INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 16 No. 6 (2024) 21-30
https://publisher.uthm.edu.my/ojs/index.php/ijie

Evaluation of Micro Hardness of Magnesium Alloy Coated with ZnO and Al_2O_3

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DOI: https://doi.org/10.30880/ijie.2024.16.06.003

Article Info

Received: 21 September 2023
Accepted: 18 September 2024
Available online: 9 October 2024

Keywords

Magnesium, micro hardness, friction, wear, tribology

Abstract

Magnesium and its alloys have wide range of application in automobile and aircraft industries because of their excellent mechanical properties i.e. high strength to weight ratio, availability and good castability etc. However, some challenges occur in using magnesium in aquas service applications, due to its highly reactive nature and low tribological properties such as low wear and corrosion resistance. In this work an experimental study has been done on uncoated and ceramic coated AZ91 alloy to compare hardness and wear properties of both. The coating was applied on varying weight percentage of ZnO and Al_2O_3 ceramic particles. The effect of presence of ceramic particles on the surface of substrate was studied using scanning electron microscope. Micro Vickers hardness test and wear tests were conducted on the samples, and it was found that coating of ceramic particles made the surface harder and wear resistant as compared to the uncoated Mg alloy. Contribution of Al_2O_3 particles plays an important role in increasing the hardness and wear resistance of coated surface. A decrease of 84% in coefficient of friction, increase in wear resistance by 99% and increase in micro hardness by 92% was reported for Al_2O_3 coated sample as compared to the uncoated surface.

1. Introduction

Magnesium and its alloys are one of the most desirable metals due to their excellent mechanical and tribological properties (1). Using magnesium alloy is one of the ways to reduce the weight of automobiles and aircrafts(2), and hence minimizing the fuel consumption which finally results into the reduction of pollution in atmosphere without compromising the performance of the vehicle(3). Now a days magnesium is replacing aluminum as well as iron also as a structural material owing to its light weight and better mechanical properties. This is the reason magnesium is now a days replacing iron and even aluminum also in various air craft parts i.e. oil pump housing, gear box, engine block etc. (4). Magnesium and its alloys have better strength to weight ratio as compared to other existing structural materials(4)(5)(6). The main restriction with magnesium is its poor wear and corrosion resistance. Due to high chemical activity magnesium gets corroded very easily when comes in contact with air or moisture even at room temperature(7).

There are two main ways to overcome this issue. One is to reinforce the metal with other metals(8) and the other is to modify the surface only by applying coating on it(9). In making the composite, the whole material properties get changed and it may deteriorate some of the desired properties of the substrate material(10). While coating the surface, only surface properties get modify. So in places where the properties of magnesium i.e. light weight, strong, less expansive, good castability etc are desired but at the same time the component has to

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withstand high wear and corrosion property, coating the material surface can be done to keep the mechanical properties intact(11).

Ceramics are the materials with high toughness and hardness, if coated on magnesium substrate, may enhance its mechanical and tribological properties. In a study Al_2O_3/ Al composite coating was applied by plasma spray method and it was found that the toughness , wear resistance and strength of the metal increased manifolds(12). In another study magnesium alloy AZ91 was coated with CeO_2 doped Al_2O_3 and a comparison of mechanical and tribological properties was done and the results showed that CeO_2 doping has contributed significantly in enhancing the wear rate and resistance towards corrosion of the material(13). In another research authors coated Al_2O_3 and TiO_2 particles in different varying concentrations on steel discs, In scanning electron microscopy a homogeneous distribution of ceramic particles was observed and hardness was found to be enhanced with increasing percentage of TiO_2 due to the densification of coated surface(14). In a recent research a composite coating of Al_2O_3 / Zn-Ni was applied on mild steel plate by ultrasonic pulse plating method and the result that the hardness of coating enhanced from 340 HV to 750 HV(15). A research done for analyzing the effect of doping of rare earth oxide on Al_2O_3 and TiO_2 coating on stainless steel substrate revealed that the inclusion of rare earth oxide significantly enhanced the wear rate of the substrate and the optimum percentage of Y_2O_3 addition was found to be 20% by weight(16).

