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Examining the tribiological behavior of magnesium matrix composites reinforced with TiO_2 nanoparticles

Polimera Bala Karthik, Gokul Nath E, P. Chandrasekar, Galam Ananda Rao*
Department of Mechanical Engineering
Indian Institute of Industry Interaction Education and Research, Chennai, Tamil Nadu 600066

Abstract : The present study examines the tribological behavior of titanium dioxide (TiO_2) nanoparticle-reinforced magnesium matrix composites (MMCs). By using tribological testing and systematic microstructural analysis, we investigate how the TiO_2 nanoparticle content affects the wear and friction properties of the composites. Previous studies have shown that magnesium-alloy composites supplemented with TiO_2 and Hydroxyapatite (HAp) may be successfully fabricated using powder metallurgy, which results in improved mechanical and cytocompatibility qualities. Our goal in this work is to clarify the fundamental processes that underlie any noted gains in tribological performance. The results underline the importance of nanoparticle reinforcement in modifying the tribological properties of magnesium-based materials and offer insightful information on the possible uses of TiO_2-reinforced Mg-MMCs in a variety of industrial sectors.

Keywords:-Magnesium matrix composites; reinforcement Mechanical properties; Microstructure Powder metallurgy; and Nano-sized titanium dioxide (TiO_2)

I. INTRODUCTION

In recent years, there has been increased interest in magnesium (Mg) and its alloys because of their low density, impressive mechanical characteristics, and potential applications across different industries such as automotive, aerospace, and biomedical fields [1].Magnesium matrix composites (MMCs) represent a highly promising category of lightweight materials due to their outstanding blend of high strength, good ductility, and excellent corrosion resistance. However, their performance in applications involving friction and wear is significantly influenced by their tribological behavior, encompassing factors such as friction, wear, and lubrication. One strategic approach to improving these tribological properties is by reinforcing MMCs with nanoparticles [2,3].

However, their widespread use is limited by inherent drawbacks like poor wear resistance and low hardness. Addressing these challenges, researchers have been exploring innovative methods to improve the friction and wear properties of magnesium-based materials [4]. A promising method involves incorporating reinforcing nanoparticles into the magnesium matrix to create metal matrix composites (MMCs). Titanium dioxide (TiO_2) has emerged as a standout choice among various nanoparticles due to its exceptional mechanical properties, chemical stability, and abundance [5].When TiO_2 nanoparticles are dispersed within magnesium matrices, they have the potential to enhance the mechanical strength, wear resistance, and thermal stability of the resulting composites [6]. This study aims to explore the tribological behavior of magnesium matrix composites reinforced with TiO_2 nanoparticles.

By systematically analyzing wear mechanisms, frictional properties, and microstructural characteristics, this research aims to offer valuable insights into the potential applications and optimization strategies for these advanced materials [7,8]. Understanding the synergistic effects between the magnesium matrix and TiO_2 nanoparticles will facilitate the development of high-performance magnesium-based composites tailored for specific industrial applications. The knowledge gained from this study can be utilized for the development of new MMCs with enhance tribological properties for different tribological applications. The main contributions of this paper is presented below:

- The present study aims to investigate the tribological performance of TiO_2 nanoparticles-reinforced magnesium matrix composites.
- In this study, the effect of varying TiO_2 nanoparticle content will be studied systemically on the friction and wear performance of the composites.
- The investigation on the probable mechanisms for the performance enhancement will also be done through microstructural and worn surfaces analysis.

The rest of this paper is arranged as follows. Section 2 presents the materials and methods used for analyzing the the tribiological behavior of magnesium matrix composites reinforced with TiO_2 nanoparticles. Section 3 presents the outcomes of the analysis and section 4 concludes this paper.

II. MATERIALS AND METHODS

2.1 Materials used

S D Fine-Chem Limited is the supplier of magnesium powder (matrix), which has a mean particle size of 177 microns and a purity of more than 99.995%. The majority of the composite material is made up of this magnesium powder. Nanoparticles of titanium dioxide (Reinforcement) These particles, which are supplied by Merck Life Science Private

Limited, are more than 99.5% pure and have a size of less than 45 microns. To enhance the magnesium matrix's qualities, they are scattered throughout it.

