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Synopsis Of

DRY SLIDING AND FRETTING WEAR BEHAVIOUR OF CaO REINFORCED MAGNESIUM NANOCOMPOSITES

A Thesis

To be submitted by

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For the award of the degree

Of

DOCTOR OF PHILOSOPHY

Abstract

This study reported the dry sliding tribological behaviour of pure magnesium and magnesium / Calcium oxide (CaO) nanocomposites with four different amount of weight percentages produced using disintegrating melt deposition (DMD) Process. Pin-on-disk setup was used to perform the experiments with four distinct load and sliding velocity ranges. At lower loads and sliding velocity of 5 N, 8N at 0.6 m/s pure magnesium exhibits lower wear rate than nanocomposites. The influence of design parameters on wear loss is reported through the Response Surface Methodology (RSM). ANOVA was used to confirm the soundness of the developed regression equation. The results indicate the contribution of linear, quadratic, and interaction terms of design parameters on response. 3D response surface and 2D contour plots are indicated the interaction effect. The result shows that an increase in sliding velocity contributes to a decrease in the wear loss of the composites because of the emergence of protective oxidative layer at the surfaces of the pins, which is confirmed through FESEM and EDAX analysis of the pin surfaces. Wear loss of the material decreased as amount of CaO increased. The ANOVA analysis concluded that the sliding distance and load contribute significantly to wear loss of the composites and their percentage of contribution is 64.02 and 3.69 percentages.

1 Introduction

In the past decades, raid changes occur in environmental and increase pressure in legislative onto the automobile sector necessiates to make lighter, good performance vehicles with greater fuel efficiency resulted the utilization of magnesium materials Mordike and Ebert (2001). Wear can be defined as a progressive loss of material due to the relative motion between the contacting surfaces or substances. It is an unavoidable and significantly serious concern in several industrial sectors Lim *et al.* (2005). The wear damage can be minimized by using materials with enhanced mechanical properties. Though magnesium based materials offer an extensive enhanced properties and merits their tribological behaviour much have not been explored in detail as compared to aluminium based materials Ye and Liu (2004). Wear phenomenon is an important consideration when magnesium based materials are being utilized in automobile components. One of the huge component used in the automobile sector is engine components which takes up of about 30 percentage of the whole weight of the vehicle. Hence the reduction in weight directly imcreases the fuel efficiency Pekguleryuz and Kaya (2003).When two metallic surfaces are in contact under static or dynamic loading, the loading surfaces adhere together due to the solid state welding of asperities and subsequent detachment of the contact surfaces or asperities will results in loss of material. Such material loss in termed as adhesive wear or dry sliding wear (ASM Handbook, 1992). At high operating temperatures, adhesion is the main cause of material failiure during sliding and galling wear because the tendency to adhesion increases exponentially with increasing temperature Kaviti *et al.* (2018). Several factors influencing the dry sliding dry sliding wear of the magnesium nanocomposites they are i) metallurgical factors such as chemical composition, they type of phases present, volume of percentage of precipitates ii) service factors such as contact pressure, temperature, relative velocity of the sliding surfaces, sliding distance / duration Zhang *et al.* (2018). The enhanced tribological characteristics are essential on those parts. With respect to the relative motion between the mating component surface damage encounters in several ways like fretting, seizure, adhesion and so on Gaol and Webb (2014). Moreover, sliding wear under dry sliding is a prime importance in processing of materials through rolling, forging and extrusion. In addition to the above discussion, the study of fretting wear behaviour in magnesium nanocomposites are very crucial in the field of biomedical. Fretting is observed when the contacting bodies are subjected to micro motion produced through vibration. Fretting wear produces localized surface damage at the mating interface it can be seen in a joint's circumference. Fretting also produces detrimental effects in bio-implants. The deterioration caused by fretting are classified into debris, pits, metal transfer, scratches and subsurface cracks.

2 Research Gap

In the findings of prominent contestant for light weight concept, a plenty of materials are presently available for structural and non-structural applications. Even though the adoption of polymer based composites and engineering plastics in aircraft applications have increased, the utilization of metallic alloys also a prime competitor for these light weight applications owing to their extreme mechanical damages and low processing cost. Composites have been identified as an effective alternative to the traditionally used materials. Genrally, composite materials composed of two phases one is matrix phase and another one is reinforcing phase which are binded with each other. Depending upon the base material that is matrix phase it has been classified into three main categories namely, metal matrix composites (MMCs), polymer matrix composites (PMCs) and ceramic matrix composites (CMCs).

Generally, the magnesium composites are produced through conventional casting process similar to the fabrication of aluminium based composites. The main difficulties to be overcome while processing is to ensure the homogeneous distribution of reinforcements to get desirable mechanical properties. The final properties and performance of the composite primarily depends upon the processing route and combination of reinforcement. Both the solid state and liquid state synthesis route has been used to produce magnesium composites.

3 Objectives

The key objective of this study is to explore the dry sliding and fretting wear behaviour of the magnesium nanocomposites against oil hardened non shrinking steel (OHNS). Besides this, the following points outline other objectives of the present work.

- To study the dry sliding wear behaviour of the composites at various conditions and to examine the sliding induced surface and sub-surface.
- To study the fretting wear behaviour of the composites subjected to various loads under constant slip amplitude and frequency of motion
- To conduct the design of experiment study to infer the significant parameter plays a vital role in dry sliding wear behaviour.

4 Methodology

In the current study, pure magnesium material has been used as a matrix. Four different weight percentages of 0.5, 1.0, 1.5 and 2.0 of calciumoxide (CaO) nanoparticles of 40 nanometer diameter calcium oxide powder were used as the prime reinforcements in the first part of the study to produce Mg/CaO nanocomposites using disintegrated melt deposition which is part of liquid state process. For a synthesis of nanocomposites extrusion was used as a secondary processing after casting the composites. The extrusion process was used here to achieve minimum amount of porosity.

4.1 Materials

Commercially pure magnesium material in the shape of turnings acquired from Acros Organics, USA and ceramic phase of calciumoxide nano partilces of 40 nm in diameter acquired from nanoshell, USA were used to synthesize Mg/CaO nanocomposites.

4.2 Primary Processing

Pure magnesium and its nanocomposites were synthesized using the liquid state processing. The farication of composites done using the disintegrated melt deposition method. To assist the process, pure magnesium turnings and calcium oxide nanoparticle were heated in an inert argon environment around 750 °C. After reaching that temperature, the melt was mixed using a twin-blade mild steel impeller coated with Zirtex 25 to remove any contaminants from the melt. Following mixing, the melt is poured into the mould using gravity. The melt is fragmented before entering the mould using two jets of argon gas, which is typically injected into the molten metal and the ingot was made with the diameter of 40 mm.

4.3 Secondary Processing

The ingots were machined into 36 mm diameter and 45 mm length. To achieve homogenisation the billets were kept at a constant temperature of about 400 C for 60 minutes in a resistance furnace. Billets were extruded using a hydraulic press of 150 ton at a temperature of 350 C with an extrusion ratio of 20.30:1 to obtain 8 mm diameter rod.

4.4 Density Measurement

The Mg/CaO nanocomposites density were measured by following Archimedes principle. The machined samples were weight measured in air as well as distiled water and the weighing balance machine with an accuracy of 0.0001 g were utilized to measure the mass of the sample. Mixture rule of composite is used to calculate the theoretical density of the composite. The difference between experimentally measured and theoretical densities were refereed as a porous amount of the samples.