In a study a composite coating of ZnO and Al_2O_3 was fabricated on the top surface of mild steel specimen and the tests for micro hardness, corrosion and wear, surface roughness was conducted and the results revealed that the addition of ZnO enhanced the corrosion rate by 96% and micro hardness of the substrate enhanced by 184% (5). In another research, a ZnO coating was fabricated on aluminum alloy to enhance the corrosion resistance of the substrate to make it fit to be used in aerospace and transportation industries and after conducting the tests it was found that resistance towards oxidation increases almost four times than that of the substrate material (17). A group of researchers found out Mg and its alloys enhances its mechanical properties and corrosion resistance after severe plastic deformation. In this research it was found that after undergoing severe plastic deformation magnesium becomes more resistant towards corrosion and also its mechanical properties got enhanced due to existence of different strengthening mechanisms like grain boundary strengthening, grain refinement etc.(18). Magnesium alloys are good biomaterials owing to bone like properties. It is a suitable material for medical implants in joint replacement, intravascular stents, hip, and knee replacement etc. However, depletion of ions during its corrosion makes an adverse effect on it and can cause medical abnormalities inside the body(19).

In another work a group of researchers fabricated the layer of Al_2O_3 ceramic on AZ91 Mg alloy and conducted tests for different mechanical properties of the modified surface. The observations revealed the microhardness of fabricated surface enhanced from 50 VHN to 250 VHN due to the homogenous distribution of hard ceramic particles(20). In a recent work, researchers conducted experiments on Pin on disc setup for thermal spray coating and it was found out from the results that load effect plays an important role in wear rate among other parameters like diameter of pin, angular velocities(21).

From the literature presented above, it was found that no work has been reported on comparison of micro hardness and tribological properties of bare AZ91 alloy and AZ91 alloy coated with ZnO and Al_2O_3 particles in varying weight percentage.

2. Methodology and Sample Preparation

In the current research work, magnesium alloy AZ91 was taken as substrate material. ZnO and Al_2O_3 ceramics were selected as coating materials. Methodology adopted for applying coating and sample preparation method is discussed in following sub-sections.

2.1 Materials

In this study, following composition of AZ91 alloy was selected as substrate material due to its light weight, availability and its use in automobile and aircraft industries. Magnesium alloy AZ91 was procured in the form of a sheet of thickness 5mm. Powders of ZnO and Al_2O_3 with average particle size -48+21 µm and -50+15 µm respectively were procured as coating material. Atmospheric Plasma Process was selected as the coating process. Composition of the procured AZ91 sample is given Table 1.

Table 1 *Composition of procured AZ91 alloy*

Component	Al	Zn	Fe	Mn	Cu	Si	Mg
Percentage	8.089	1.021	0.019	0.486	.001	0.131	90.253

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2.2 Sample Preparation and Coating Formulation

Pre coating operations are needed for better deposition of coating layer on the substrate material. For pre coating operations all the eleven samples were cleaned by acetone and polished with 200 to 900 grit size sandpaper followed by sand blasting to increase the roughness of the surface so that the cohesion between substrate and ceramics can be enhanced to achieve a better coated surface.

From the procured sheet of AZ91 alloy, eleven samples of dimensions 100 x100 x 5 mm^3 were prepared using wire EDM. ZnO and Al_2O_3 ceramics were selected for coating the surface of magnesium alloy owing to their similar chemical activity and almost similar melting point. These Prepared eleven samples were coated in varying weight percentage of ZnO and Al_2O_3 ceramic powders by Plasma Spray Process using MF4 torch. 300 µm thick coating layer was applied on all the eleven samples. The schematic diagram of plasma spray coating process is shown in Figure 1. Following Coated samples with their coating composition as mentioned in Table 2 have been prepared. The Images of coated samples and uncoated sample are shown in Figure 2.