2.2 Microstructure categorization

SEM analysis is a useful method for examining composite material microstructure. It offers comprehensive details on the location, shape, and bonding of reinforcement particles inside the matrix [9]. Understanding the mechanical and physical characteristics of the composite material requires knowledge of this information.In order to comprehend the distribution of reinforcement inside the MMC matrix, the polished surface of composite samples is examined as part of the micro structural analysis process. In this instance, the investigation employed a scanning electron microscope (SEM) to examine an MMC composite reinforced with TiO_2. Researchers were able to see the size and form of the reinforcing particles in the MMC matrix thanks to the SEM. It provided information on the interfacial connection between the matrix and the reinforcement.The research revealed information regarding the basic homogeneity of the composite structure. Additionally, the degree of particle agglomeration or clustering was evaluated. Overall, the SEM study gives light on how homogenous the composite's whole structure is. For best results, a consistent matrix structure and a homogenous particle dispersion are preferred.

2.3 Hot extrusion

Extrusion is a manufacturing method that molds materials by forcing them through a die under high pressure, similar to squeezing toothpaste out of a tube but with considerably more force and for tougher materials [10]. For this magnesium composite, extrusion offers many major advantages.

Stronger Bonds: Extrusion improves the connection between the various components, namely the matrix and reinforcement, resulting in stronger bonds within the composite.

Finer Grains: The compressing action of extrusion refines the grains inside the matrix, resulting in a more uniform structure that may boost the material's strength.

Sharper Grain Boundaries: Extrusion also helps to refine the grain boundaries, which can improve the material's overall characteristics.

A specialized instrument called as a die was created to give the composite item the correct form. This die was created to meet the final dimensions needed for the composite. Given magnesium's tendency to become flexible at high temperatures, the extrusion process was carried out at temperatures ranging from 250°C to 450°C. The composite's original diameter was 15 millimeters. It was crushed to 12 mm in diameter during the extrusion process, resulting in a 20% decrease. This decrease equates to a 1.56:1 extrusion ratio, which is computed by dividing the original diameter by the final diameter.

A temperature gradient was used to ensure that the composite material flowed smoothly and reduce stress on the die. The die was prepared to 200°C using a band heater, and the composite was heated to 350°C in a separate furnace. The temperature differential helped to ease the flow of the composite material through the die. The actual extrusion procedure was carried out utilizing a powerful 100-ton hydraulic press. To force the composite material through, a specially constructed punch with a tapered end that matched the form of the die was used. Lubrication oil was used to minimize friction between the punch and die walls during the extrusion process.

After the extrusion process, the composite turned into a cylinder measuring 12 mm in diameter and 18 mm in length. This reshaping not only changed the composite's physical dimensions, but it may also have improved its mechanical characteristics, making it more suitable for its intended purposes.

2.4 Sintering

Sintering is a specialized heat treatment procedure that compacts loose powder materials into a solid mass without causing them to melt. This procedure increases the material's grain size and overall strength, making it an important stage in material processing. The Two-Stage Sintering Approach used in this work consists of several phases, each with a unique function for achieving the desired material qualities. During Stage 1, the samples were exposed to a temperature of 500°C for four hours. The major goal of this step was to promote early bonding and strength growth inside the material [11].

To guarantee that the procedure did not cause any undesired interactions with the air, an inert nitrogen atmosphere was maintained. The samples were placed in separate crucibles, which were then organized on a tray and placed within a muffle furnace. Nitrogen gas was pushed through a ceramic tube at pressures ranging from 0.5 to 1 bar. Each batch of eight samples took around six hours to complete, including the heating and chilling steps. Following Stage 1, Stage 2 began, which included keeping the temperature at 400°C for an hour. This stage intended to refine the material's grain and grain boundaries, hence increasing its strength.

Similar to the previous step, an inert nitrogen environment was maintained throughout to ensure uniformity in the sintering process. The material was heated for one hour to attain the necessary temperature, then kept at 400°C for another hour to finish the stage. The two-stage sintering process is projected to have various advantages. Initiating the procedure at a lower temperature in Stage 1 may allow bonding without excessive grain formation, guaranteeing progressive material strength. Furthermore, Stage 2's lower temperature and shorter time may result in smaller grain size and better grain boundaries, thereby improving the material's mechanical characteristics.