4.5 Grain size Analysis

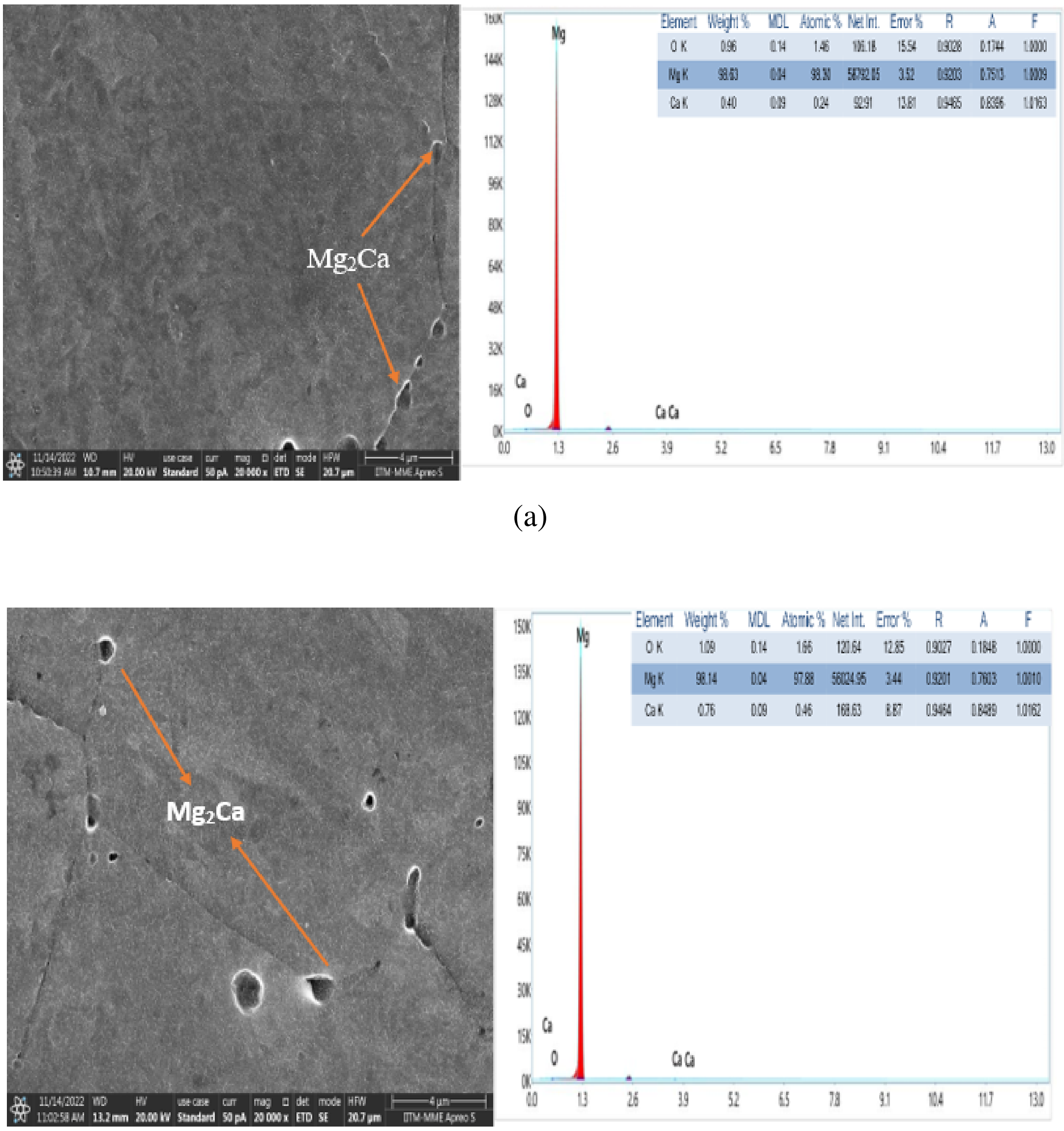
In order to reveal the grain size of the composite, the samples were polished to achieve mirror finish and properly etched with (60 ml ethylene glycol, 1 ml nitric acid, 20 ml acetic acid and 20 ml distiled water) using a Zeiss MC43 optical digital microscope. Image J software used to measure the grain size of the composites as shown in table.1.

Table 1: Grain size of Pure Mg and Mg Nanocomposites

Sl.No	Weight Percentage of Reinforcement	Grain Size (μm)
1.	Pure Mg	23
2.	0.5 wt%-CaO	5.2 ± 1.2
3.	1.0 wt%-CaO	8.3 ± 1.6
4.	1.5 wt%-CaO	8.4 ± 2.1
5.	2.0 wt%-CaO	6.6 ± 1.6

5 Microstructural Analysis

Figure.(1 (a-d)) shows the microstructure of pure magnesium in an unetched condition. This micrograph is purposely placed here to compare the effect of addition of CaO nanoparticles on microstructural development. It is observed that the CaO nanoparticles uniformly distributed in the case of 0.5 weight percentage reinforcement. As the weight percentage of CaO nanoparticles increased from 0.5 to 2.0 more amount of particles are observed at grain boundaries. This results in agglomeration of particles for higher concentration of CaO in nanocomposites.