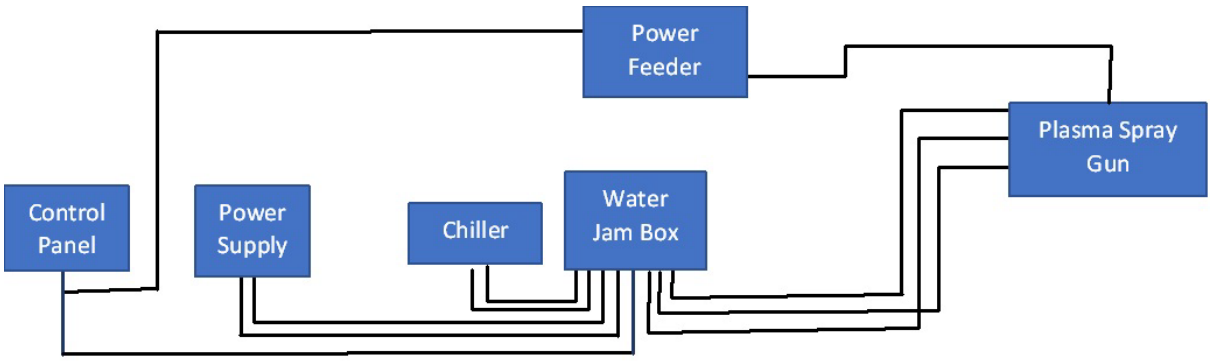


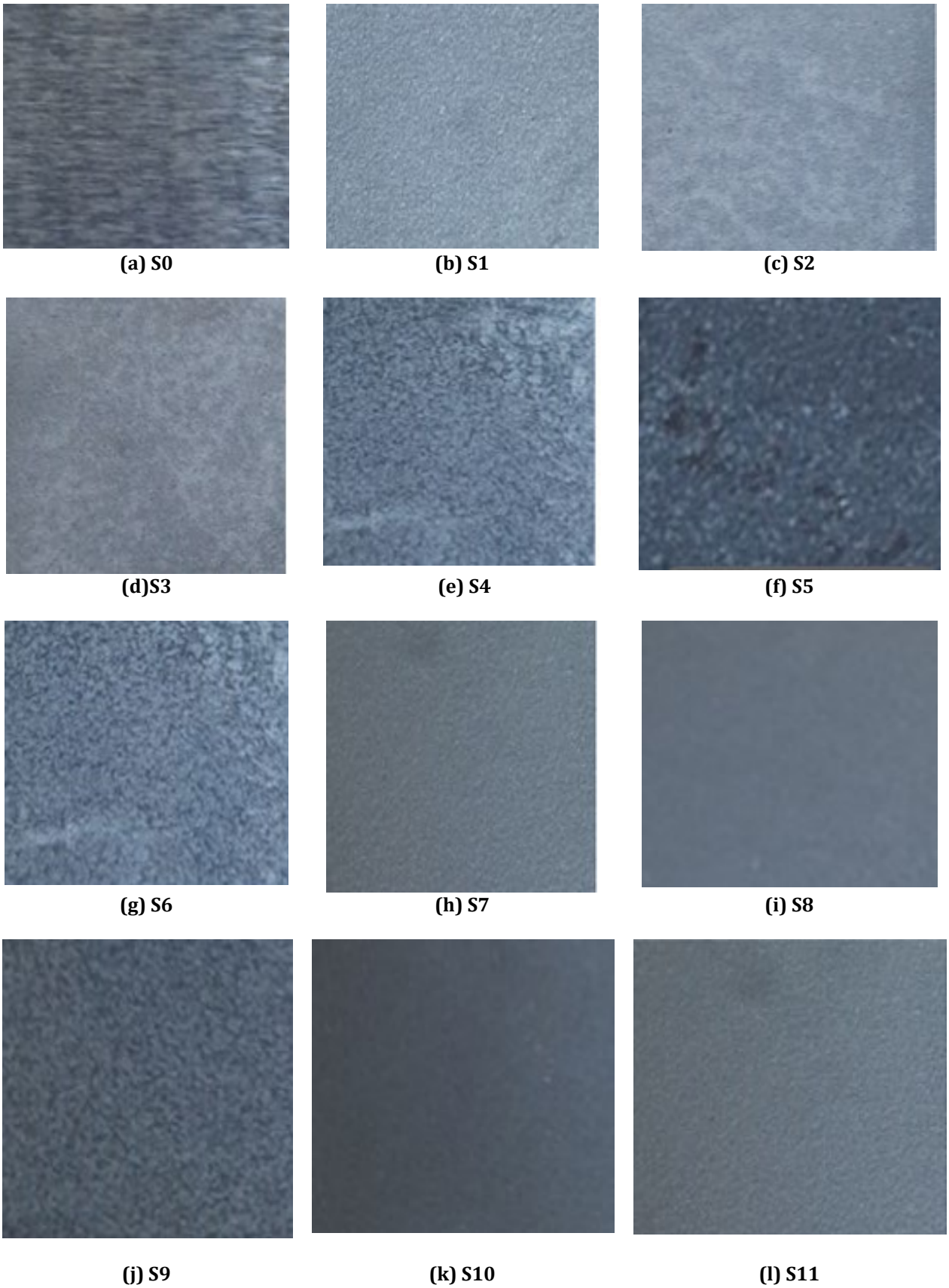
Fig.1 *(a) Schematic representation of atmospheric plasma coating process*