2.5 Density analysis

The density of extruded magnesium composites with varied weight percentages of titanium dioxide (TiO_2) at 4, 8, 12, 16, and 20 wt% was determined using Archimedes' principle. To improve accuracy, each sample's measurements were taken three times. The samples were weighed in the air and then immersed in water to quantify the buoyant force. Using Archimedes' principle, the density of each composite was estimated using the known density of water (1 g/cm³).The experimental density of the composite was determined in accordance with the ASTM D792 standard. This standard provides how to determine the density of buoyant materials. It is most likely done using Archimedes' principle, which states that the buoyant force on an item immersed in a fluid is proportional to the weight of the fluid displaced by the object.

2.6 Mechanical testing

Porosity is the proportion of empty space in a composite material. It may be computed by subtracting the experimental density from the theoretical density, dividing by the theoretical density, then multiplying by 100% to get the percentage. It is computed as shown as follows:

$$Porosity = \frac{TD - ED}{TD} \times 100 \qquad (2.1)$$

Where the theoretical density and experimental density is represented as TD and ED.

A Vickers hardness tester [12] was used to determine the microhardness of the samples. Microhardness is the hardness of a very tiny portion of a material. The test was carried out in accordance with the ASTM E384 standard, which outlines the techniques for microindentation hardness testing. A test load of 25 grams-force (gf) was applied with a dwell duration of 15 seconds. The dwell time relates to how long the load is held on the material. The findings were calculated using the average of five indentations done on the samples. This helps to account for differences in the material's hardness throughout its surface.

The composite samples were then compressively tested at room temperature. Compressive testing measures a material's capacity to endure crushing pressures. The testing followed the ASTM E9 standard, which specifies how to conduct compression tests on metallic materials. Compressive loads were applied using a Universal Testing Machine (UTM). The crosshead speed of the UTM was set at 0.05 millimeters per minute. This determines the rate at which the load is applied to the sample. To prevent 740arreling (the sample bulging outwards during compression), Teflon tape was used as a lubricant between the machine's steel plate and the sample surface. The failure strain (%) is computed as follows:

$$Failure\ strain\,(\%) = \frac{variation\_in\_length}{overall\_length} \times 100 \qquad (2.2)$$

2.7 Wear testing

Wear testing was performed on samples with a diameter of 10 mm and a height of 35 mm, following the ASTM G99 standard and using a pin-on-disc wear tester. Prior to testing, the specimen pin's surface was carefully polished to guarantee appropriate contact with the flat steel disc face. The surface was then completely cleaned with a solvent and dried before the experiments began. The initial and final weight measurements of the sample pin were taken before and after the wear testing process to determine the level of wear. The wear rate (mg/m) is computed as follows:

$$Failure\ strain\,(\%) = \frac{variation\_in\_length}{overall\_length} \times 100 \qquad (2.3)$$

2.8 Cytocompatibility

Cell viability assays are critical experimental techniques used to identify viable (alive) from non-viable (dead) cells within a cell population. These tests are important in many disciplines, including drug discovery, medical trials, and therapeutic applications, where determining cell viability and cytocompatibility is critical. In vitro testing, the MTT assay, which stands for 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide, is a well-known technique for assessing cell viability. This assay uses an indirect contact technique in line with ISO 10993-12 standards. The method starts with the addition of a yellow tetrazolium salt to the cell culture. In the presence of living cells, this salt reduces and transforms into a purple formazan molecule.

Dead cells, on the other hand, do not have this purple hue because the yellow tetrazolium salt has not been fully reduced. The intensity or optical density of the purple hue produced is directly related to the number of living cells in the culture. A spectrophotometer is used to quantify this optical density, allowing for exact measures of cell viability based on color intensity. For comparative analysis and accuracy, normal cells cultivated in a standard growth medium are utilized as controls, reflecting 100% cell viability. This control serves as a baseline for assessing the survivability of test samples, assuring consistent and trustworthy findings across studies. The cell viability (%) is computed as follows:

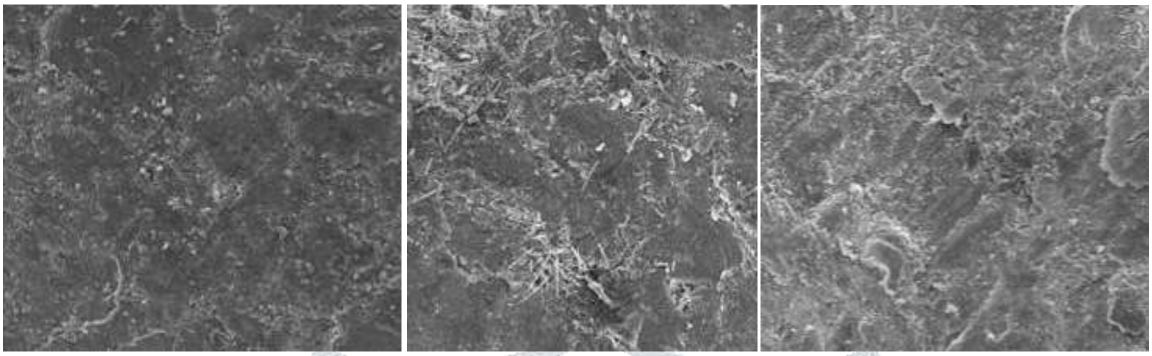
$$Cell\_viability(\%) = \frac{OD_{SAMPLE}}{OD_{CONTROL}} \times 100 \qquad (2.4)$$

Where OD represents the optical density.

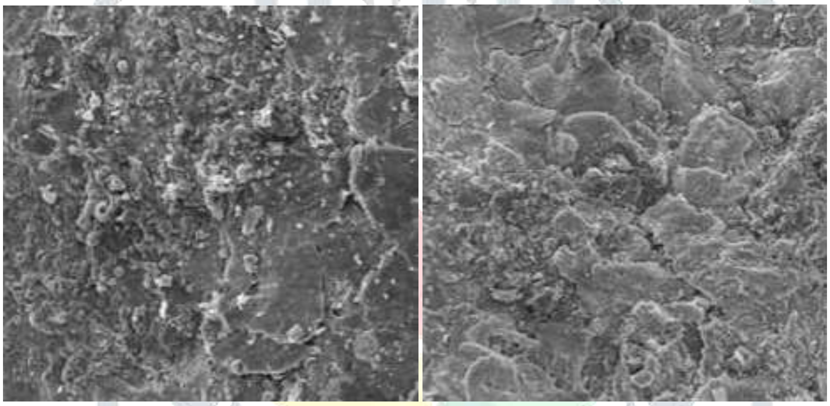
III. RESULTS AND DISCUSSION

3.1 SEM analysis

SEM examination was carried out on Mg-TiO_2 elemental mixes to confirm the equal distribution of TiO_2 reinforcement across the Mg matrix at different weight percentages. The investigation sought to establish that a uniform distribution would improve bonding via grain development, hence enhancing material characteristics. Micrographic pictures acquired from SEM inspection of extruded specimens as shown in Figure 1 demonstrated that the nanocomposites' structure and grain development improved continuously as TiO_2 reinforcement increased. While pure magnesium had bigger grain sizes and more micropores, the addition of nano TiO_2 resulted in a refined grain structure and a smoother surface with fewer micropores in the Mg-TiO_2 composite. This suggested a consistent distribution of TiO_2 reinforcement throughout the matrix.



(a) 4% TiO_2 (b) 8% TiO_2 (c) 12% TiO_2



(d) 16% TiO_2 (e) 20% TiO_2

Figure 1: Nano TiO_2 particles analysis in Mg-TiO_2 composites with different TiO_2 composition.

3.2 Micro hardness analysis

The study aimed to compare the mechanical characteristics of pure magnesium (Mg) with composite materials made by integrating nano-sized titanium dioxide (TiO_2) particles into the Mg matrix and the results are shown in Figure 2. Mechanical characteristics, notably hardness, were assessed at room temperature, most likely using a Vickers hardness test with the stated load and dwell time settings. Pure magnesium had rather low hardness values. In contrast, the addition of nano-sized TiO_2 particles resulted in a considerable increase in hardness compared to the pure Mg equivalent. Two key reasons contributed to the increase in hardness. To begin, the inclusion of nano-sized TiO_2 particles most likely contributed to the refinement of the magnesium grains in the composite, since finer grains are often associated with enhanced hardness.

