MECHANICAL CHARACTERIZATION OF HYDROXYAPATITE REINFORCED WITH MAGNESIUM METAL MATRIX COMPOSITE FABRICATED BY STIR CASTING

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Abstract

Biocompatibility and biodegradability are the two important properties of magnesium alloys, resulting in increased use of magnesium alloys for biomedical applications. Hydroxyapatite is highly bioactive and has a chemical and crystallographic structure to bone. HA is a potential ceramic material for manufacturing MMC of Mg that will have the advantages of both Mg and HA. The need is to design magnesium alloys with suitable mechanical properties and corrosion rates that can be calibrated. Metal matrix composite made of magnesium alloys may be used as a solution to this problem. In the present work, an MMC made of magnesium alloy AZ31B as a matrix and hydroxyapatite (HA) particles as reinforcements have been examined for mechanical characteristics by varying the hydroxyapatite by 0%, 5%,10%, and 15% by weight. The structural properties of the produced samples were investigated by Rockwell hardness and Optical microscope. Optimization is carried out using Taguchi L27 orthogonal array. The mechanical properties of the MMC-HA were adjustable by the choice of HA particle size and distribution. Pin on disc machine was used for wear test to find the influence of HA on stir casted composite. The main objective is to minimize the resistance caused by wear. The optimum parameters for the low wear and friction are determined by Taguchi-ANOVA using Minitab-19, and analyzing the microstructure of the samples through an Optical microscope. The wear and coefficient of friction have been evaluated by varying factors like applied load 10, 20, and 30N, percentage of Hydroxyapatite with 5%, 10%, and 15 %, and speed of 250,500, and 750 rpm. At 10%reinforcement,10N, and speed at 250rpm, it was discovered that the wear and friction were reduced.
Keywords:
Magnesium; Hydroxyapatite; Metal matrix composite; Corrosion; Mechanical properties; Cytotoxicity

INTRODUCTION

Magnesiumalloys are a suitable class of biodegradable metal implants[1]. Current research indicates a high demand to design magnesium alloys with adjustable reinforcement with mechanical properties that can be enhanced. There is a need for magnesium alloys with changeable in vivo corrosion rates that do not degrade[2]. Metal matrix composite based on magnesium alloys may be a solution[2, 3]. The MMCs have gained increasing interest in the last decade particularly in automotive applications[4-6] and general transportation based on their higher specific stiffness, strength, creep resistance, and minimized sensibility to galvanic corrosion[7-9]. By selecting the suitable reinforcement for the composites, MMCs can have tailormade mechanical properties (Young's modulus, tensile strength), and customizable corrosion properties. Especially calcium is known to reduce the susceptibility of magnesiumto corrode when added in amounts of a few tenths of weight percentages [4,5]. Hydroxyapatite (HA), a component of natural bone, is well known for having limited solubility in bodily environments. Therefore, HA particles seem tobe suitable as reinforcements in magnesium-based MMCs. In previous studies, magnesium alloy AZ91D revealed local corrosion attacks in vitro and in vivo [8,9].In this study, a metal matrix composite made of AZ31B as a matrix and HA particles as reinforcements have been investigated in vitro for mechanical and wear tests.

MATERIALS AND METHODS

MATERIAL PRODUCTION

The MMCs were produced by mixing 5,10, and 15 percent by weight of HA powder(particle size < 44 micrometer)and magnesium AZ31B alloy. Magnesium AZ31B alloy blocks are used for fabrication through stir casting. The matrix material (Mg AZ31B) was heated to the melting point by setting the temperature to 900^{0}C. The reinforcement particles are distributed into a molten matrix by mechanical stirring. With the help of a stirrer, the reinforcement was uniformly distributed into the molten metal. The resultant molten alloy with ceramic particles is then transferred into the mould. Venting of the mould was done by tight screws, to create a perfect vacuum in the mould. The samples are then machined and various other tests are performed.

Fig 1 Equipment of Stir casting machine

MECHANICAL AND METALLURGICAL TESTING

In Tensile testing, the samples were prepared according to ASTM EB standards[10].Three samples of MMC-HA were extruded for uniform distribution of HA. Samples are subjected to controlled tension until failure[11]. Properties that are measured via a tensile test are ultimate tensile strength, breaking strength, maximum elongation, and reduction in area. The sample after the tensile test is shown in Fig 2. The characteristics that can be calculated from these measurements are Young's modulus, poison ratio, yield strength, and strain hardening, UTM is used for measurement shown in Fig 3 and 4.