Table 2 *Sample wise composition*

Sample	Sample Name	Al	Zn
Sample 0	S0	0	0
Sample 1	S1	100	0
Sample 2	S2	90	10
Sample 3	S3	80	20
Sample 4	S4	70	30
Sample 5	S5	60	40
Sample 6	S6	50	50
Sample 7	S7	40	60
Sample 8	S8	30	70
Sample 9	S9	20	80
Sample 10	S10	10	90
Sample 11	S11	0	100

2.3 Test Methods

Micro Vickers hardness test was performed at Spectro Analytical Lab in New Delhi using a micro hardness tester at 300g load for 15seconds. The hardness for each sample was calculated at eight equidistant points on the surface of each sample along its diagonal. FESEM (FEI) QUANTA 200F was used to determine microstructure of coated and uncoated samples. The scanning electron microscope available at IIT Delhi was used to observe the microstructure. Coefficient of friction and wear tests were conducted to calculate sliding wear on a pin on disc wear and friction test rig. Pin on disc tribometer available in Delhi Technical University, Delhi, was used to compare wear rate of coated and uncoated samples. Experiments on sliding wear were performed on a Tribometer with a Pin and Disc in atmospheric conditions. Circular discs of diameter 100mm and thickness 5mm were machined from coated samples and alloy steel EN31 was used as a pin. Pin of 10mm diameter was used to examine wear rate. Tests were conducted under various applied load i.e. 20N, 30N and 40N keeping sliding distance as 10m. The sliding wear rate was recorded by calculating volumetric wear loss per unit time.



(a) S0 **(b) S1** **(c) S2**

(d)S3 **(e) S4** **(f) S5**

(g) S6 **(h) S7** **(i) S8**

(j) S9 **(k) S10** **(l) S11**

Fig. 2 *(a)-(l) Image of prepared samples*

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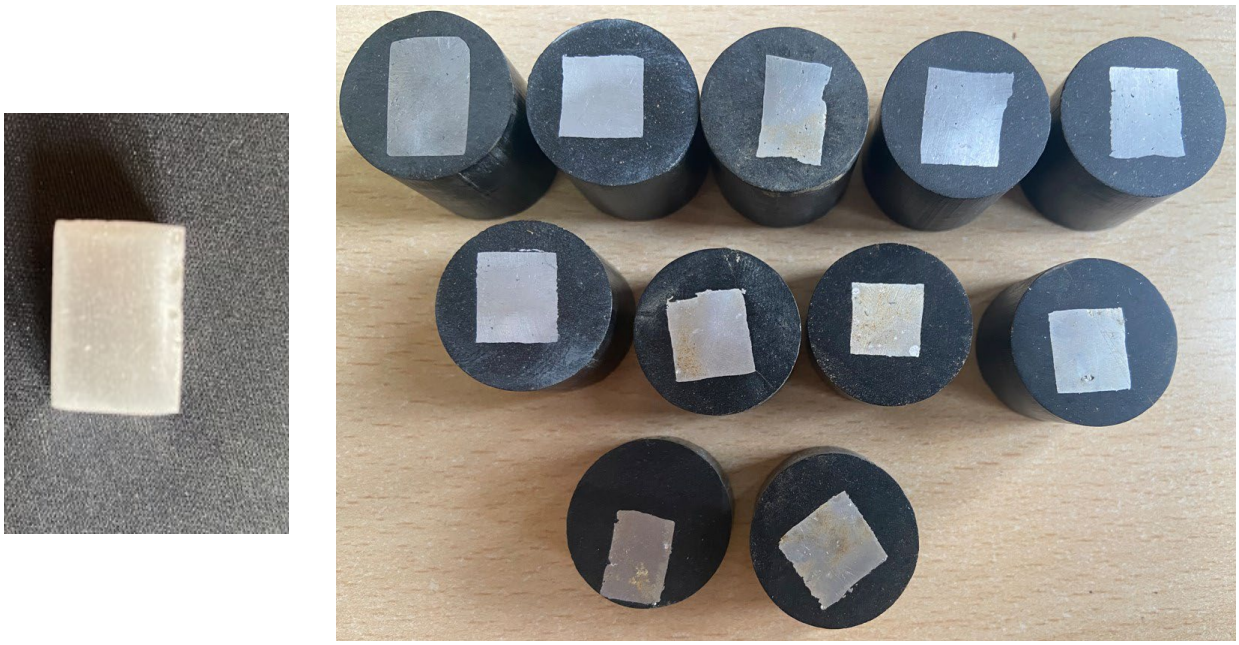
3. Results and Discussion