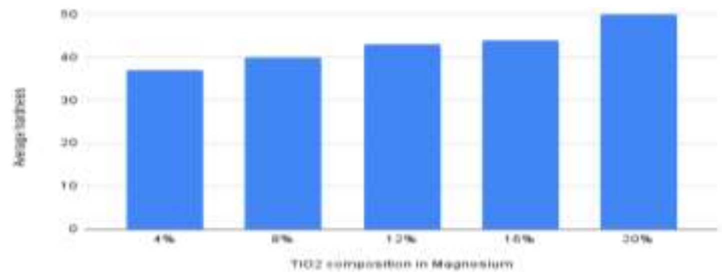


Figure 2: Micro hardness analysis.

3.3 Density and porosity analysis

Table 1 presents the density and porosity data for various bio-composite samples, measured experimentally using Archimedes' principle and calculated theoretically using the rule of mixture. The results showed close agreement between the experimental and theoretical densities, highlighting the efficacy of the powder metallurgy process in producing near-dense bio-composites. Among the samples, "20% TiO_2" exhibited the lowest porosity at 0.14%, indicating it was the most densely packed sample.

Table 1: Density and porosity identification of MG-TiO_2 powder mixture.

Sample composition of TiO_2 in magnesium	Density		Porosity (%)
	Experimental (g/cm^3)	Theoretical (g/cm^3)	
4%	2.2612	2.2610	0.1314
8%	2.2001	2.2069	0.1122
12%	2.1423	2.1457	0.1159
16%	2.0821	2.0891	0.1186
20%	2.0112	2.0331	0.1456

3.4 Compression test

The study examined magnesium (Mg) composites supplemented with nano-sized titanium dioxide (TiO_2) particles in a variety of compositions (4-20 wt%) and the results are shown in Figure 3. Compressive strength was measured at room temperature using samples with varied TiO_2 concentration and a diameter of 12mm. The results showed that increasing the TiO_2 concentration enhanced both micro hardness and compressive strength. This improvement in mechanical characteristics was most likely due to the TiO_2 particles limiting grain formation, resulting in enhanced density and hardness in the composite materials.

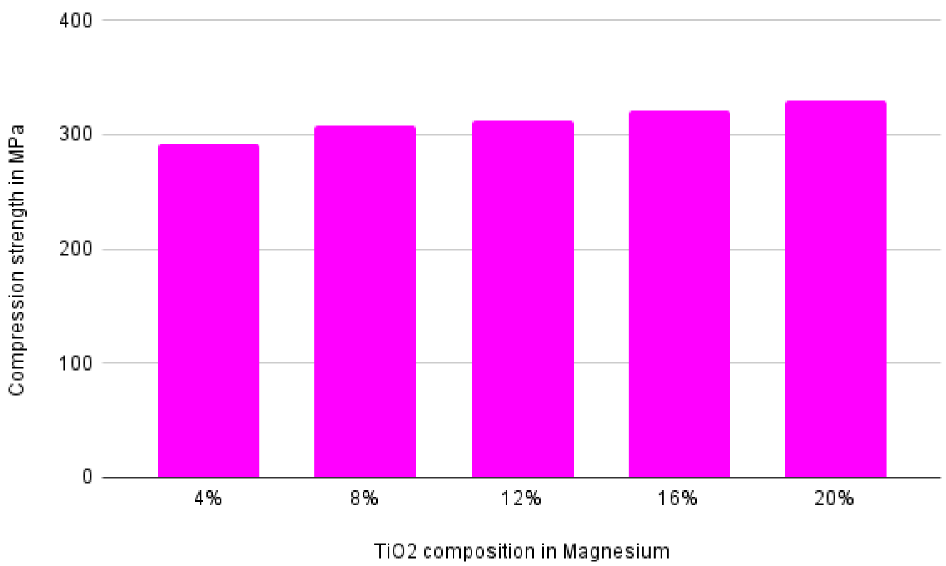


Figure 3: Compression strength analysis.

3.5 Wear behavior analysis

Figure 4 illustrate the link between wear rate and applied load. The rate of wear lowers as the quantity of hybrid reinforcement in the magnesium matrix rises. This suggests that increased reinforcement content results in better wear resistance. The greater connection between the reinforcements and the matrix is thought to be responsible for this improvement. Better bonding results in more efficient load transmission, which reduces wear. Among the samples, 20% TiO_2 has the lowest wear rate, most likely due to the favorable benefits of its special hybrid reinforcement combination.