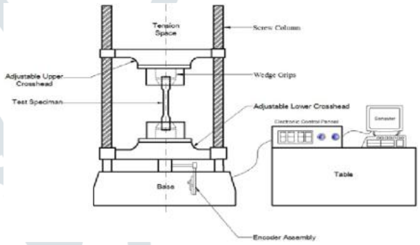

Fig 2 Sample after tensile test. Fig 3 UTM Fig 4 Schematic of UTM

HARDNESS TESTING

Hardness testing is used for determining a material's hardness or resistance to penetrationwhen test samples are very small or thin, or when small regions in a composite sample or plating are to be measured. It can provide precise and detailed information about the surface features of materials with a fine microstructure, multi-phase, non-homogeneous or prone to cracking. The microhardness test can measure surface-to-core hardness on carburizedor case-hardened parts (case depths) and surface conditions such as grinding burns, carburization, or decarburization. The hardness testing machine is shown in fig 5

Fig 5 Hardness Testing Machine

WEAR CHARACTERIZATION

Frictional force and wear test of magnesium sample is carried on a pin on disc [12] shown in Fig 6.The input parameters for the experiment are normal load (ie 10N,20N & 30N) the sliding velocity (ie 500m,1500m & 2500mm) A high precision electronic balance having accuracy 0.001g has been used to calculate the wear losses.

MECHANISM OF WEAR TEST

Magnesium metal matrix composites test samples have been prepared as pins of dimensions 10mm diameter and 30mm height. It is important to ensure that the test samples surfaces have been flat and are polished by using metallographic techniques prior to wear testing. The procedure involves grinding of composite Magnesium surface manually using 240,320, and 400 grit silicon carbide papers after that using a low speed polishing machine to polish with 0.5,1,5m alumina. This method of preparation has made it possible to significantly relieve the surface tension between the hard and soft magnesium matrix. Followed by cleaning by acetone and methanol solutions. Before each wear test, the counter face material was similarly polished and cleaned using an ultrasonic cleaner and acetone and methanol solutions. The tests were all carried out at room temperature after the steel slider was polished.

Fig 6 Pin on Disc Sliding Wear Testing machine with an integrated system

The tests have been carried out by applying normal loads such as 10, 20, and 30 N at different sliding distances such as 250m, 500 m, and 750m at different reinforcements such as 5, 10, and 15 %. The wear rates will be converted into volumetric wear loss. The wear losses of the specimens have been measured using a high precision (accuracy 0.001g) electronic balance. The fig 6 shows the working on Pin on disc tester for parameters. The weight loss of the total test samples was measured under dry conditions. The composite volume loss has been computed using the weight loss information. There is scatter in the results since wear testing yields inconsistent results. As a result, the wear rate calculated for any sliding distance is determined as the average of values collected across the period. The parameters considered during the performance of the wear test are normal load, Percentage of Reinforcement, and sliding distance, with these three levels. The corresponding inputparameters are tabulated. As per the DoE from Taguchi Method in Minitab-19 for three levels and four factors the orthogonal array has been generated and are tabulated in Table 1.

Table.1 DoE L27 Orthogonal Array for Tribology study of Magnesium MMC

RUNS	% Of CompositeReinforcement	Applied Load, L (N)	RPM
1	5	10	250
2	5	10	500
3	5	10	750
4	5	20	250
5	5	20	500
6	5	20	750
7	5	30	250
8	5	30	500
9	5	30	750
10	10	10	250
11	10	10	500
12	10	10	750
13	10	20	250

14	10	20	500
15	10	20	750
16	10	30	250
17	10	30	500
18	10	30	750
19	15	10	250
20	15	10	500
21	15	10	750
22	15	20	250
23	15	20	500
24	15	20	750
25	15	30	250
26	15	30	500
27	15	30	750

RESULTS

OPTIMIZATION OF PARAMETERS

Results from pin on disc, Wear rate and co-efficient of friction observer from pin of disc equipment for the factors percentage of composite, applied load, sliding velocity and sliding distance with varying levels has been shown in the following table 2