In flowing sections the experimental results of hardness, coefficient of friction and wear rate has been discussed.

3.1 Micro Hardness

Micro Vickers hardness test was conducted to study the behavior of coated surface. All the eleven samples were tested under a load of 300g for 15 seconds duration. An average of hardness on eight equidistant points on each sample was recorded. In this micro hardness test a diamond indenter was pressed against the sample surface under a load of 300g for the duration of 15seconds. A mold was created for the sample to get fit into the hardness tester. Samples prepared for micro hardness are shown in Figure 3. In this test, the load was applied to the material using 136° pyramidal diamond indenter which forms an indent of square shape on the surface of material. After the indenter is detached from the surface, both the diagonals of the indenter X and Y are measured for calculating their arithmetic mean. The hardness values of micro Vicker's hardness of the coated samples are given in below Table 3.



(a) **(b)**

Fig. 3 *Samples prepared for micro hardness test (a) Uncoated; (b) Coated samples*

Table 3 *Hardness values of samples*

Samples	S0	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11
Hardness Value (VHN)	76	978.4	942	916	872.3	832.8	736	702	624	574.2	423	354

Micro Vicker's hardness values have been plotted for all the samples to compare their hardness number as shown in Figure 4. From the test conducted, It was observed that hardness of ceramic coated samples increases many folds as compared to the uncoated sample S0.The value of Vickers hardness is 76VHN for S0 and it enhanced by almost 91% for sample S1.This result is supported by earlier researches(22) .However, it was observed that the hardness of coated samples is decreasing with the decreasing percentage of Al_2O_3 particles. Al_2O_3 is one of the hardest ceramic materials having very high melting point. It can be seen clearly from the graph that micro hardness of the samples is decreasing very slightly up to 50% Al_2O_3 composition and beyond that the hardness is decreasing rapidly with decreasing percentage of Al_2O_3 particles.

The hardness of surface depends upon the crystal size of particles present at the surface. The hardness decreases with increasing crystal size of the particle present on the surface. ZnO particles have wurtzite structure(23). This structure of ZnO makes it a stackable complex crystal. Because of which ZnO particles have bigger crystal structure. Then, with increasing ZnO particles on the surface the dislocation of intermolecular bonds

will be lesser due to its complex structure and bigger crystal size. Bigger crystal size makes the sample porous as well as less hard as compared to the one having high percentage of Al_2O_3 particles.

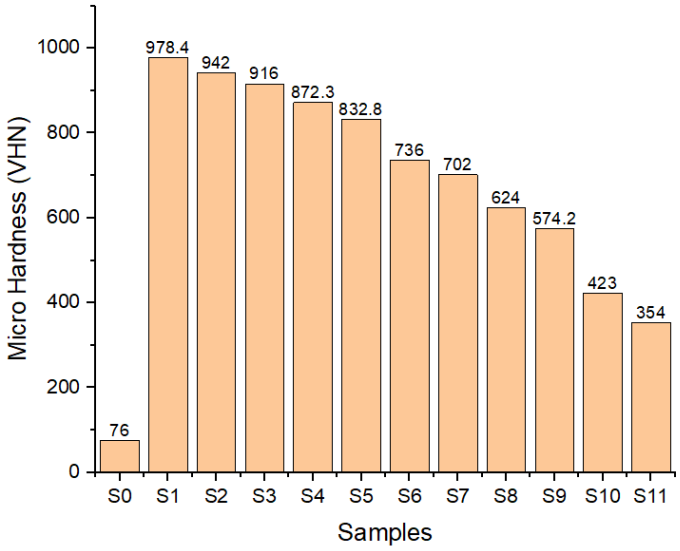


Fig. 4 *Comparison of average Micro Hardness of uncoated and coated samples*

3.2 Surface Characterization

SEM characterization of the samples was done using Scanning electron microscope FEI Quanta 200 F SEM. SEM images of the samples showed homogenous mixture of ceramic particles distributed on the surface with some porosity and micro cracks. Uniform dispersion of ceramic particles supports the adopted plasma spray technique to investigate the properties of the coated surface. All the coated samples have rough microstructure and porous surface.