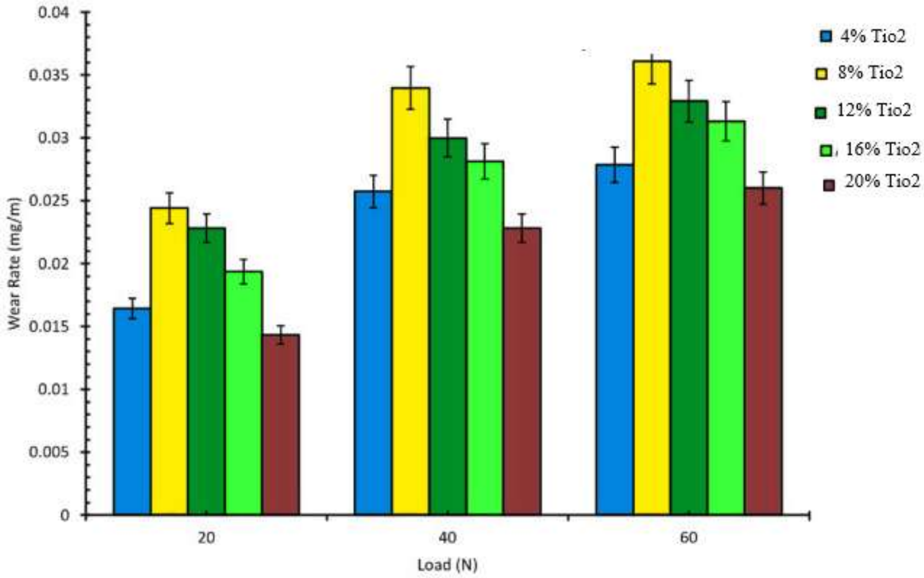


Figure 4: Wear rate for different Mg-TiO_2 composites.

3.6 Failure strain and compressive strength analysis

The table 2 displays the sample composition of TiO_2 in magnesium, representing the weight % of TiO_2 integrated into the magnesium matrix. The table also provides the failure strain values for each composite sample, which show the percentage of deformation that the material can withstand before failing under stress, most likely tension. The provided results include a standard deviation (±) to reflect measurement variability. Observational patterns show that as the composite's TiO_2 concentration grows from 4% to 12%, so does the failure strain. This implies that adding up to 12% TiO_2 increases the material's capacity to deform before it breaks. The inclusion of TiO_2 particles may reinforce the magnesium matrix, allowing it to tolerate more stress before breaking. This may explain the first rise in failure strain. However, if there is too much TiO_2, the particles may cluster together, resulting in weak areas within the material. These weak areas might cause early failure at lower strains, which explains why failure strain decreases with greater TiO_2 concentration. Overall, the table indicates that an appropriate amount of TiO_2 (about 12% in this case) may be applied to magnesium to increase its failure strain.

Adding TiO_2 particles improves the compressive strength of MMCs as shown in Table 2. This is because TiO_2 offers rigidity and support, allowing the composite to withstand compressive pressures without collapsing. However, there is a limit. Too much TiO_2 can cause overlapping particles, stress concentrations, and flaws in the material, lowering compressive strength. The difference in thermal expansion and Young's modulus between the reinforcements and the matrix can increase bonding and compatibility at the interface, resulting in greater load transmission.

Table 2: Experimental analysis using failure strain and compressive yield strength.

Sample composition of Tio_2 in magnesium	Failure strain	Compressive yield strength
4%	17.4±0.9	208±2 MPa
8%	19.6±1.6	257±5 Mpa
12%	22.0±1.2	259±5 Mpa
16%	20.8±1.0	248±6 Mpa
20%	17.5±0.8	204± 3 Mpa