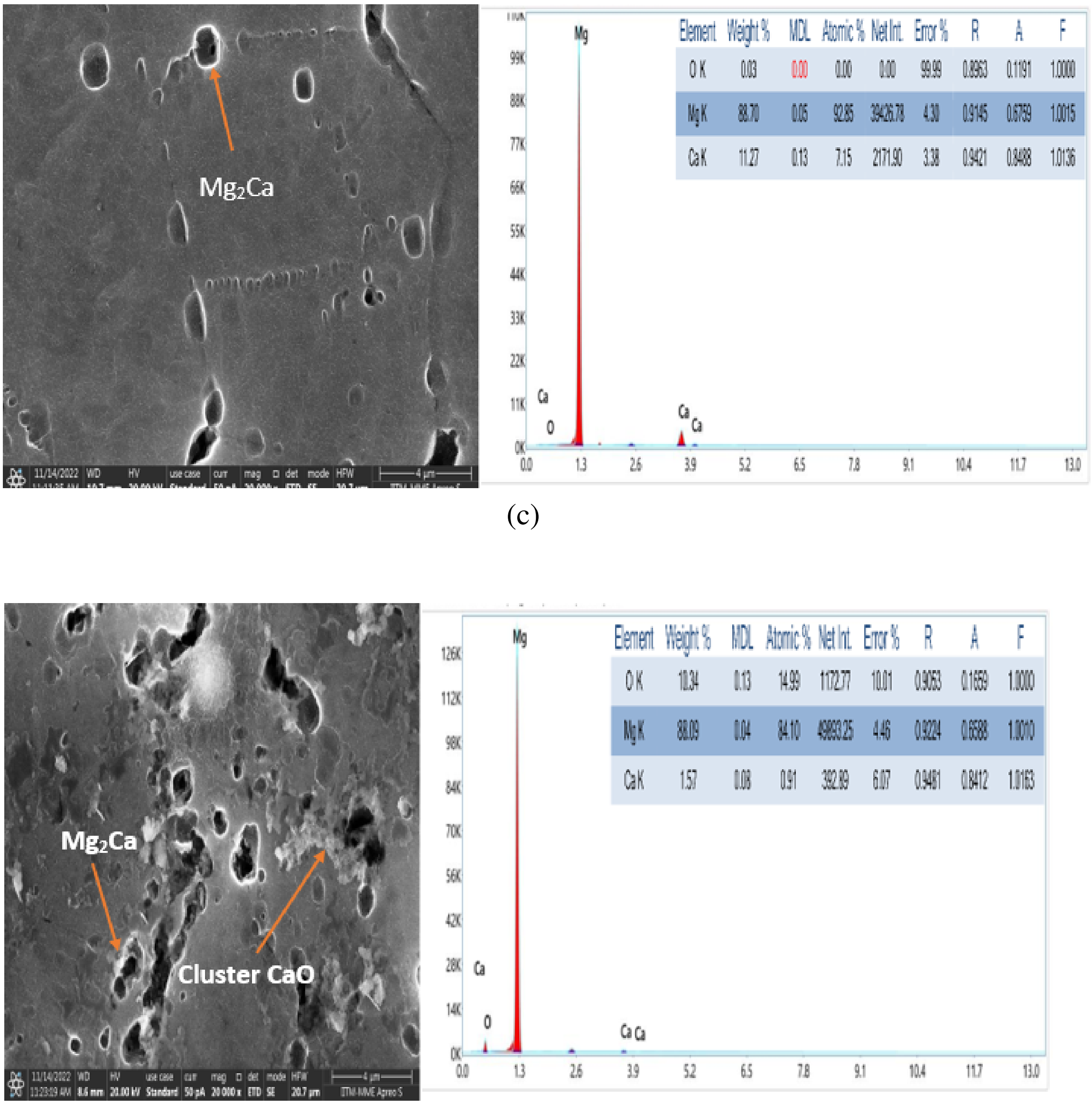


Element	Weight %	MDL	Atomic %	Net Int.	Error %	R	A	F
O K	0.96	0.14	1.46	106.18	15.54	0.9028	0.1744	1.0000
Mg K	98.63	0.04	98.30	56792.05	3.52	0.9203	0.7513	1.0009
Ca K	0.40	0.09	0.24	92.91	13.81	0.9465	0.8396	1.0163

(a)

Element	Weight %	MDL	Atomic %	Net Int.	Error %	R	A	F
O K	1.09	0.14	1.66	120.64	12.85	0.9027	0.1848	1.0000
Mg K	98.14	0.04	97.88	56024.95	3.44	0.9201	0.7603	1.0010
Ca K	0.76	0.09	0.46	168.63	8.87	0.9464	0.8489	1.0162

(b)



Element	Weight %	MDL	Atomic %	Net Int.	Error %	R	A	F
O K	0.03	0.00	0.00	0.00	99.99	0.8963	0.1191	1.0000
Mg K	88.70	0.05	92.85	39426.78	4.30	0.9145	0.6759	1.0015
Ca K	11.27	0.13	7.15	2171.90	3.38	0.9421	0.8488	1.0136

(c)

Element	Weight %	MDL	Atomic %	Net Int.	Error %	R	A	F
O K	10.34	0.13	14.99	1172.77	10.01	0.9053	0.1659	1.0000
Mg K	88.09	0.04	84.10	49893.25	4.46	0.9224	0.6588	1.0010
Ca K	1.57	0.08	0.91	392.89	6.07	0.9481	0.8412	1.0163

(d)

Figure 1: SEM image of Magnesium Nanocomposites a) 0.5 b) 1.0 c) 1.5 d) 2.0 weight percentages of CaO

6 Development of correlation between wear loss and operating parameters

Response surface methodology is the most commonly used mathematical method for modeling, designing, and evaluating problems/parameters when more than two input variables affect process output response, giving a full understanding of the process within the design regime. In the current study, load, sliding velocity, percentage of CaO amount, and sliding distance were selected as input design parameters,and the wear loss in terms of mass loss is considered as a response. The experimental plan has been designed with a central composite design with all four factors at their five levels. The developed second-order quadratic equation was used to analyze the response of the process. The data normality is concluded by means of Normal probability plot. The normality of residuals data of wear loss is shown in Figure.2. From the plot it can be deduced that the maximum number of residuals were aligned on and nearest of the inclined line reveals that the errors are distributed normally. Figure.3 (a-l). represents the 3D surface plot and 2D contour plot of the design factors on the response, that is, wear loss of the composite. It is inferred from the graph that the lack of wear of the composite with an increase in sliding speed has raised the load up to 35 N. The main result of the variables, along with their association and quadratic effect, was known as the normal probability plot of the impact of the parameters, as shown in Figure.4.

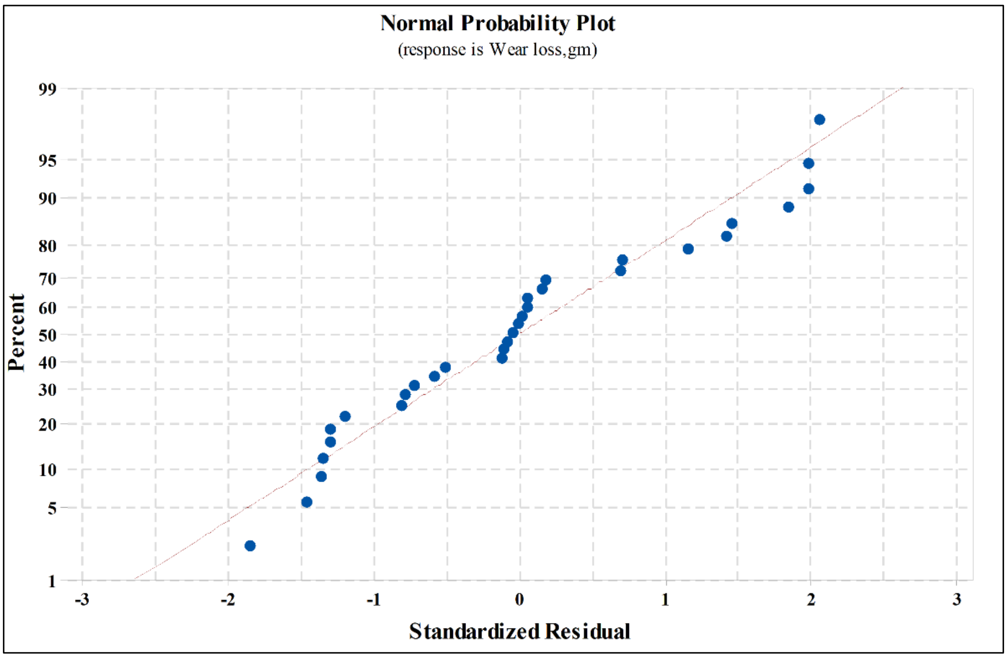
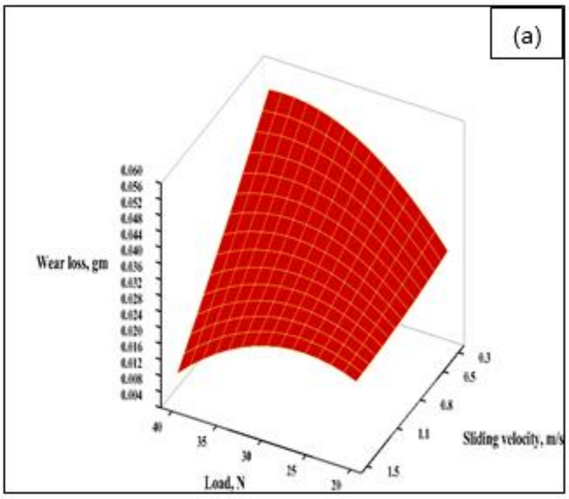
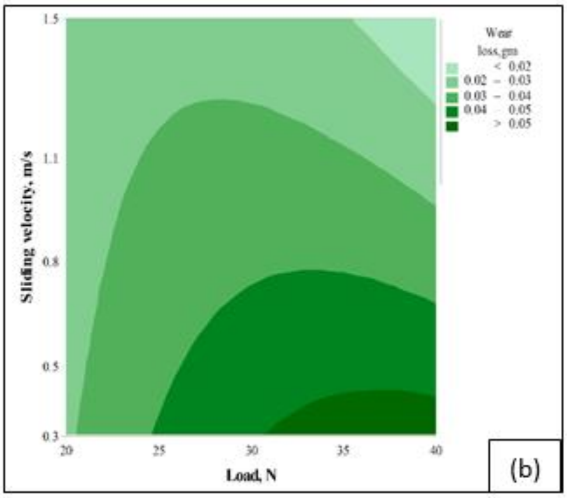
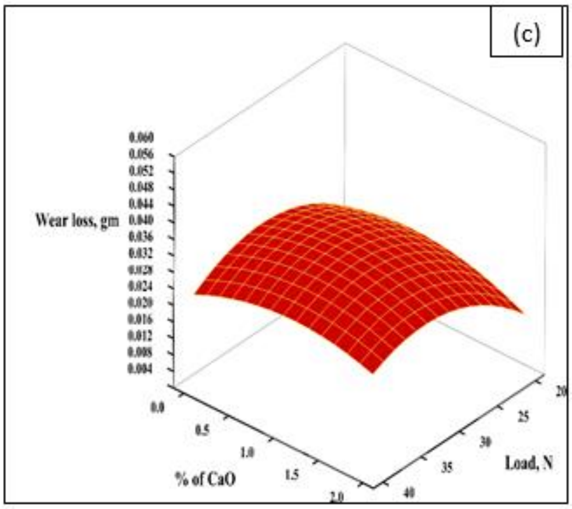
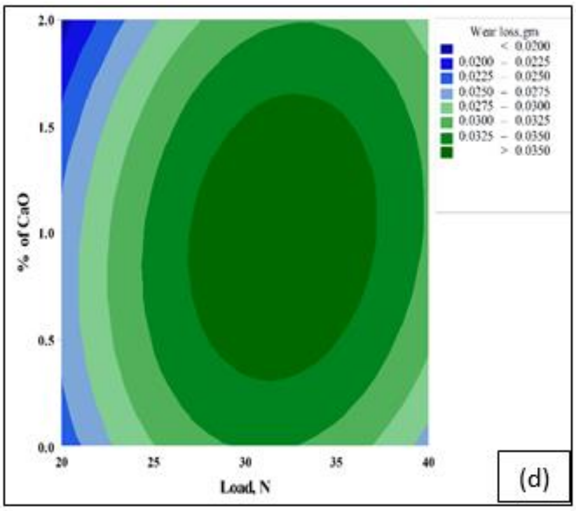