ZnO particles have lower melting point as compared to Al_2O_3. During the plasma spray process the ceramic particles are impinged on the surface in the form of melted plasma, after that the applied layer gets solidified with in a fraction of time, this rapid solidification makes a part of melted plasma evaporate from the surface and the surface appears to be porous. Hence on increasing the percentage of ZnO particles, the surface becomes porous. The percentage of pores seen from SEM images depicts that the coating becomes dense on increasing the percentage of Al_2O_3 particles. SEM images for all coated samples are shown in Figure 5.

3.3 Coefficient of Friction

Experiments were conducted for coefficient of friction and wear rate for all coated and uncoated samples against varying load i.e. 20, 30 and 40 N and keeping the sliding distance as 10m. A noticeable improvement in wear rate and coefficient of friction between uncoated and all other coated samples can be observed from Fig. 6 and Fig.7. Which proves that coefficient of friction of coated magnesium alloy got enhanced by almost 84% than the uncoated sample. The lesser coefficient of friction and wear rate is mainly because of the high hardness of Al_2O_3 top layer which minimizes the excavated component of friction compared to ZnO mixed coating. The observation says that in starting with decreasing percentage of Al_2O_3 the coefficient of friction rises slightly but after that a sudden hike can be seen in coefficient of friction. The reason is, at the start of the experiments, EN31 pin crushed the irregularities present on the surface and this newly created debris filled the cracks and pores of the surface, hence coefficient of friction improved very slightly. And as the experiment continues, the coefficient of friction increases gradually with a remarkable amount with increasing cracks and pores on the tool path. Due to the high hardness and of sample S1, the frictional force between the sample surface and the counter body (EN31) was minimum hence the coefficient of friction was observed as lowest for S1.

Coefficient of friction gradually increases as the percentage of Al_2O_3 particles decreases. This is the reason why alumina is widely used as industrial wear resistant ceramic. Tests showed that samples had sudden increment in coefficient of friction when the percentage of alumina decreases from 40 to 30 percent. Coefficient of friction got enhanced by 84% by coating the surface with Al_2O_3. The formerly shown hardness results justify the noticed trend in wear. The sample with higher hardness serves lower wear rate.

