lately, the magnesium matrix has been treated with ultrasonic therapy (UST) to integrate several particles, including SiC , CNT , AlN , and Al_2O_3 . Researchers have successfully combined ultrasonic treatment with stir casting to incorporate tungsten carbide nanoparticles into the AZ31 matrix. Mg- SiC nanocomposites were made in 2012 by Nie et al. using ultrasonic treatment (UST), and they were successful in achieving a uniform distribution of the reinforcing material [4]. Mg- AlN nanocomposites were successfully made by some without any agglomeration. UST generates mechanical vibrations by energizing piezoelectric crystals with high-frequency electric electricity. The vibrations then become more intense, which causes a large number of small bubbles to form. Although the mechanical properties are enhanced by the addition of tougher materials, there is a disadvantage because these hard particles are abrasive. When a specific weight is applied, this causes a high friction coefficient in the contact area. In the metal matrix, solid self-lubricating agents such as graphite, WS_2 (platelets of the 2H polytype), and MoS_2 are utilized to improve the lubricating properties when friction needs to be decreased. Because of their layered chemical structure, these solid lubricants have weak van der Waals (inter-layer) interactions. As a result, the shearing strength is low and there is little resistance to tangential pressures [5]. It has been found that WS_2 is a better friction modifier than MoS_2 . It is resistant to high temperatures and performs well in both dry and humid environments. Based on its chemical structure, WS_2 can be divided into two varieties: IF (inorganic fullerene architecture) composed of multi-layered spherical 'onion-like' cages with rounded edges, and 2H (layered platelets). At a temperature of 25 °C, a study found that 2H- WS_2 reduced friction more effectively than IF- WS_2 . The investigation involved a dry sliding contact. The remaining work is allocated as follows: a literature review of the task is provided in section 2. Section 3 lists the supplies and techniques. Section 4 presents the results and discussion section. Section 5 concludes with a summary of the work.

II. Review of Literature

The abrasive wear characteristics of AZ31- WC nanocomposites with varying weight percentages of nanoparticles (0, 0.5, 1, 1.5, and 2) were examined by Banerjee et al. [6]. The stir casting method with ultrasonic vibration assistance was used to create AZ31- WC nanocomposites. Cast composites are analyzed by optical microscopy, energy dispersive x-ray analysis, and scanning electron microscopy. The density and microhardness of nanocomposites are also being examined. A pin-on-disc tribotester is used to measure the effect of abrasive particle size and sliding distance on the wear and friction properties of nanocomposites. In order to determine the main wear process on worn surfaces under various experimental conditions, a 3D optical surface plotter, a SEM, and energy-dispersive X-ray spectroscopy (EDAX) are used in the research of worn surfaces. Fahad et al.'s study [7] described an effort to improve the AZ91D alloy's wear properties by adding hard particles as reinforcements, such as silicon dioxide (SiO_2) and tungsten carbide (WC). Three distinct composites were produced for this study using the ball milling process: AZ91D – WC , AZ91D – SiO_2 , and AZ91D – ($WC + SiO_2$). Next, these composites' tribological properties were examined with a pin-on-disc apparatus. The results of the investigation showed that the AZ91D alloy's hardness was greatly increased by the inclusion of WC and SiO_2 particles. According to a study on wear, the ploughing effect was mostly responsible for the wear rate increase of AZ91D alloy and its composites as the applied load increased. In contrast, as the sliding speed rose, the wear rate decreased, mostly as a result of the formation of a lubricating