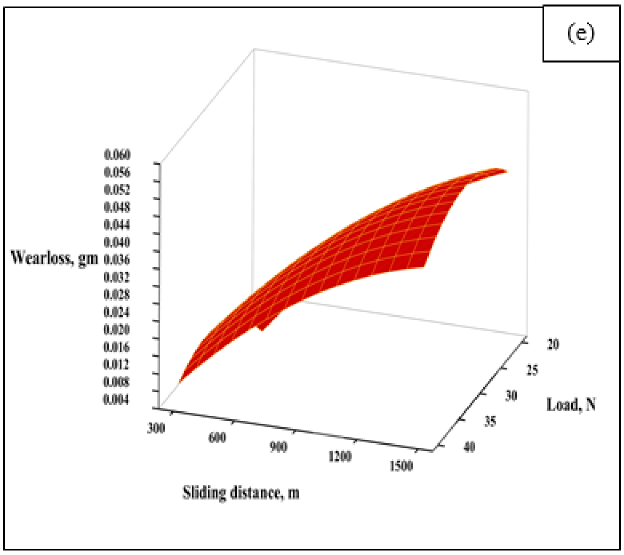
Figure 2: Normal probability plot of wear loss

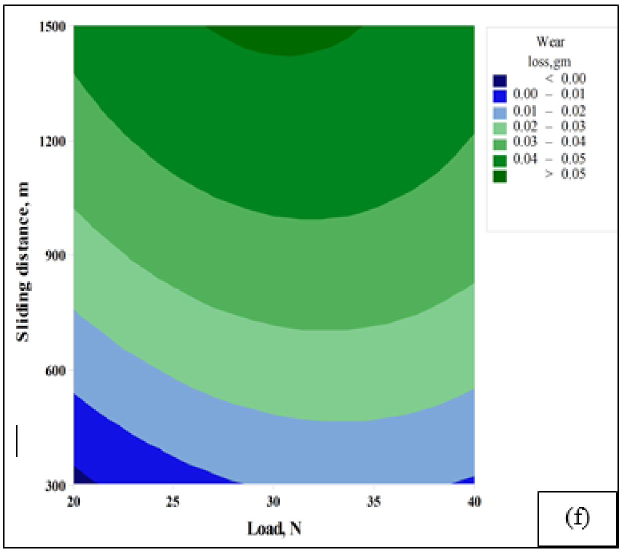

(a)
Wear loss, gm
0.060
0.056
0.052
0.048
0.044
0.040
0.036
0.032
0.028
0.024
0.020
0.016
0.012
0.008
0.004
Load, N
40
35
30
25
20
Sliding velocity, m/s
0.3
0.5
0.8
1.1
1.5

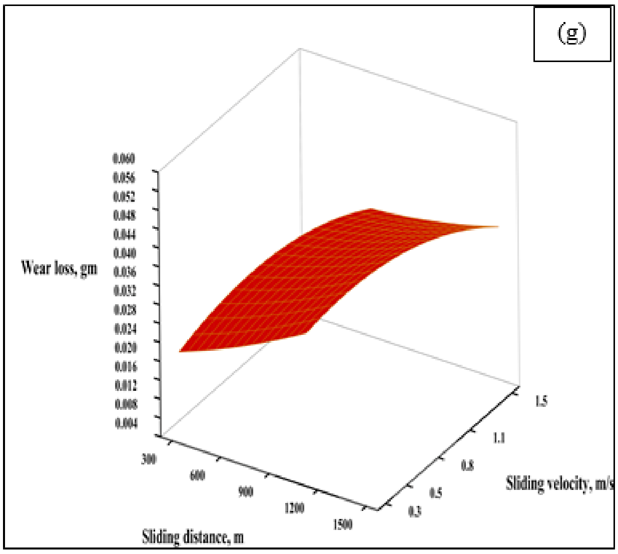

(b)
Sliding velocity, m/s
1.5
1.1
0.8
0.5
0.3
Load, N
20
25
30
35
40
Wear loss, gm
< 0.02
0.02 – 0.03
0.03 – 0.04
0.04 – 0.05
> 0.05

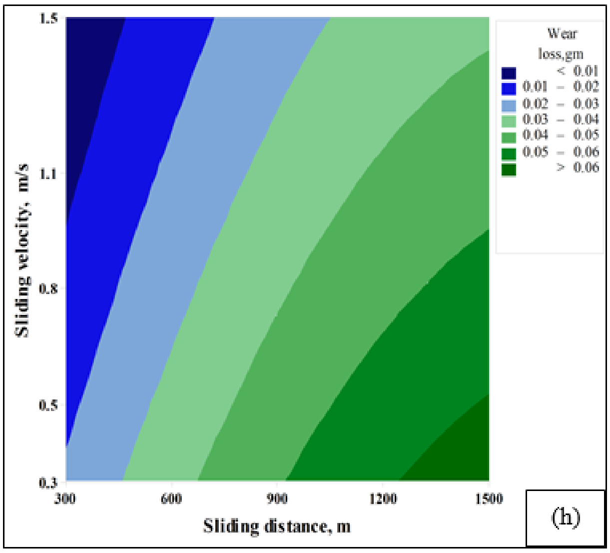

(c)
Wear loss, gm
0.060
0.056
0.052
0.048
0.044
0.040
0.036
0.032
0.028
0.024
0.020
0.016
0.012
0.008
0.004
% of CaO
0.0
0.5
1.0
1.5
2.0
Load, N
20
25
30
35
40