Table.5.1 Pin-on-Disc-Results L27 -Orthogonal Array for Magnesium MMC

Runs	% reinforce ment	Load(N)	RPM	Wear (µm)	SNRA1 (db)	Friction(N)	SNRA2 (db)
1	5	10	250	205	-49.19	2.8	-12.34
2	5	10	500	200	-50.16	4.2	-14.51
3	5	10	750	225	-50.32	5.1	-15.67
4	5	20	250	251	-50.92	6.2	-16.85
5	5	20	500	230	-49.76	6.8	-16.91
6	5	20	750	253	-49.98	7.8	-17.87
7	5	30	250	450	-54.85	4.1	-17.69
8	5	30	500	501	-53.98	6.2	-16.11
9	5	30	750	540	-52.11	11	-19.15
10	10	10	250	110	-40.56	5.5	-15.78
11	10	10	500	120	-43.76	6.1	-16.45
12	10	10	750	125	-45,44	6.8	-16.34
13	10	20	250	232	-51.50	6.2	-16.77
14	10	20	500	280	-53.90	6.6	-16.89
15	10	20	750	295	-52.22	7.8	-17.36
16	10	30	250	260	-40.13	3.6	-13.50
17	10	30	500	180	-42.22	4.9	-14.67
18	10	30	750	260	-44.32	5.5	-15.89
19	**15**	**10**	**250**	**90**	**-38.63**	3.1	**-13.39**
20	15	10	500	101	-39.23	3.7	-13.79
21	15	10	750	105	-41.66	6.5	-16.98
22	15	20	250	130	-38.96	5.7	-15.76
23	15	20	500	110	-39.87	6.2	-16.86

24	15	20	750	116	-38,66	6.5	-16.97
25	15	30	250	150	-39.31	4.5	-14.33
26	15	30	500	115	-38.22	4.9	-14.97
27	15	30	750	121	39.24	6.1	-16.56

The Taguchi technique has been used to optimize and assess the test parameters, such as the weight percentage of the applied load of the particle reinforcement and RPM on wear and friction, with suitable Signal-to-noise (S/N) and analysis of variance (ANOVA).

WEAR:

The SN ratio response for the wear, and the Magnesium metal matrix composites (MMC) are presented in Fig 7 and Fig 8 which shows that among all the factors, the percentage of Composite particulate of reinforcement has been the most influential and significant parameter followed by, load and RPM.

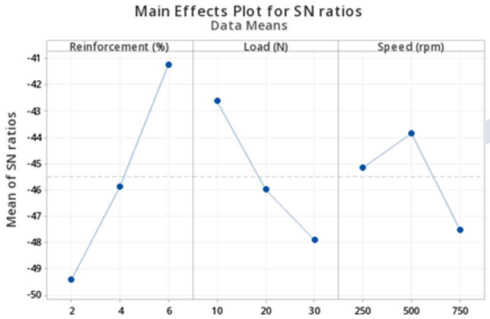

Fig 7 SN ratio of Wear

Table 3 Response of SN ratios- Wear of AZ31B –HAP MMC

Level	Percentage of reinforcement	Load	RPM
1	-49.40	-42.61	-45.15
2	-45.86	-45.98	-43.83
3	-41.22	-47.89	-47.50
Delta	8.18	5.28	3.66
Rank	1	2	3

Effect Of Parameters on Signal-To-Noise Ratios

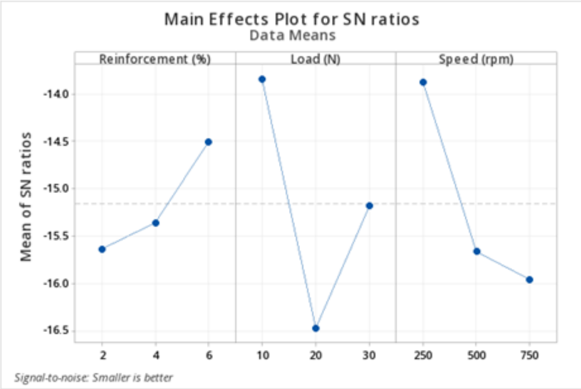

Fig 8 S/N ratio for friction

Table 4 Response of SN ratios friction of AZ31B MMC

Level	% of Reinforcement	Load	Rpm
1	-15.63	-13.84	-13.87
2	-15.36	-16.47	-15.66
3	-14.50	-15.18	-15.95
Delta	1.13	2.62	2.08
Rank	3	1	2

STATISTICAL ANALYSIS BY ANOVA

Using ANOVA, it has been observed that the percentage of reinforcement (53.71%), load (9.35%)RPM (1.35%) have a greater influence on the wear. The effect of RPM has been found to be minimum (1.35%), indicating an appreciable increase in the wear by increasing the composite reinforcement.