tribolayer. Furthermore, when exposed to a load of 10 N and a sliding speed of 150 mm/s, the AZ91D – (WC + SiO₂) composite demonstrated a wear rate of 0.0017 mm³/m and a coefficient of friction of 0.33. Hybrid WC and SiO₂ particles were used to obtain these advantageous characteristics. The wear and friction properties of magnesium-tungsten carbide nanocomposites at high temperatures were studied by Banerjee et al. [8]. Using the ultrasonic vibration assisted stir casting approach, magnesium-tungsten carbide (Mg-WC) nano-composites were created with tungsten carbide (WC) in various weight percentages of 0.5%, 1%, 1.5%, and 2%. Optical microscopy, scanning electron microscopy, and energy dispersive x-ray patterns are used to characterize composites. The results of the characterisation confirm the presence of the reinforcing phase and the composites' structural integrity. We are examining the micro-hardness and density of the base alloy and the composites. The study looks into how the wear and friction characteristics of composites are affected by the sliding distance, applied load, and operating temperature. A high temperature tribotester is used for this, especially in dry sliding conditions. The applied load can be adjusted between 20 and 40 N, and the operating temperature can be adjusted between 50 and 250 °C. The results on micro-hardness confirm that adding WC to composites improves their mechanical properties. The wear and friction properties of magnesium alloy are considerably enhanced by the use of WC nanoparticles as reinforcement. Banerjee together with others. [9] This study investigates the effects of WC nanoparticles on the wear and friction characteristics of nanocomposites based on magnesium. The study's weight percentages of WC range from 0.5% to 2%. Using ultrasonic vibration in the stir casting process creates nanocomposites. Energy dispersive x-ray patterns, optical microscopy, and scanning electron microscopy are used to characterize the base alloy and the produced composites. The micro-hardness values of as-cast composites are ascertained using Vicker's micro-hardness tester. A pin-on-disk tribotester is used to test the wear and friction properties of produced composites in a dry sliding environment at room temperature. The sliding speed range is from 0.1 to 0.4 meters per second, and the operating load range is from 10 to 40 Newtons. Particles are present in the characterisation of magnesium composites, and more reinforcing results in better microstructural integrity. The amount of WC nanoparticles in a composite increase in direct proportion to its hardness.

III. Substances and Procedures

3.1 Composite metallurgy sample testing

A matrix material comprising magnesium powder (purity > 99%) with particle sizes ranging from 63 to 250 µm was procured from Sigma-Aldrich Inc. To improve the mechanical strength of the matrix, 30 µm-sized silicon carbide (SiC) particles from Thermo Fisher Scientific Inc. were utilized as a reinforcing phase. The second phase of reinforcement used to reduce friction and wear was made up of 2 µm-sized average tungsten disulfide particles that were procured from Sigma-Aldrich Inc. The proportion of tungsten disulfide (WS₂) particles was 10 wt%, whereas the weight fraction of silicon carbide (SiC) ranged from 15 to 20 wt%. All composites were consolidated using Spark Plasma Sintering (SPS). The production process involves utilizing a cryomill (Retsch, Germany) to combine the filler powders and basic matrix material equally. The mixture is subsequently sent through an SPS system (FCT Systeme GmbH, model HP D1050, Germany) in a vacuum environment for pre-compaction at 10 MPa and consolidation at 450 °C and 50 MPa. When sintering the mixed powder, a cylindrical graphite die with a 20 mm diameter was employed. The temperature was observed using a thermal couple. It was inserted into a pre-drilled hole in the die's outer wall's center. A tiny piece of graphite foil was placed along the inner surface of the die before the mixed powders were filled in. This prevented welding and allowed for a more equal flow of electric current. The temperature was heated at a rate of 50 °C per minute, with a dwell time of 5 minutes at 450 °C and a uniaxial pressure of 50 MPa. The magnesium powder was sintered using the same method in its native state to make comparisons with matrix composites easier. Additionally, an extruded AZ31 that is sold commercially (Luxfer MEL Technologies, Manchester, UK) was looked at for comparison.