(d)
% of CaO
2.0
1.5
1.0
0.5
0.0
Load, N
20
25
30
35
40
Wear loss, gm
< 0.0200
0.0200 – 0.0225
0.0225 – 0.0250
0.0250 – 0.0275
0.0275 – 0.0300
0.0300 – 0.0325
0.0325 – 0.0350
> 0.0350


(e)
Wearloss, gm
0.060
0.056
0.052
0.048
0.044
0.040
0.036
0.032
0.028
0.024
0.020
0.016
0.012
0.008
0.004
300
600
900
1200
1500
Sliding distance, m
20
25
30
35
40
Load, N


1500
1200
900
600
300
Sliding distance, m
20
25
30
35
40
Load, N
Wear loss,gm
< 0.00
0.00 – 0.01
0.01 – 0.02
0.02 – 0.03
0.03 – 0.04
0.04 – 0.05
> 0.05
(f)


(g)
Wear loss, gm
0.060
0.056
0.052
0.048
0.044
0.040
0.036
0.032
0.028
0.024
0.020
0.016
0.012
0.008
0.004
300
600
900
1200
1500
Sliding distance, m
1.5
1.1
0.8
0.5
0.3
Sliding velocity, m/s


1.5
1.1
0.8
0.5
0.3
Sliding velocity, m/s
300
600
900
1200
1500
Sliding distance, m
Wear loss,gm
< 0.01
0.01 – 0.02
0.02 – 0.03
0.03 – 0.04
0.04 – 0.05
0.05 – 0.06
> 0.06
(h)

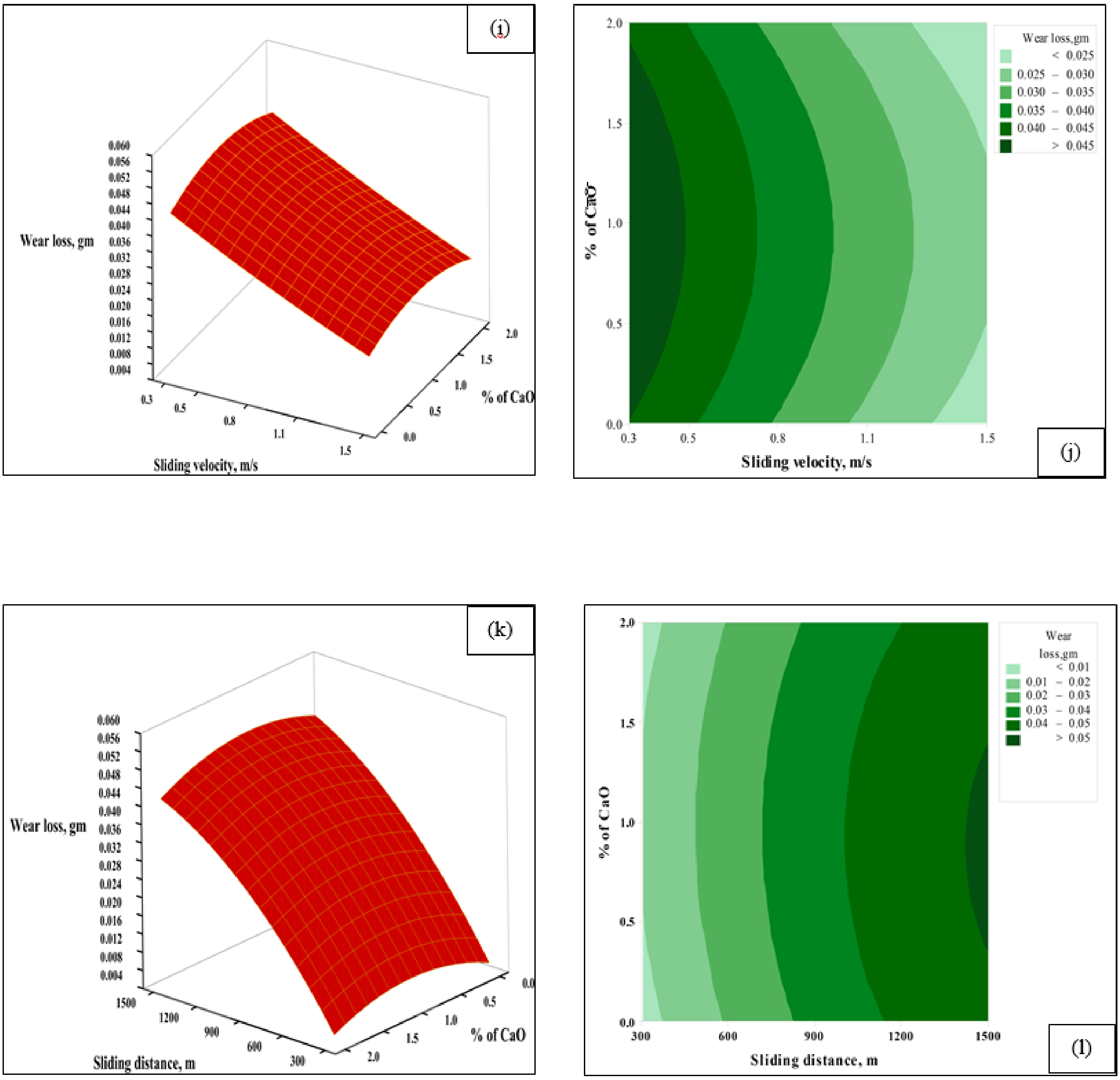


Figure 3: Two way interaction effect of wear loss in (a,c,e,g,i,k) 3D surface plot and (b,d,f,h,j,l) 2D contour plot