IV. Conclusion

In this work, MMC composites enhanced with TiO_2 were effectively produced utilizing powder metallurgical techniques. The efficiency of this technology was demonstrated by the manufacture of TiO_2 reinforced MMC composites reinforced with individual nanoparticles. SEM micrographs indicated that TiO_2 particles were efficiently bonded and uniformly dispersed inside the matrix, resulting in a homogenous composite structure. This certification demonstrates the quality of the manufacturing method used. The comparison of predicted and experimental densities revealed a high level of consistency, demonstrating the powder metallurgy process's capacity to produce dense and compact composite samples. The synergistic impact of introducing TiO_2 reinforcements resulted in considerable improvements in wear resistance (up to 27%), compressive strength (up to 26%), and microhardness (up to 13%). This improvement can be ascribed to increased affinity between the matrix and reinforcement, which optimizes load distribution within the composite. Furthermore, cell viability experiments revealed that cells grown with the extracted hybrid samples had vitality equivalent to that of whole cell culture medium, in contrast to non-hybrid composites. This research emphasizes MMC composites' superior biocompatibility, emphasizing their potential for biological applications. Overall, this work indicates the promising potential of MMC composites reinforced with TiO_2 in terms of mechanical characteristics and biocompatibility, opening the path for their use in varied industrial and biomedical fields.

References

1. Arora, G.S., Gupta, A. and Saxena, K.K., 2024. Evaluation of mechanical, microstructural, tribological characteristics and cytocompatibility in AZ31 hybrid bio-composite reinforced with TiO_2-HAp. *Results in Surfaces and Interfaces*, *14*, p.100174.
2. Radhakrishnan, G., Anirudh, P.V. and Kumar, S.A., 2019, July. Mechanical behavior of nanoparticulate TiO_2 reinforced magnesium matrix composite. In *IOP Conference Series: Materials Science and Engineering* (Vol. 574, No. 1, p. 012006). IOP Publishing.
3. Bharat, N. and Bose, P.S.C., 2023. Optimizing the wear behaviour of AA7178 metal matrix composites reinforced with SiC and TiO_2 nanoparticles: a comparative study using evolutionary and statistical methods. *Silicon*, *15*(11), pp.4703-4719.
4. Bharat, N. and Bose, P.S.C., 2022. Optimization of tribologicalbehaviour of TiO_2 nanoparticles reinforced AA7178 alloy matrix using ANN and Taguchi's methodology. *Surface Topography: Metrology and Properties*, *10*(2), p.025032.
5. Sreenivasulu, G., Mohan, C.B., GB, V.K. and Ananthaprasad, M.G., 2022. Mechanical and Tribological Behavior of Aluminum Alloy LM13 Reinforced with Titanium Dioxide Metal Matrix Composites. *Iranian Journal of Materials Science & Engineering*, *19*(4).
6. Gnanavelbabu, A., Vinothkumar, E., Ross, N.S. and Prahadeeswaran, M., 2023. Investigating the wear performance of AZ91D magnesium composites with ZnO, MnO, and TiO_2 nanoparticles. *The International Journal of Advanced Manufacturing Technology*, *129*(9), pp.4217-4237.
7. Elumalai, P.C. and Ganesh, R., 2020. Synthesis and characterisation of magnesium matrix composite reinforced with titanium dioxide nanoparticulates. *Materials Research Express*, *7*(1), p.015093.
8. Yadu Krishnan, J., Thoufiq Mohammed, K., Veera Ajay, C. and Manisekar, K., 2022. Study on the Wear Behaviour of Magnesium Metal Matrix Composites Reinforced with Titanium Dioxide (TiO_2). In *Recent Advances in Materials Technologies: Select Proceedings of ICEMT 2021* (pp. 655-662). Singapore: Springer Nature Singapore.
9. Meenashisundaram, G.K., Nai, M.H. and Gupta, M., 2015. Effects of primary processing techniques and significance of hall-petch strengthening on the mechanical response of magnesium matrix composites containing TiO_2 nanoparticulates. *Nanomaterials*, *5*(3), pp.1256-1283.
10. Elumalai, P.C. and Ganesh, R., 2020. Synthesis and characterisation of magnesium matrix composite reinforced with titanium dioxide nanoparticulates. *Materials Research Express*, *7*(1), p.015093.
11. Pc, E., Radhakrishnan, G. and Emarose, S., 2021. Investigation into physical, microstructural and mechanical behaviour of titanium dioxide nanoparticulate reinforced magnesium composite. *Materials Technology*, *36*(10), pp.575-584.
12. Shahdad, S.A., McCabe, J.F., Bull, S., Rusby, S. and Wassell, R.W., 2007. Hardness measured with traditional Vickers and Martens hardness methods. *Dental Materials*, *23*(9), pp.1079-1085.