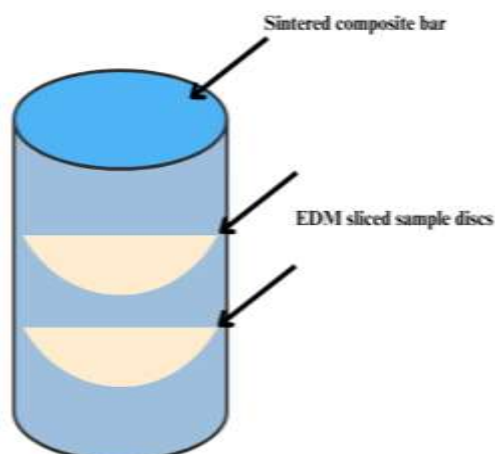


Figure 1: EDM sliced discs with SPS sintered Mg MMC

The approximate dimensions of the SPS sintered cylindrical Mg MMCs composite bars were 12 mm in length and 20 mm in diameter. Using electrical discharge machining, discs with a 19 mm diameter and 3.5 mm thickness were created (EDM). The discs that were cut using EDM and the contour of the sintered composites are shown in Figure 1. A detailed description of the experimental samples used in this investigation is given in Table 1. A high-precision balance (MS104S, Mettler Toledo, Switzerland) was used to quantify the composite density of the sintered samples with an accuracy of ± 0.0001 g. The Archimedes principle served as the basis for the measurement. Every sample was measured thrice, and the average result was utilized. A Durascan 70 micro-hardness tester made by Struers in the UK was used to conduct the Vickers hardness test on the polished surfaces of the magnesium magnesium metal composite. A Vickers diamond indenter tip was utilized with a load of 0.1 N and a hold time of 10 s for every micro-hardness indentation. For each sample, a series of twenty micro hardness indentations were made at regular intervals of one millimeter, and the mean Vickers hardness was calculated. A Bruker Hysitron TI Premier Nanoindenter was used to determine the elastic modulus, with an indentation depth of approximately $0.9\mu\text{m}$ and a normal load of 10 mN. For five seconds, each indentation was held. At 0.35, the Poisson's ratio was modified. Every sample was subjected to a matrix of 6×6 nano-indentations spaced $100\mu\text{m}$ apart, using the average elastic modulus. Using the Oliver and Pharr method, the elastic modulus was ascertained from the nano-indentation data. When analyzing the tribological behavior of a material, the values of hardness and elastic modulus are essential. Table 1 shows that the hardness and elastic modulus increase when reinforcement particles are added to the magnesium matrix. Relative density of Mg MMC2 was slightly lower than that of Mg MMC1. Because of their different chemical composition, the binding between the reinforcement particles and the matrix Mg particles is weaker than the bond between the Mg matrix particles. Relative density is expected to decrease with an increase in reinforcements. Friction testing is used to assess how these reinforcements affect the composite's tribological performance.

Table 1: Samples used for the work

Sample Designation	Materials	Fabrication Methods	Density (g/cm^3)	Relative Density %	Hardness Vickers	Elastic Modulus GPa
Mg	Magnesium	Powder metallurgy, SPS sintered	1.732	100	38.8	36.3
Mg MMC 1	75 wt.% Mg, 15 wt.% SiC, 10 wt.% WS ₂	Powder metallurgy, SPS sintered	1.97	97.5	72.7	45.1

Mg MMC 2	70 wt.% Mg, 20 wt.% SiC, 10 wt.% WS ₂	Powder metallurgy, SPS sintered	2.01	96.2	80.7	43.6
AZ31	3 wt.% Al, 1 wt.% Zn, Mg alloy	Extrusion, as received	1.7	-	91	45

3.2 Testing the friction

Using a Bruker Universal Mechanical Tester (UMT) platform, the tribological characteristics of Mg MMC samples were examined at room temperature and 110 °C. Alumina ceramic ball that was stationary was pressed up against by magnesium metal matrix composite (MMC) discs in the contact arrangement. The alumina ceramic ball measured 4 mm in diameter, 407 GPa for elastic modulus, 1365 Hv for Vickers hardness, and 0.21 for Poisson ratio. The ball was clamped into a chuck and pressed up against the oil-covered flat disc. New specimens in the shape of balls and discs were utilized for every test. Prior to the test, these specimens were carefully cleaned in an ultrasonic bath using solvents. A polyalphaolefins oil (PAO) with a density of 832 kg/m³ and a viscosity of 46.73 cSt at 40 °C and 7.96 cSt at 100 °C was used for the sliding tests. Because base oils are low polarity, they have little affinity for metal surfaces. This property was chosen on purpose to avoid any interaction between the oil molecules and the WS₂ particles for the metal surface. Magnesium metal matrix composite (MMC) discs machined by EDM were subjected to a standardized grinding and polishing process to guarantee uniform surface finishes in all samples. SiC paper with grits ranging from 1200 to 4000 was used for grinding, while alcohol-based diamond solutions with a thickness of 1 and 0.25 µm were used for buff polishing. This procedure tried to reduce oxidation brought on by contact with water. One N and four N normal loads were used for the reciprocating sliding tests. An overview of the testing circumstances is given in Table 2. Table 1's mechanical properties of magnesium metal matrix composites (MMCs) were used to estimate the corresponding initial mean Hertz pressures, which are shown in Table 3.