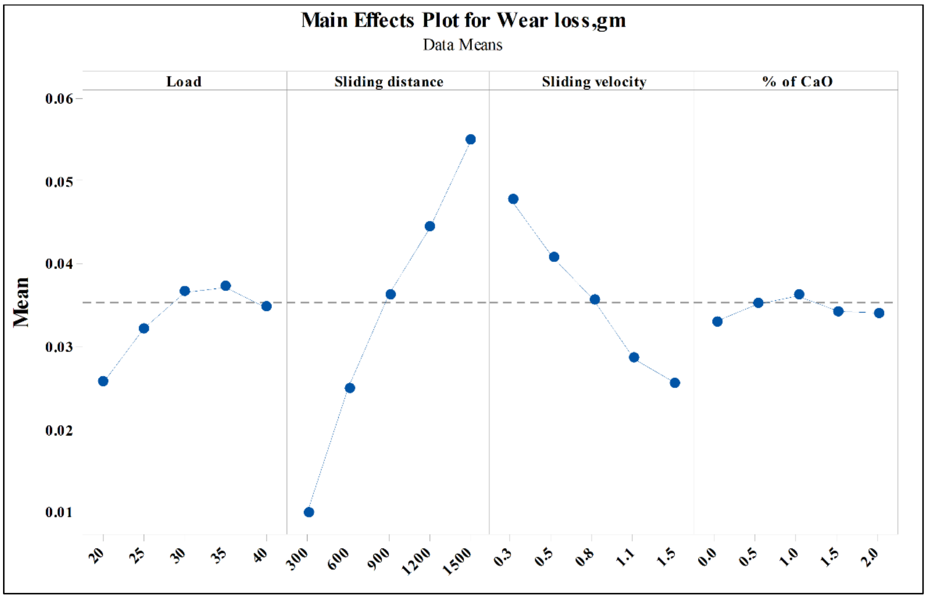
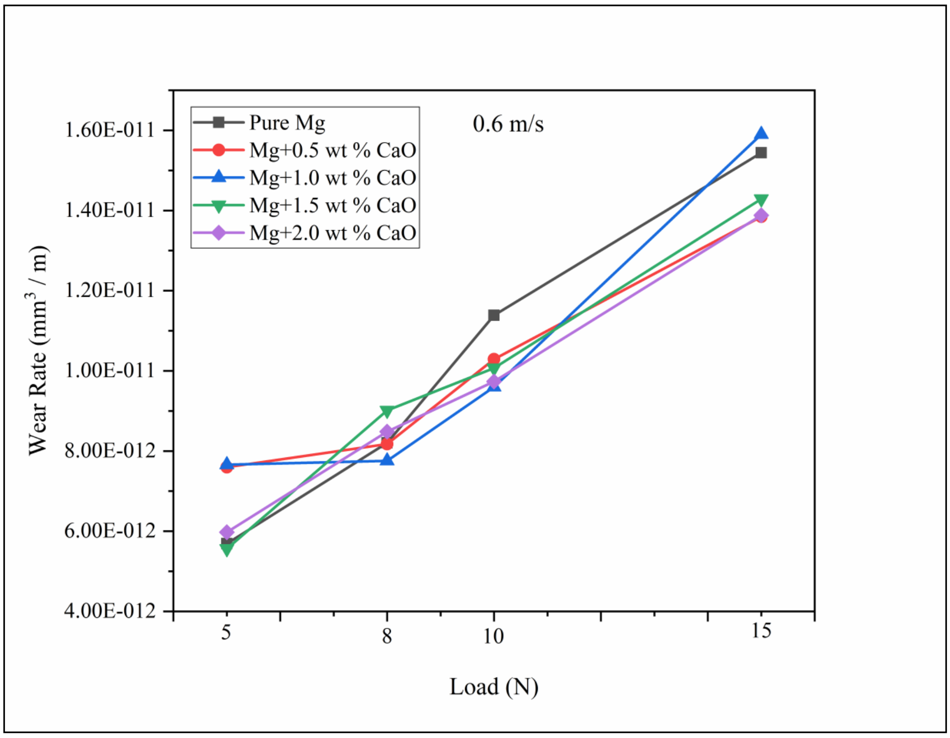


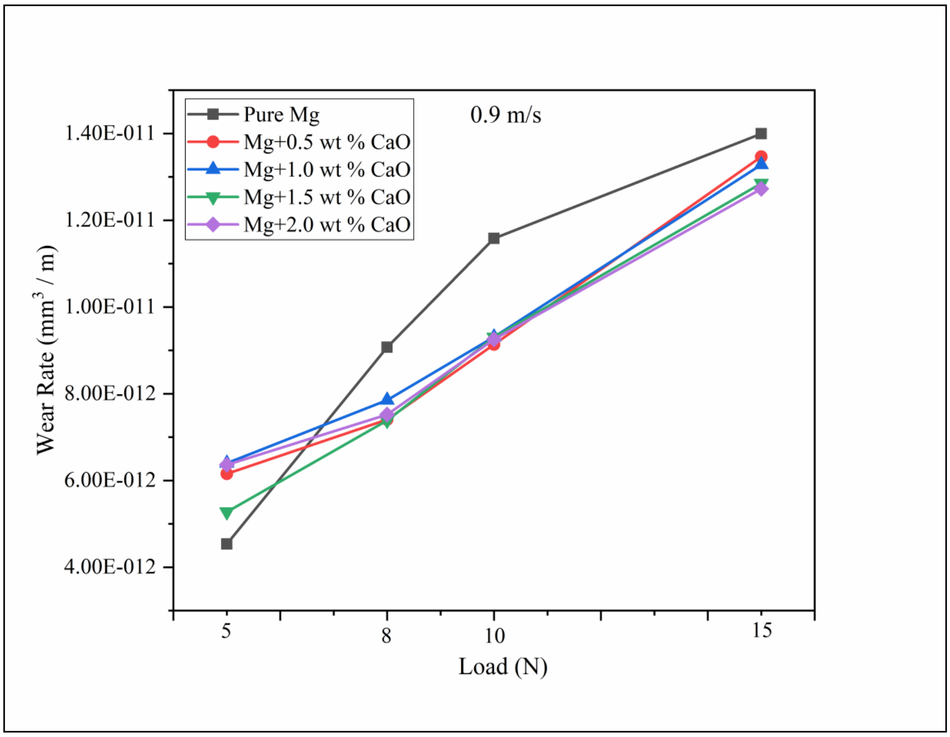
Figure 4: Main effect plot of factors affecting wear loss of the composites

7 Dry sliding wear nature of the Nanocomposites

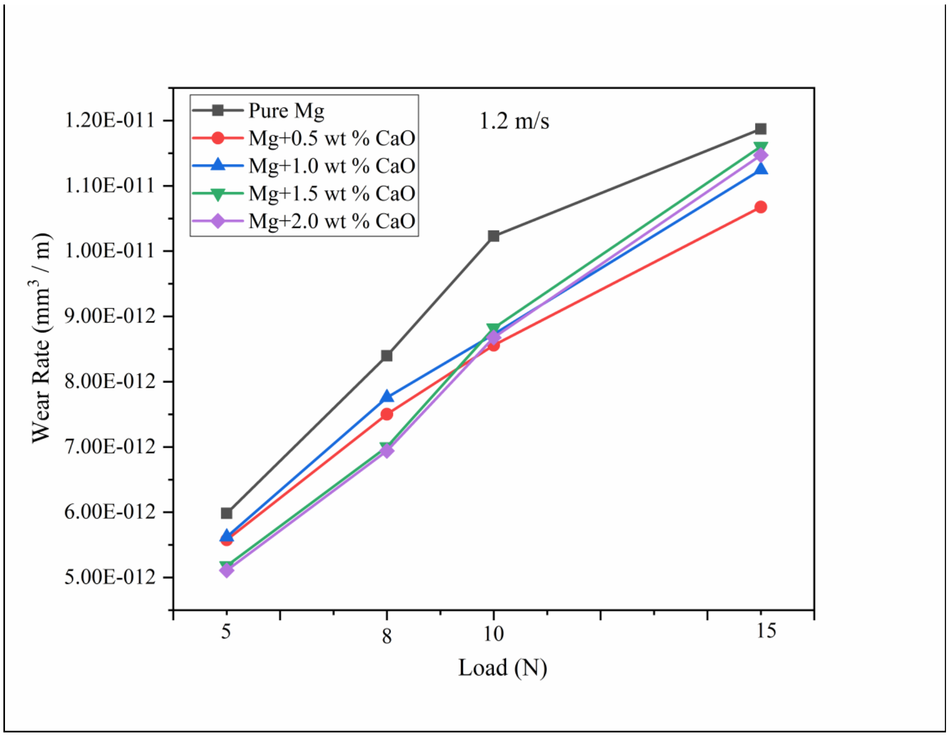
The sliding wear behaviour of the magnesium nanocomposites are performed on OHNS steel. Experimentations are performed for a defined process condition to study the wear behaviour with significant justifications.With the data acquisition system, the corresponding changes in wear, frictional force and coefficient of friction are recorded for all the test samples investigated. Four different sets of loading and sliding velocities such as 5N, 8N, 10N, 15N and 0.6 m/s, 0.9 m/s, 1.2 m/s, 1.5 m/s at a constant sliding distance of 600 m were employed. In the present study, it was observed that the wear rate of all reinforced and pure magnesium composites increases with increasing applied load for all sliding speeds as predicted by Archard's law as shown in Figure.5(a-d). Upon increasing the load from lower to higher value, the wear debris generated during sliding was retained as freely moving particles between the contacting surfaces which produces damage to both the surfaces.