Table 5 Analysis of variance-Wear

Source	DF	Seq SS	Contribution	Adj SS	AdjMS	F-Value	P-Value
% reinforcement	1	183416	48.00%	183416	183416	70.80	0
load	1	93312	24.42%	93312	93312	36.02	0.094
rpm	1	45804	11.99%	45804	45804	17.68	0.546
Error	23	59584	15.59%	59584	2591		
Total	26	382115	100.00%				

From table 6 has been observed that the RPM (34.44%), load (24.99%) composite reinforcement(3.45%) in this parameters RPM have the greater influence on the friction whereas RPM has a very less influence. the percentage of composite reinforcement factor has greater control on co- efficient of friction than the other factors. Hence, added percentage of reinforcement has becomean important parameter to be taken into account while considering co-efficient of friction[12].

Table. 6 Analysis of variance friction

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
% reinforcement	1	2.722	3.66%	2.722	2.722	1.00	0.327
Load	1	2.722	3.66%	2.722	2.722	1.00	0.327
Rpm	1	6.361	8.56%	6.361	6.361	2.34	0.140
Error	23	62.476	84.11%	62.476	2.716		
Total	26	74.281	100.00%				

Effect of parameters on Tribological Characteristics

The wear factors, defined as the wear to the percentage of reinforcement, Load (N), and RPM have been important parameters, in quantifying the wear resistance.

Effect of the Percentage of Reinforcement on Wear

Fig 9-11 shows that wear has been calculated after a constant sliding period of 5 minutes for all the samples at three loads (10N, 20N & 30N) and250,500,750 RPM. The main observation is a decrease in wear with the increase in other parameters. As load increases wear also increases. This can be attributed to the fact that an improved hardness of the composite, resulting fromthe incorporation of hard particles, which acts as a harder phase into the matrix—an increase in hardnessresults in the improvement of the wear resistance of the material.