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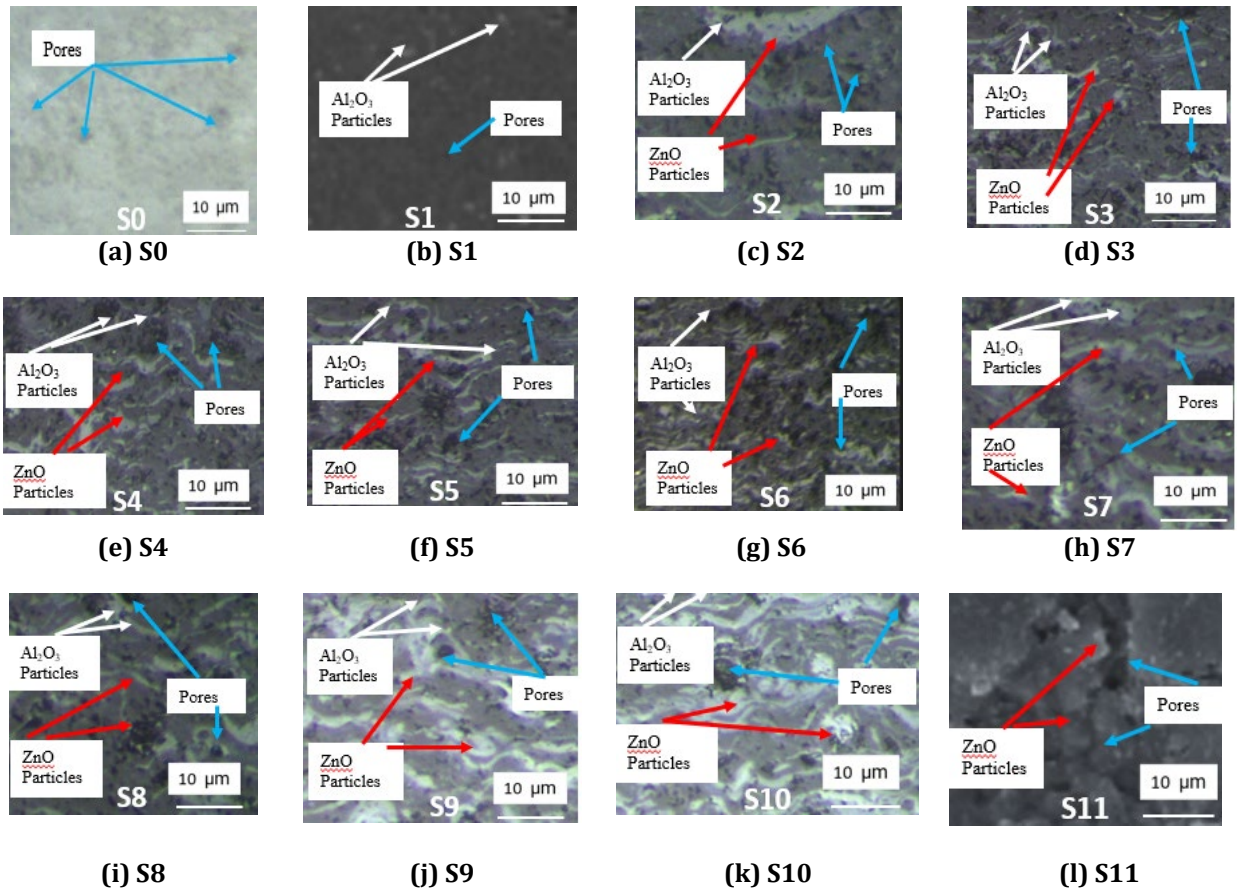
3.4 Wear Rate

The wear resistance of the samples is primarily connected with their coefficient of friction. The sliding wear rate values of all coated and uncoated samples were in sequence proportional to their hardness values. Thus, it is obvious that wear rate of the surface highly depends upon its hardness. The interdependent consequence of increase in wear resistance and hardness are the main cause of enhanced tribological and mechanical performance. From Fig.7, the wear rate of ceramic coated magnesium alloy showed a similar trend as detected in coefficient of friction. Pure Al_2O_3 coated sample showed lowest wear rate means highest wear resistance compared to other samples due to its higher hardness. Wear rate remains almost same for various applied loads i.e. 20, 30 and 40N. Coated surface resists the penetration of counter body in it due to the presence of hard ceramic particles on the surface. As a result, frictional heating occurs between the surfaces of disc and counter body. This ultimately results in thermal softening of base alloy. Hence wear resistance of material increases as the hardness increases. This is in- line with earlier observations [24].



(a) S0 **(b) S1** **(c) S2** **(d) S3**

(e) S4 **(f) S5** **(g) S6** **(h) S7**

(i) S8 **(j) S9** **(k) S10** **(l) S11**

Fig. 5 *(a)-(l) SEM Images of uncoated and coated samples*

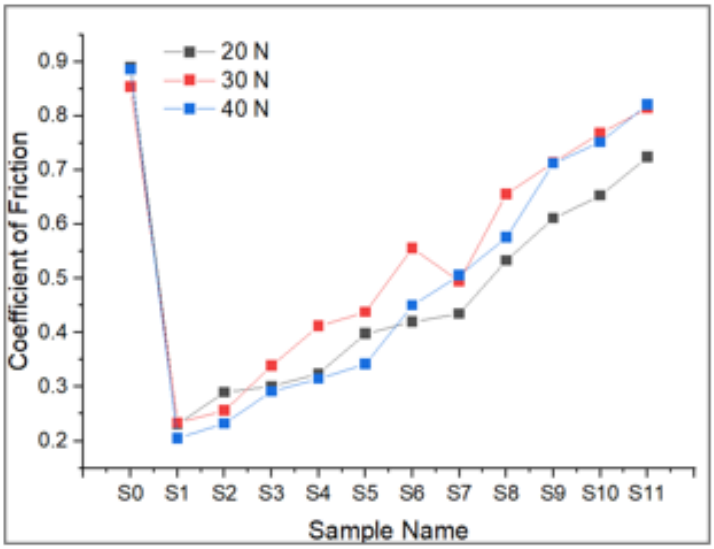


Fig. 6 *(a) Comparison of coefficient of friction of uncoated and coated samples*