Table 2. Reciprocating sliding test conditions

Normal load	Stroke distance	Sliding speed	Testing duration	Ball material	Ball diameter	Disc material	Temperature
P = 1,4N	4.5mm	22.5 mm/s (2.5 Hz)	60 min	Al ₂ O ₃	4 mm	Mg MMCs, Mg alloy	Room temperature and 110 ⁰ C

Table 3: Initial mean Hertz contact pressures for tested materials corresponding to 1N and 4N

Sample designation	Hertz pressure at P = 1 N, MPa	Hertz pressure at P = 4 N, MPa
Mg	273	434
Mg MMC1	311	494
Mg MMC2	305	484
AZ31	310	493

3.3 Characterization

Understanding the existence of distinct phases, the distribution of particles, and the composition of the manufactured material is made easier with the help of microstructural characterization. It also helps to ascertain how well the samples are developing. Before being characterized in the current study, specimens go through a rigorous cleaning and etching procedure. Several grades of SiC abrasive paper are used to polish the samples. The samples are then polished some further with cloth. An etching solution containing 4 grams of picric acid, 10 milliliters of acetic acid, 70 milliliters of ethanol, and 10

milliliters of water is applied to the samples below. To investigate the different stages of interfacial bonding of the resulting nanocomposites, an LEICA optical microscope is employed. The dispersion of particles in nanocomposites is measured using a Japanese JEOL scanning electron microscope. The JSM-6360 model from Japan, which is the EDAX instrument, is utilized to examine the nanocomposites' compositional properties.

IV. Findings and discussion

The coefficient of friction (CoF) changes with time when the alumina ball interacts with magnesium composites or alloys at various testing temperatures and typical loads, as shown in Figure 2. The addition of WS₂ has improved the composites' tribological performance significantly across all testing conditions. During testing, a lubricating layer was seen to periodically form and burst at the interface between the Al₂O₃ ball and Mg MMC disk at room temperature (Figure 2(a)). Throughout the course of the testing, the coefficient of friction (CoF) for the two mixed metal composites (MMCs) is nearly the same and follows a regular pattern. The lubricating layer formed and stayed constant for about 17 minutes after the initial rise in coefficient of friction (CoF) during the running-in phase, with a CoF value as low as 0.097. The application of MoS₂ additives—which have a crystal structure with WS₂—helped to form and preserve a lubricant layer when the magnesium alloy and steel slid against one another. The experiment found that adding 1 weight percent of MoS₂ to the engine oil increased the friction coefficient from 0.08 to 0.12. The findings of this study validated his findings and confirmed that WS₂ had a positive effect on reducing interfacial friction. Moreover, Mg Metal Matrix Composites (MMCs) showed no increase in coefficient of friction (CoF) with applied loads at room temperature. This observation indicates a higher load-bearing capacity, which could be related to the use of reinforcing elements like WS₂ and SiC [10].