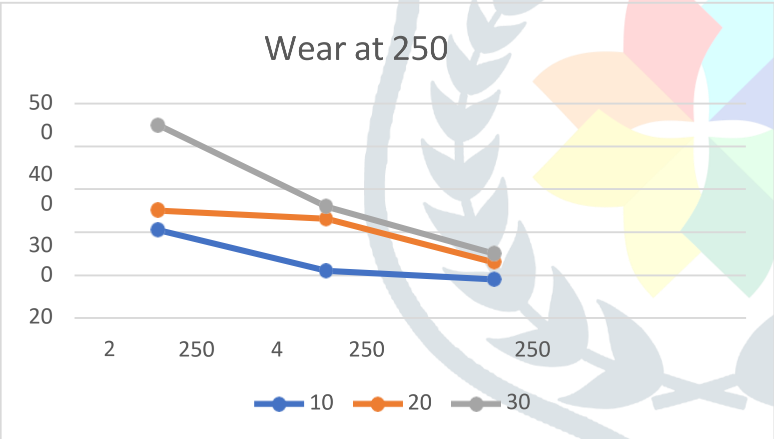

Fig 9 Variation of Wear with percentage reinforcement @ 250 RPM

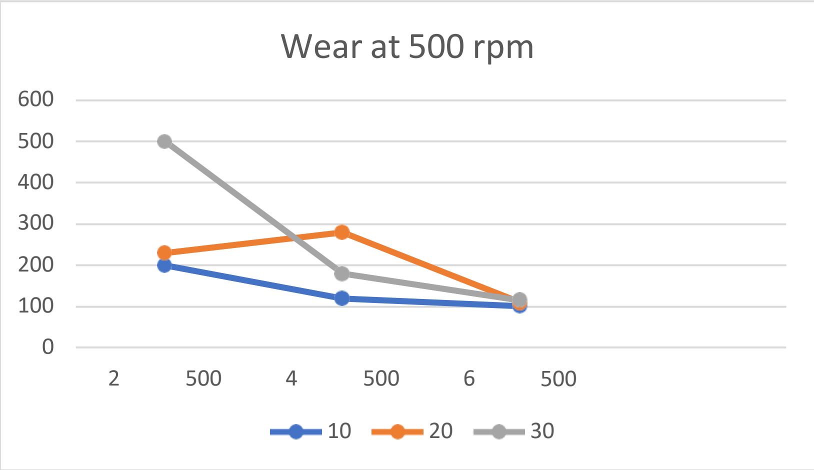

Fig 10 Variation of Wear with percentage of Reinforcement @ 500 RPM

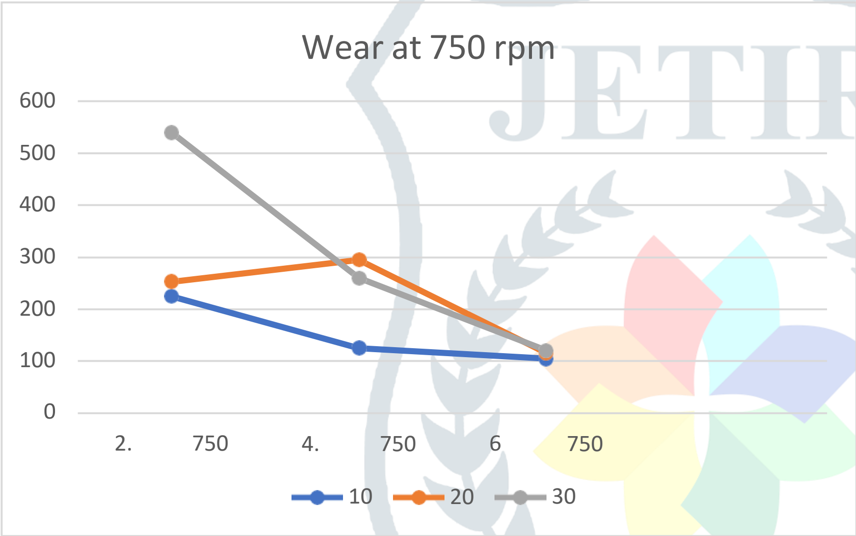

Fig 11 Variation of Wear with Percentage of Reinforcement @ 750RPM

HARDNESS:

The ASTM standard E92 was used for the Rockwell hardness test[13]. Fig 12 shows the Hardnessvalue chart. Strength, toughness, ductility, elastic stiffness, plasticity, and strain play a vital role in determining the hardness of the material. The samples of metal matrix composites had been evaluated using the Vickers Hardness Test for hardness and the results are plotted. The tests wereconducted to identify surface composite behavior for all three composites. Results of the Rockwell hardness test are shown. Sample 3 shows the highest hardness indicating that the composition of reinforcement with a volume percentage 15% AZ31B shows a higher hardness than the pure AZ31B. The addition of reinforcement has only a little impact on the hardness. The hardness of samples 5 & 10% is low compared to 15%.

Table. 7 Hardness

samples	Hardness									Avg. Hardness
	Trail 1			Trail 2			Trail 3			
5	71	68	69.8	70	69	73	67	70	67.9	**69.5**
10	76	69	70.2	70.4	73.3	69	71	73	72	**72.1**
15	77	71	70	72	73	70	72	74	73	**73.4**

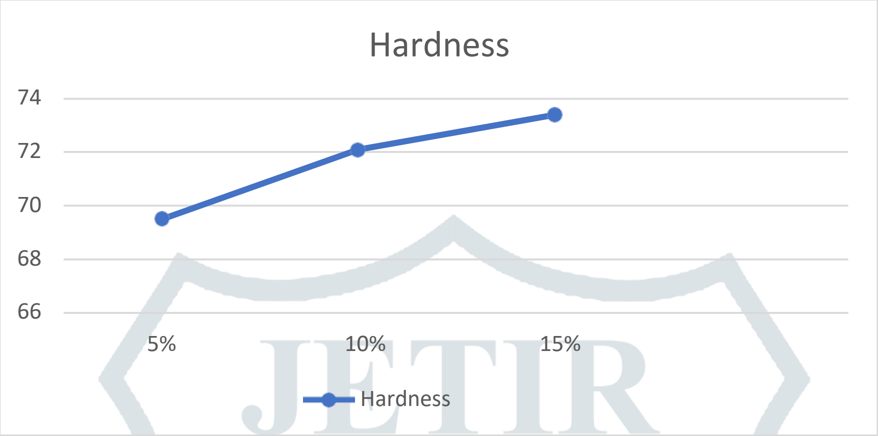

Fig 12 Hardness test on AZ31B- HAP with different percentage

OPTICAL MICROSCOPE:

The properties of composites depend on the microstructure and interface characteristics between reinforcements and matrix. Fig 13 shows an optical microscope.Fig.14-20 shows the optical microstructures of 5, 10, and 15 wt. % HAP reinforced MMCs respectively. From microstructural analysis, clustering and non-homogeneous distribution of HAP particles in the Mg matrix were observed. This was due to thevariation of contact time between HAP particles and molten Mg during composite processing, high surface tension, and poor wetting behavior of HAP particles in the liquid Mg. Non-homogenization of HAP particles in the Mg matrix can be observed in the microstructure of 15 wt. percent HAP-reinforced MMC as shown in Fig 19 Therefore increasing the weight percentage of HAP particles increases entrapped air resulting in a higher amount of porosity The microstructures reveal that HAP particles were distributed uniformly. Due to Severe plasticdeformation occurs during the stir casting process technique some deformation of grain structure occurs in developed surface composites.

Fig 13 Optical Microscope

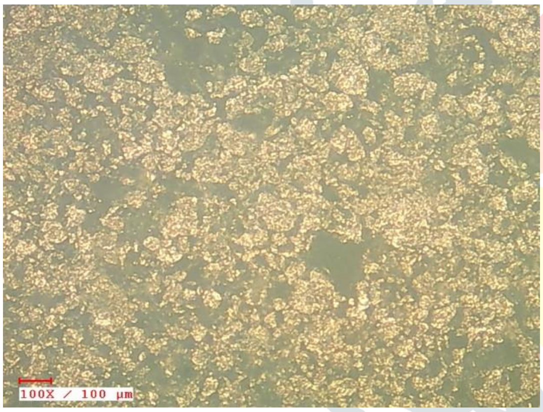

Fig 14 HAP 5% at 100X

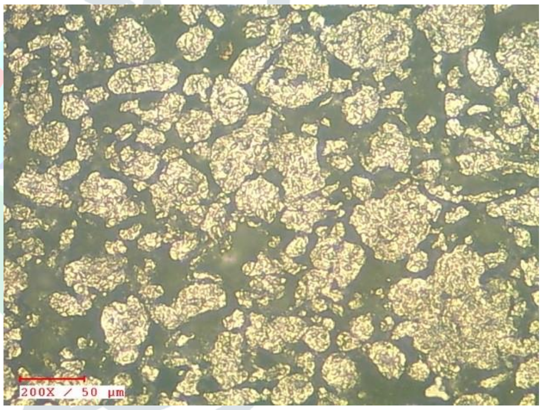

Fig 15 HAP 5% at 200X

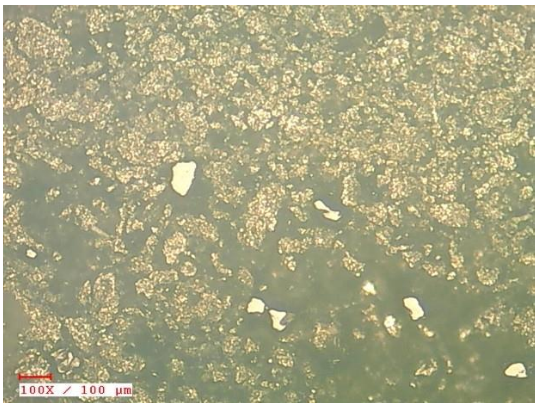

Fig 16 HAP 10% at 100X

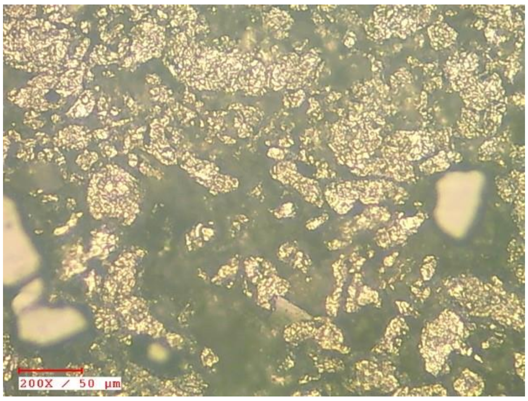

Fig 17 HAP 10% at 200X

Fig 18 HAP 15% at 100X

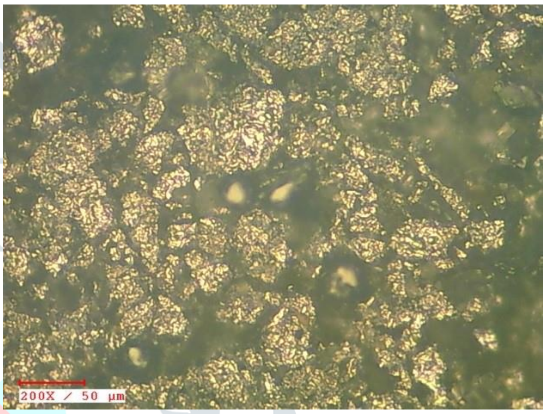

Fig 19 HAP 15% at 200X

Fig 20 HAP 5% at 50X

Fig 21 HAP 5% at 200X

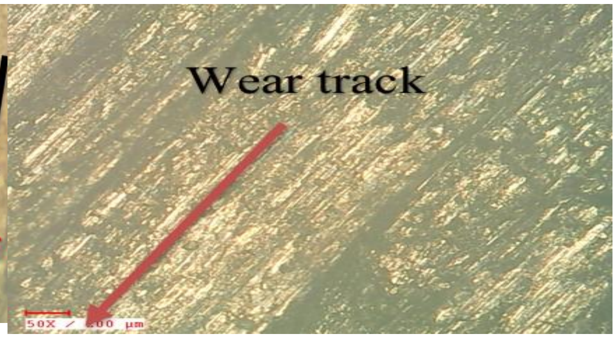

Fig 22 HAP 10% at 50X

Fig 23 HAP 10% at 200X

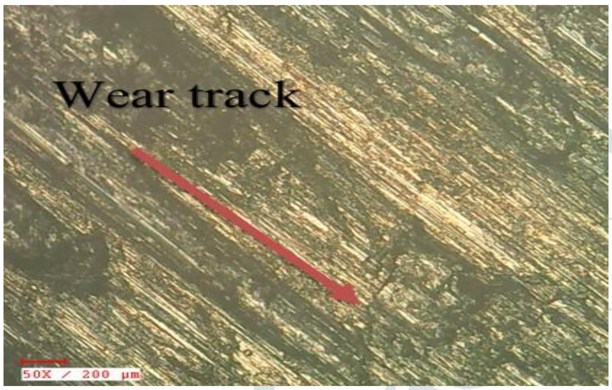

Fig 24 HAP 15% at 50X

Fig 25 HAP 15% at 200X