Because of the efficient plastic deformation of the hard surface of coated sample, the contact area reduces between the pin and wear track. This results in improving the wear resistance of coated surface. Tests conducted show that Wear rate follow the same trend as coefficient of friction for various applied load. As a result, wear resistance improved by 99% for coated samples as compared to uncoated surface. The same pattern was reported in another researches(25)(26).

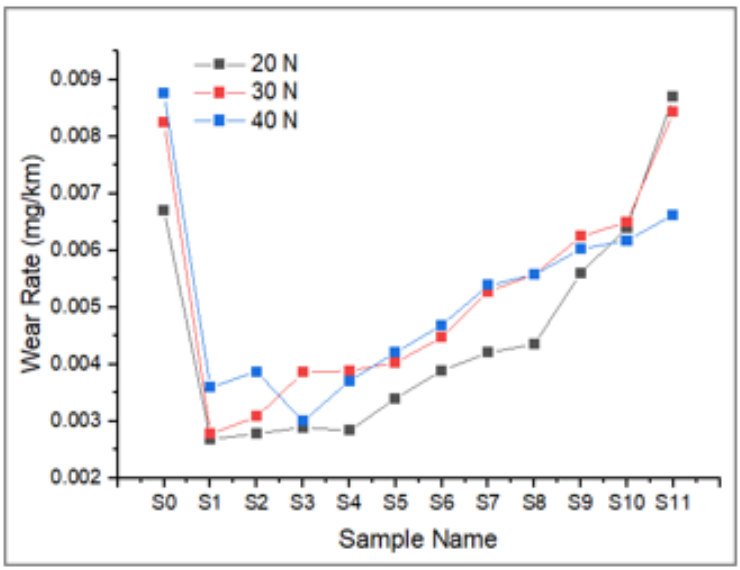


Fig. 7 *(a) Comparison of wear rate of uncoated and coated samples*

4. Conclusions

An experimentation study was done on uncoated magnesium alloy and ceramic coated magnesium alloy samples to compare their hardness and wear properties. The coating was performed by varying the weight percentage of coated ceramic particles. From the experimental results, following conclusions are drawn:

1. Coating of ZnO and Al_2O_3 particles was successfully applied on Mg alloy using plasma spray process. A material with enhanced hardness and wear resistance properties as compared to bare alloy was fabricated.
2. Micro hardness test results show that sample-1 is the hardest material among all other coated and uncoated samples as it has the maximum composition of hard Al_2O_3 particles. Further on decreasing the percentage of Al_2O_3 particles, micro hardness decreases gradually up to its 60% concentration and beyond it the hardness

decreases rapidly with increasing coefficient of friction and wear rate. Overall hardness of coated samples got improved by 92% compared to uncoated one.

3. SEM images show that porosity of the samples increases with increasing percentage of ZnO particles, which supports the results obtained from the hardness test.
4. All the samples underwent wear test, and their values of wear rate and coefficient of friction were compared. The surface having better hardness value provided a lower wear rate which means higher resistance towards wear. It was found that coated samples show better wear resistance as compared to uncoated one and samples having high percentage of alumina have higher resistance towards wear. This indicated that coating of ceramic particles improved the hardness and wear resistant properties of substrate material. Wear resistance of the surface was found to be improved by 99% after coating with ceramic particles.
5. As sample-1 was found to be the hardest material and showed the best wear resistance property also, so it can be used in various automobile and aircraft parts where hard and wear resistant materials are required.

Acknowledgement

I would like to express my sincere gratitude to Spectro Analytical Lab, New Delhi and Tribology Lab, DTU, New Delhi for providing access to the scanning electron microscope and micro hardness tester. Their contribution has been invaluable in conducting the analysis in this study.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Divya Chandra, Nathi Ram Chauhan,* ***data collection:*** *Divya Chandra;* ***analysis and interpretation of results:*** *Divya Chandra, Nathi Ram Chauhan;* ***draft manuscript preparation:*** *All authors reviewed the results and approved the final version of the manuscript.*

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