0.6 m/s
Pure Mg
Mg+0.5 wt % CaO
Mg+1.0 wt % CaO
Mg+1.5 wt % CaO
Mg+2.0 wt % CaO
Wear Rate (mm^3 / m)
1.60E-011
1.40E-011
1.20E-011
1.00E-011
8.00E-012
6.00E-012
4.00E-012
5
8
10
15
Load (N)

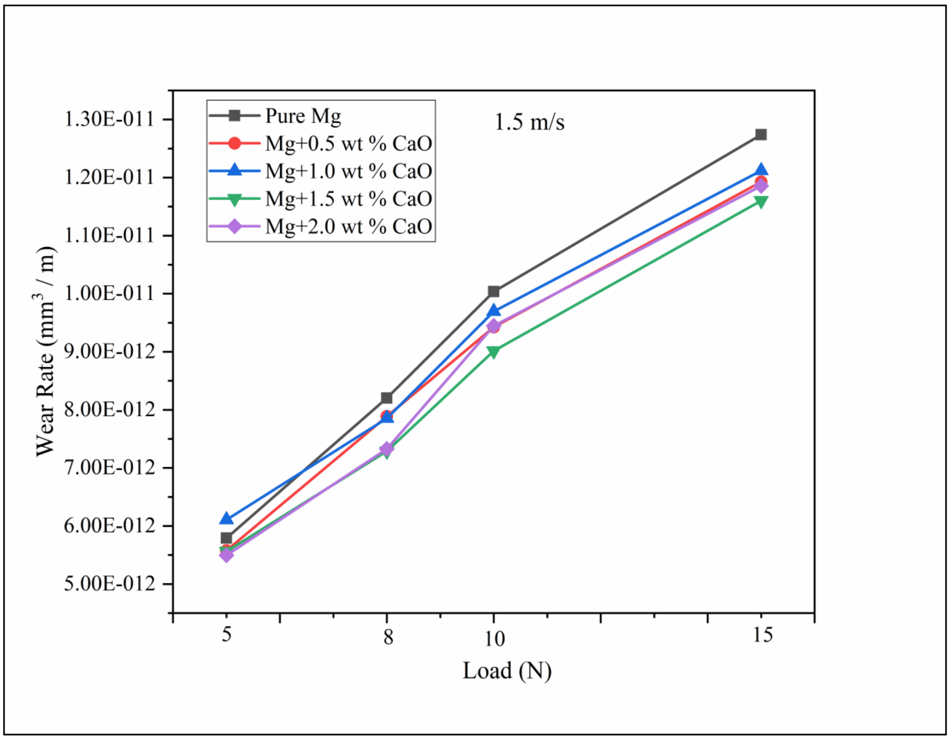
(a)


0.9 m/s
Pure Mg
Mg+0.5 wt % CaO
Mg+1.0 wt % CaO
Mg+1.5 wt % CaO
Mg+2.0 wt % CaO
Wear Rate (mm^3 / m)
1.40E-011
1.20E-011
1.00E-011
8.00E-012
6.00E-012
4.00E-012
5
8
10
15
Load (N)

(b)



(c)



(d)

Figure 5: (a-d) Wear rate as a function of load under different sliding speeds

7.1 Wear mechanisms

The different mode of wear mechanisms existed with respect to the applied load and sliding speed has been illustrated through worn surface analysis using scanning electron microscope (SEM) associated with energy dispersive x-ray spectroscopy (EDS). The worn out pin surface, wear debris and steel counter disk has been inferred at all wear testing conditions for pure magnesium and nanocomposites individually and the comparative study has also been reported at the end of the section.

Form the obtained experimental results, it is to be concluded that there are three predominant wear regimes persisted depending upon the testing conditions involved for pure magnesium and magnesium nanocomposite variants. They are a) low wear, b) mild wear and c) severe wear as shown in Figure.6. Further section covers a discussion about wear mechanisms which influences the wear rate within these regions.

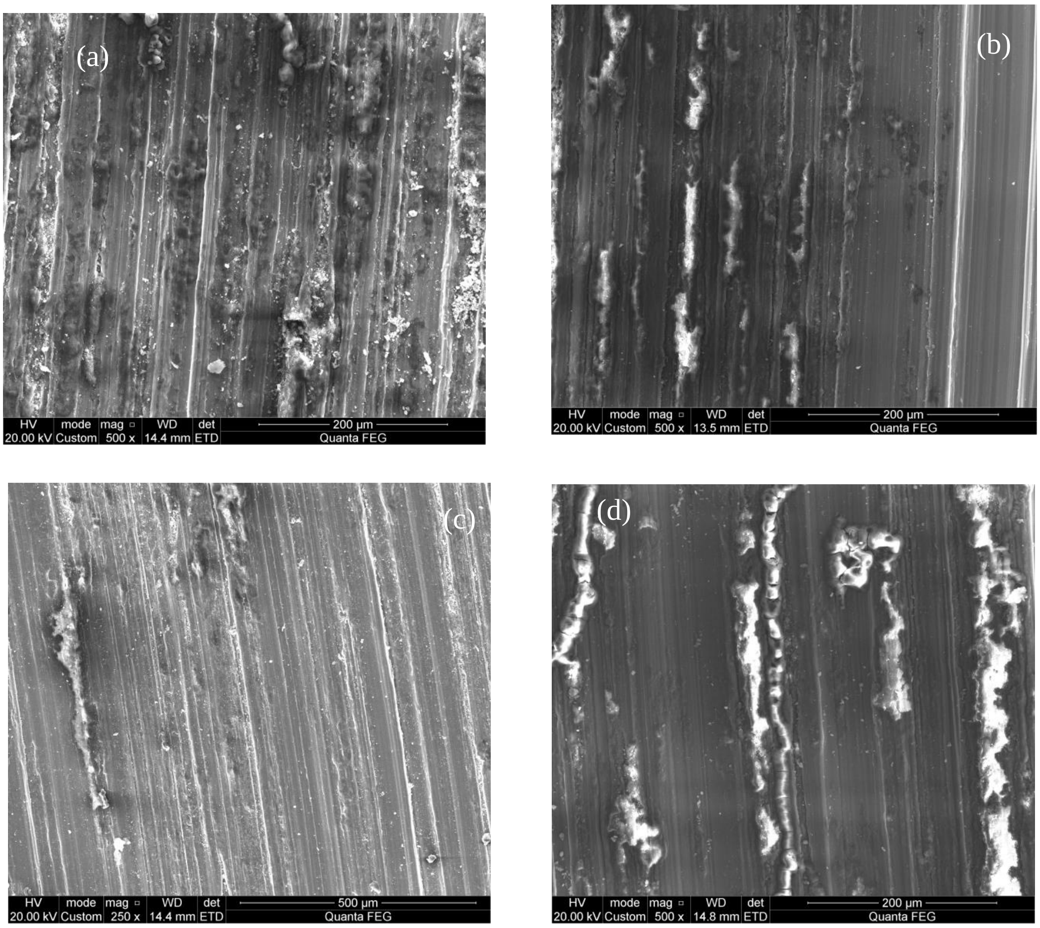


Figure 6: SEM images of the pure Mg pin tested at 0.6 m/s a)5N, b)8N, c)10N, d)15N

8 Fretting wear

In this study, Mg pin with flat end and steel plate has been used to conduct the fretting wear experiments are described here. The plate sample used at bottom was encountered with oscillations due to fretting and the normal load at the interface is regulated by the pin. Disintegrated Melt Deposition processed pure Mg and Mg composites reinforced with different amount of CaO nanoparticles were used as a pin material and commercially available EN31 steel plate were produced as 40*40 mm cross section with 5 mm thickness plate used as a counter material.Wire cut electrical discharge machining (EDM) was used to produce the plate with above mentioned dimensions. The pin material was machined to 6 mm in diameter with 40 mm length with flat end configuration with conventional CNC machining operation.