Fig. 20-25 shows the Optical micrographs of an AZ31B with 5, 10, 15. Cracks are observed significantly at 200 magnification and wear tracks are also visible. Grain size is cleared atsome magnification. The worn surface of the various composites is tested under sliding conditions whichare analysed using Optical microscopy[14].As the load and sliding velocity increase the wearrate increases due to severe plastic deformation which can be identified from the flake layer formation over the surface. Optical microscopy testing has been done on the samples.

As the reinforcement is increased the worn surface is dominated by abrasive wear[15],since the contact area between the sliding surface becomes minimal due to the exposure of hard particles during sliding, there is a drastic decrease in the wear rate of the composite compared to the specimen having no reinforcement.

CONCLUSIONS:

Pin-on-disc dry sliding wear tests with pins of Magnesium alloy composite were investigated against a steel counter face in the load range of 10-30N with RPM 250,500,750 etc. The mainconclusions of the investigation are as follows:

- The hardness value of sample 3 (AZ31B with 15% Hydroxyapatite, 107.6HV) is higher than all the samples,the hardness of samples 1 i.e., (AZ31B with 5% Hydroxyapatite, 74.83HV) and 2(AZ31B with 10% Hydroxyapatite, 89.1HV) is low as compared with the sample 3(AZ31B with 15% Hydroxyapatite).
- Percentage of Reinforcement effects more significantly on the wear, contributing 53.71% concerning all parameters. Next to the percentage of Reinforcement, load with 9.35 % has a major contribution in wear.
- RPM has a significant contribution with 34.44%, it has less effect on friction. Load also plays an important

role next to the RPM with 24.99%.