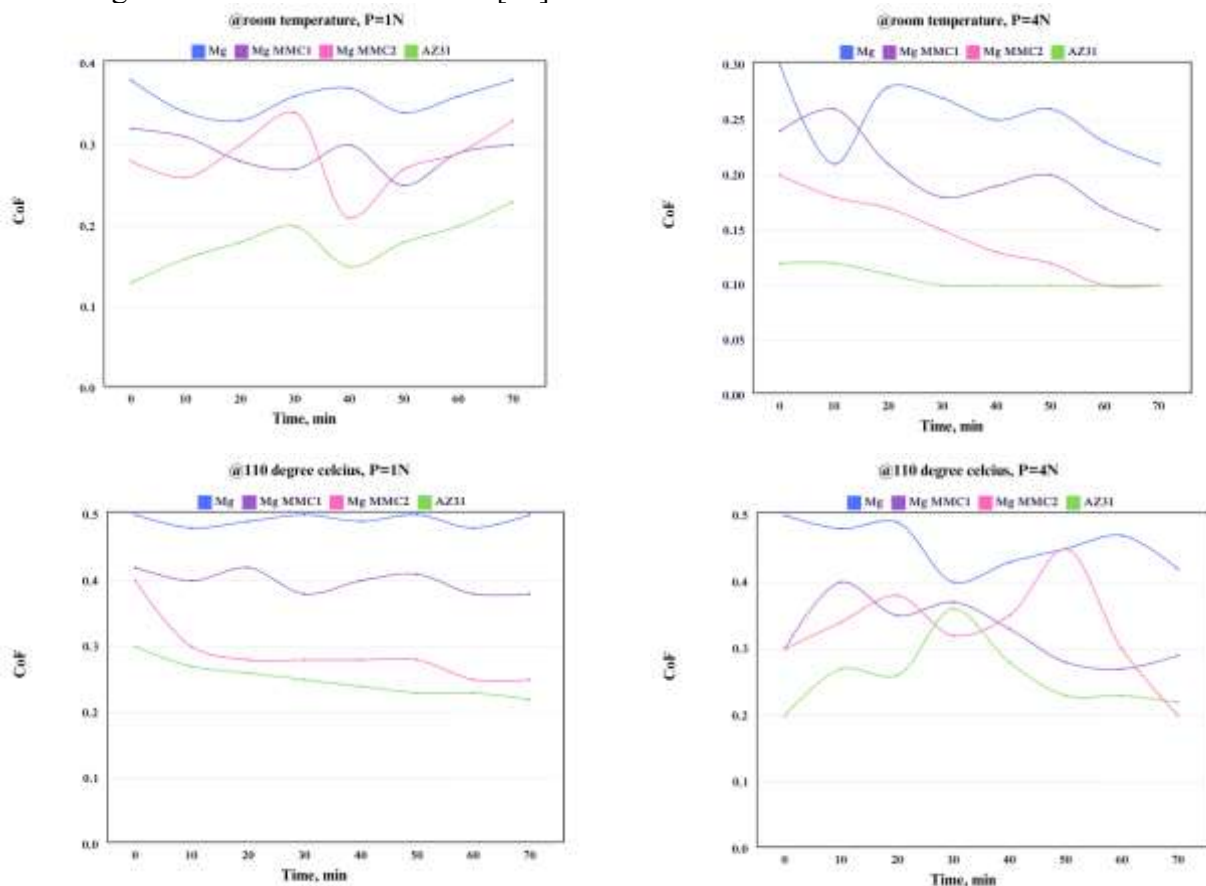


Fig 2: The coefficient of friction for Mg MMCs and Mg alloy varies with reciprocating sliding time under different conditions: (a) at room temperature with a load of 1 N, (b) at 110 °C with a load of 1 N, (c) at room temperature with a load of 4 N, and (d) at 110 °C with a load of 4 N.

The effects of temperature on the frictional properties of alloys and magnesium metal matrix composites (MMCs) are shown in Figures 2(c) and (d). The highest friction coefficient was found in sintered pure magnesium, closely followed by AZ31. At 110 °C and 1 N of stress, Mg MMC1 showed the least degree of friction, with an average coefficient of friction (CoF) of 0.15. As can be shown in Figure 2(c), composite Mg MMC2 had a CoF of 0.19 in contrast. Ratoi found that WS₂ reacted with the metal substrate when it was exposed to high pressure and high temperature in sliding contact, forming a significant chemical tribo-film in the process. The composites' improved tribological capabilities were caused by this tribo-film. Due to the bulk material's thermal softening at high temperatures, the coefficient of friction (CoF) for both pure Mg and AZ31 was somewhat decreased at 110 °C and 4 N of pressure. The coefficients of friction for both composites fluctuated throughout the first running-in phase before stabilizing after 30 minutes of rubbing. For the remaining stabilized stage, the average coefficient of friction (CoF) for both magnesium (Mg) composites was found to be 0.2. The wear rate ascertained from the wear volume data acquired from the Alicona profilometry is shown in Figure 3. Mg composites outperform pure Mg and Mg alloy in terms of wear resistance under all testing conditions. The composite's wear resistance was significantly increased by the addition of SiC and WS₂. The Mg MMC2 material, which has a higher percentage of SiC (20 wt%), shows a lower wear rate of $2.03 \times 10^5 \mu\text{m}^3/\text{N/m}$ at a temperature of 110 °C and a typical load of 4 N, compared to the wear rate of $3.62 \times 10^5 \mu\text{m}^3/\text{N/m}$ for Mg MMC1. Under identical conditions and at room temperature, the wear rate of magnesium MMC2 was $0.78 \times 10^5 \mu\text{m}^3/\text{N/m}$. About 25% of the wear rate of pure magnesium and 50% of the wear rate of AZ31 are represented by this number. For two Magnesium Metal Matrix Composites (MMCs), the wear rate did not increase with temperature at a load of 1 N. Previous studies have also demonstrated that the addition of WS₂ to the metal composites led to a notable reduction in the subsurface depth of plastic deformation, which in turn decreased bulk material wear.