8.1 Characteristics of Displacement loops

Figure 7.shows the fretting hysterisis loops generted for composite variants after 10000 cycles in air environment. The area of hysterisis loop represents the frictional energy dissipated during fretting process. In general, larger area of the loop indicates more energy dissipation in the form of friction at the contact. We can observe that the loop pattern changes as the load increases. This phenomenon represents the shift in fretting regimes.

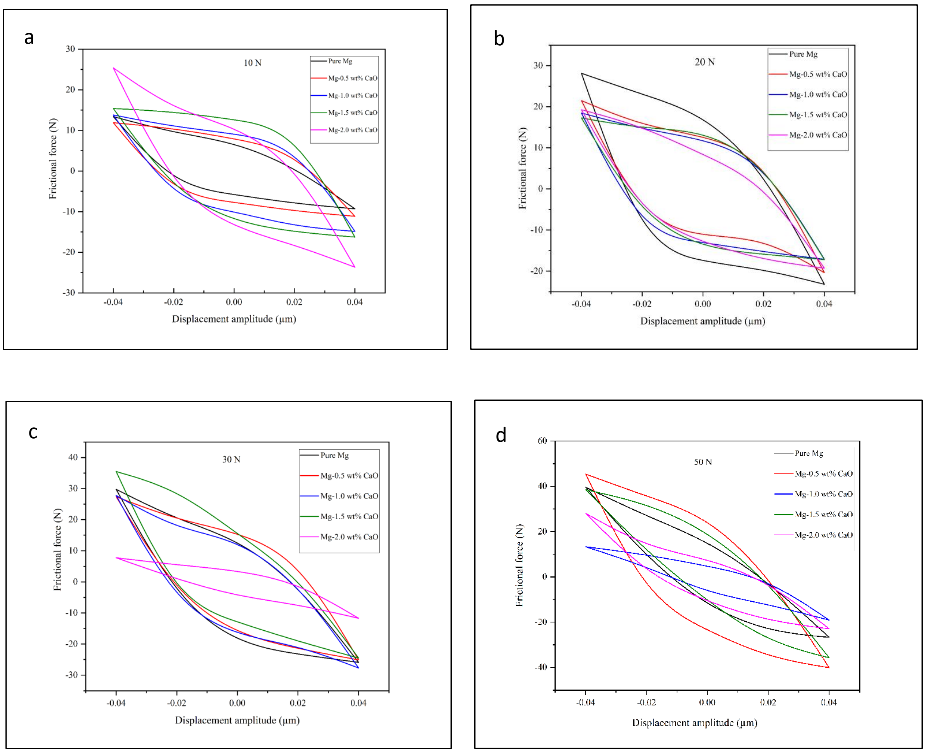


Figure 7: Friction force versus displacement loops a)10N, b)20N, c)30N and d)50N

8.2 Coefficient of friction

Figure 8. represenst the CoF variations through out the entire cycles. At lower load of 10 N Mg-2.0 wt.percentage CaO reinforced composite exhibits higher friction coefficient.

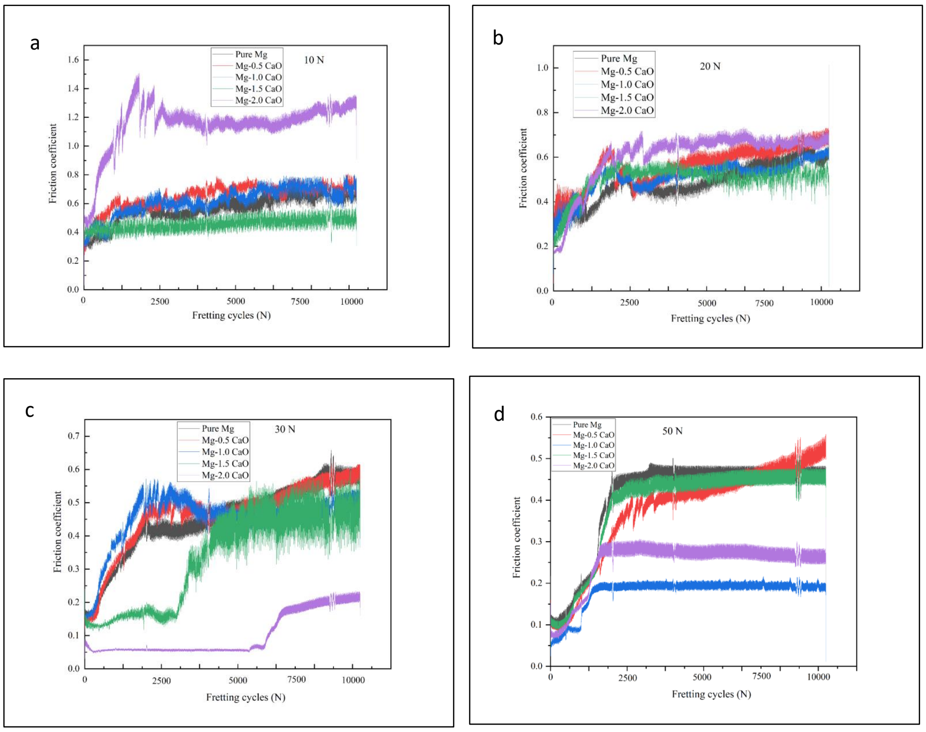


Figure 8: Variation of coefficient of friction (COF) of Mg and Mg-CaO composite a) 10N, b) 20N, c) 30N and d) 50N

9 Organization of the Thesis

The proposed outline of the thesis is as follows:

(a) Chapter 1: Introduction

(b) Chapter 2: Literature Survey

(c) Chapter 3: Experimental Methodology

(d) Chapter 4: Microstructural characterization of Nanocomposites

(e) Chapter 5: Statistical analysis through Response Surface Methodology

(f) Chapter 6: Dry sliding wear nature of the nanocomposites

(g) Chapter 7: Fretting wear behaviour of magnesium nanocomposites

(h) Chapter 8: Conclusions

(i) Chapter 9: Scope For Future Work

10 List of Publications

[1]. S.Kartheesan, B. Shahul Hamid Khan, M.Kamaraj, Sravya Tekumalla and Manoj Gupta, 2022. Dry sliding wear behavior of magnesium nanocomposites using response surface methodology. Journal of Tribology - ASME Transactions, 144(1), p.011704. https://doi.org/10.1115/1.4051410. **(IF =2.5)**

[2]. S.Kartheesan, B.Shahul Hamid Khan, M.Kamaraj, Sravya Tekumalla and Manoj Gupta"Dry sliding wear behavior of CaO reinforced magnesium nanocomposites" (Under Review)

11 List of Conferences

(a) S.Kartheesan, B.Shahul Hamid Khan, "Dry Sliding wear Behavior of Magnesium Nanocomposites- A statistical Approach",ICOFT 2021, National Institute of Technology Pondichery - Karaikal, December 2021.

(b) S.Kartheesan, B. Shahul Hamid Khan, M.Kamaraj "Dry sliding wear study of CaO reinforced magnesium matrix nanocomposites", 4th International Conference on HTSE (Heat Treatment and Surface Engineering) 2023 – ASM International.

(c) S.Kartheesan, B.Shahul Hamid Khan, M.Kamaraj "Influence of CaO Nanoparticles on Fretting Wear Characteristics of Mg Nanocomposites", 4th International Conference on HTSE (Heat Treatment and Surface Engineering) 2023 – ASM International.

References

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