- To get minimum friction on AZ31B with Hydroxyapatite. The optimum parameters to be taken are RPM is 250, Load is 10 N, & Percentage of Reinforcement is 15%.
- Stir-casting process parameters-speed of the stirrer, stirring time, stirrer blade design, reinforcement size, and melt temperature play a significant role in AZ31B composite characteristics. Optimization can be done to achieve suitable parameters.

REFERENCE

[1] V. Tsakiris, C. Tardei, and F. M. Clicinschi, "Biodegradable Mg alloys for orthopedic implants–A review," *Journal of Magnesium and Alloys,* vol. 9, no. 6, pp. 1884-1905, 2021.

[2] S. Agarwal, J. Curtin, B. Duffy, and S. Jaiswal, "Biodegradable magnesium alloys for orthopaedic applications: A review on corrosion, biocompatibility and surface modifications," *Materials Science and Engineering: C,* vol. 68, pp. 948-963, 2016.

[3] A. Luo, "Processing, microstructure, and mechanical behavior of cast magnesium metal matrix composites," *Metallurgical and Materials Transactions A,* vol. 26, pp. 2445-2455, 1995.

[4] D. Miracle, "Metal matrix composites–from science to technological significance," *Composites science and technology,* vol. 65, no. 15-16, pp. 2526-2540, 2005.

[5] L. Singh, B. Singh, and K. K. Saxena, "Manufacturing techniques for metal matrix composites (MMC): an overview," *Advances in Materials and Processing Technologies,* vol. 6, no. 2, pp. 441-457, 2020.

[6] M. Y. Khalid, R. Umer, and K. A. Khan, "Review of recent trends and developments in aluminium 7075 alloys and metal matrix composites (MMCs) for aircraft applications," *Results in Engineering,* p. 101372, 2023.

[7] E. Ghali, W. Dietzel, and K.-U. Kainer, "General and localized corrosion of magnesium alloys: a critical review," *Journal of materials engineering and performance,* vol. 13, pp. 7-23, 2004.

[8] W. Ferrando, "Review of corrosion and corrosion control of magnesium alloys and composites," *Journal of Materials Engineering,* vol. 11, no. 4, pp. 299-313, 1989.

[9] H. Hu, X. Nie, and Y. Ma, "Corrosion and surface treatment of magnesium alloys," *Magnesium Alloys-Properties in Solid and Liquid States,* pp. 67-108, 2014.

[10] L. Zhang, W. Harrison, M. A. Yar, S. G. Brown, and N. P. Lavery, "The development of miniature tensile specimens with non-standard aspect and slimness ratios for rapid alloy prototyping processes," *journal of materials research and technology,* vol. 15, pp. 1830-1843, 2021.

[11] I. Murray and A. Tarantino, "Mechanisms of failure in saturated and unsaturated clayey geomaterials subjected to (total) tensile stress," *Géotechnique,* vol. 69, no. 8, pp. 701-712, 2019.

[12] J. An *et al.*, "Dry sliding wear behavior of magnesium alloys," *Wear,* vol. 265, no. 1-2, pp. 97-104, 2008.

[13] D. A. College, "Analysis of Spherical Indenter Rockwell Superficial Hardness Tests on Thin Annealed Low-Carbon Steel," *Journal of Materials Engineering and Performance,* vol. 32, no. 7, pp. 2892-2905, 2023.

[14] J. Arsecularatne, N. Chung, and M. Hoffman, "An in vitro study of the wear behaviour of dental composites," *Biosurface and Biotribology,* vol. 2, no. 3, pp. 102-113, 2016.

[15] S. Das, D. Mondal, S. Sawla, and N. Ramakrishnan, "Synergic effect of reinforcement and heat treatment on the two body abrasive wear of an Al–Si alloy under varying loads and abrasive sizes," *Wear,* vol. 264, no. 1-2, pp. 47-59, 2008.