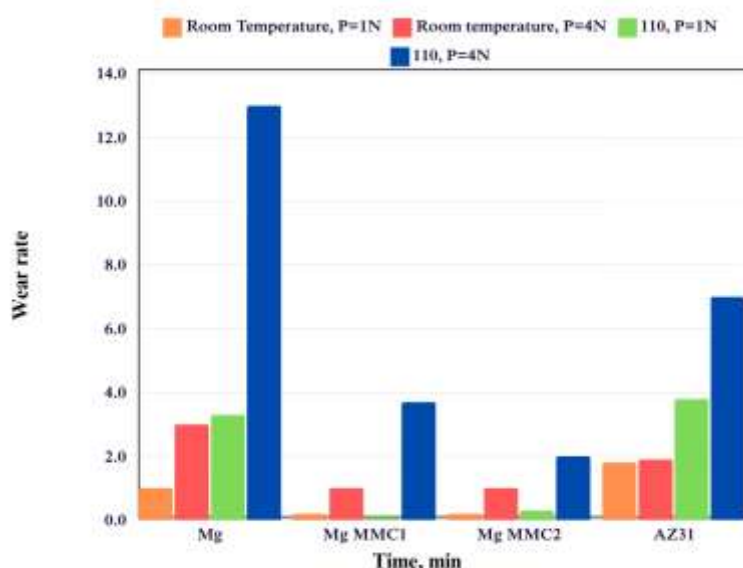


Fig 3: Wear rate of the materials being examined while sliding against an Al₂O₃ ball under different testing settings

An overview of the wear track metrics, including wear volume, wear scar depth, and width for all the materials that were looked at, is shown in Table 4. Under every testing condition, magnesium metal matrix composites (MMCs) showed remarkable antiwear properties. The wear scars observed on Mg MMC1 and MMC2 were significantly smaller than those on unreinforced Mg and AZ31 at elevated

testing temperatures and under normal load. More specifically, the wear scar depths for unreinforced Mg and AZ31 were 43.42 μm and 30.5 μm , respectively, while the wear scar depths for Mg MMC1 and MMC2 were measured to be 15.3 μm and 10.26 μm , respectively.

Table 4. Wear track dimensions and wear rate for tested materials subject to varying testing conditions

		Room temperature			110°C		
		Max.dept h, μm	Width, μm	Wear, volume, $\mu\text{m}^3 \times 10^7$	Max.depth, μm	Width, μm	Wear, volume, $\mu\text{m}^3 \times 10^7$
Mg	P = 1 N	8.80	439	0.84	15.78	674	2.75
	P = 4 N	18.01	711	2.45	43.41	1050	10.41
Mg MMC1	P = 1 N	2.81	199	0.20	2.09	214	0.13
	P = 4 N	5.10	309	0.54	15.2	619	2.93
Mg MMC2	P = 1 N	5.38	204	0.22	4.37	229	0.30
	P = 4 N	6.44	299	0.62	10.25	479	1.63
AZ31	P = 1 N	8.27	399	1.14	8.90	610	3.12
	P = 4 N	21.17	479	1.20	30.49	759	5.60

V. Conclusion

The objective of this study is to identify the optimal parameters for wear rate and coefficient of friction (COF) reduction in nanocomposites, as well as the combined effects of these two output parameters. Using the Response Surface Methodology (RSM) technique, this will be accomplished. Ultrasonic treatment is used in the stir-casting process to create nanocomposites. The SEM analysis's findings show that the WC nanoparticles are distributed uniformly throughout the magnesium matrix. The presence of WC particles in the material is verified by the EDS spectra. The influence of specific input parameters (weight percentages of WC, load, and speed) and their corresponding values on output parameters (wear rate and coefficient of friction) is analyzed. The coefficient of friction and the rate at which materials deteriorate are expressed mathematically. Surface plots show how several input factors affect output parameters together. The weight percentage of WC, speed, the square of the WC weight percentage, the square of the load, and the product of the WC weight percentage and speed are all highly significant factors, according to the wear rate ANOVA study. Conversely, the product of the weight percentage of the WC and the load, as well as the square of the speed, are important variables. The weight percentage of tungsten carbide (WC), load, the square of the WC weight percentage, the square of the speed, the product of the WC weight percentage and load, and the product of load and speed are all found to be highly significant factors according to the analysis of variance (ANOVA). Furthermore, the square of the weight and speed are also important factors. To find possible wear processes, the worn surfaces under ideal conditions are analyzed using the SEM.

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