J. Min. Metall. Sect. B-Metall. 59 (2) (2023) 279 - 286

Journal of Mining and Metallurgy,
Section B: Metallurgy

STUDY ON WEAR BEHAVIOR CHARACTERISTICS OF ZrO_2 AND ZrN COATED AZ91D Mg ALLOY

A. Haiter Lenin [a*], P. Kumaradhas [b], M. Sivapragash [c], S.C. Vettivel [d]

[a] WOLLO University, Kombolcha Institute of Technology, Department of Mechanical Engineering, Kombolcha, Ethiopia
[b] University of Technology and Applied Sciences, Department of Mechanical Engineering, Salalah, Oman
[c] Narayanaguru College of Engineering, Department of Mechanical Engineering, Manjalumoodu, Tamilnadu, India
[d] Chandigarh College of Engineering and Technology, Department of Mechanical Engineering, Chandigarh, India

(Received 23 March 2023; Accepted 17 August 2023)

Abstract

The as-received AZ91D Mg alloy was coated with ZrO_2 and ZrN ceramics using the Physical Vapour Deposition (PVD) process. Dry sliding wear investigations were performed in a pin-on-disc wear tester at a sliding velocity of 2-8 m/s and a normal load of 2-10 N. The wear mechanisms such as abrasion, delamination, thermal softening, and oxidation were observed. The results showed that PVD coating increased the wear resistance of AZ91D Mg alloy. The worn surface was examined with Scanning Electron Microscope (SEM).

Keywords: *AZ91D Mg alloy; PVD coating; Dry sliding wear*

1. Introduction

Magnesium and its alloys are one of the low density materials having high strength to weight ratio that show good mechanical properties and damping capacity which have been useful for automotive, structural and aircraft industries [1-5]. However, the corrosion and wear resistance performance of Mg alloys are low [6]. The wear and corrosion performance of the Mg alloy have been improved by effective coating methods [7]. The PVD is a highly suitable surfaces coating method for Mg alloy and exhibits excellent surface properties [8-11]. PVD hard coating of Mg alloys is widely preferred for different tribological applications [12]. The wear resistance of AZ91D Mg alloy was improved by laser surface remelting method and the result revealed that 70% of wear volume loss was minimized [13]. The wear behavior of AE42 Mg alloy and its composites was studied in pin on disc tester. The finding shows that higher wear rate were attained at higher load for the alloy and its composites [14]. Palta et al. [15] reported the wear behavior changed in according to the applied load and alloying elements. The wear behavior of PEO and hybrid coated MRI 230 Mg alloy exhibited that up to 2N the coating was not damaged and at 5N micro fracture occurred in the coating reduced the wear behavior of the alloy [16]. The titanium coated Az91D Mg alloy by PVD process improved wear resistance properties and reduced the mechanical damage [17]. The laser surface melted AM60B Mg alloy showed good wear resistance compared to as-received AM60B Mg alloy [18]. The Al+SiC cladded AZ91D Mg alloy exhibited improved wear resistance of the AZ91D Mg alloy [19]. The wear rate of AZ91D and 3wt% RE Az91 Mg alloy increased on increasing sliding speed and testing temperature [20].

The ZrO_2 and ZrN coatings have been shown to enhance the hardness, wear and corrosion resistance of the substrate [21, 22]. The ZrO_2 coat exhibit high mechanical properties, temperature resistance and chemical stability [23]. The importance of using Mg alloy for tribology applications are very much essential and no literature were reported on coated ZrO_2 and ZrN in AZ91D Mg alloy using the PVD method.

The aim of this investigation is to develop wear map and mechanism for uncoated, ZrO_2 and ZrN coated AZ91D Mg alloy using the PVD method.

2. Experimental Procedure

2.1. Materials

Corresponding author: drahlenin@kiot.edu.et

https://doi.org/10.2298/JMMB230323024A

The AZ91D Mg alloy was used as substrate in this study. The yttrium stabilized ZrO_2 and ZrN sputtering were employed in the coating process.

2.2. Heat Treatment

Cast AZ91D Mg alloy bar was heat treated at 400 °C for 24 h followed by water quenching and cooling air treatment at 216 °C for 6 h [24]. Three sample specimens were cut from the bar of size 25 x 25 x 5 mm^3 and polished well with sand grit of range 800 to 1200. Once the process was completed, the specimens were washed with distilled water and acetone and placed in warm air to remove the moisture from it.

2.3. PVD Coating Process

AZ91D cast magnesium alloy was cut into sample with size 25 x 25 x 5 mm^3. Specimen samples were polished using emery paper with 1500 grade and Al_2O_3 paste of average size 1 µm. The polished samples were washed in acetone and distilled water, finally dried in warm air at the temperature of 40° – 60° for 30 min - 60 min [25]. Furthermore, these samples were used for PVD coating. "HINDHI VAC Planar Magnetron RF/DC Sputtering Unit, Model: 12"MSPT" was used for doing the PVD coating. The high purity Zirconium Dioxide (ZrO_2) with cylindrical shape and 0.5 to 1 inches thickness with the following optimum conditions were required for doing the ZrO_2 coating: Power input of 200 W, Chamber pressure of 3 x 10^{-3} bar and Argon as the gas in one and Nitrogen as the gas in the other [26, 27].

2.4. Wear Testing

Wear tests were performed in dry sliding condition using a pin-on-disc wear machine. The oil hardened steel of diameter 150 mm was used as disc of wear tester. In accordance with the ASTM G99, the disc surface roughness was maintained at 0.15 µm. Before starting every test, the disc was rubbed with 800 grid SiC emery paper and then cleaned by acetone to remove the deposits on the wear track. The sample size of 15 x 15 x 5 mm^3 was clamp by a suitable holder which acted as pin. The tests were conducted at room temperature in four different loads 2, 5, 8 and 10 N and velocity of 2, 4, 6 and 8 m/s for a sliding distance of 500 mm. The samples were weighed before and after the test using an electronic balance with an accuracy of 0.0001 g to find the mass loss. The wear rate in mm^3/m was calculated from mass loss and coefficient of friction was measured through data acquisition system. The worn out surface were examined using SEM with EDX.

3. Results & Discussion

3.1. SEM Microstructure

The Fig. 1 & 2 show the cross section SEM image and EDAX of ZrO_2 and ZrN coated Mg alloy.

The approximation deposition of 1.4 µm ZrO_2 layer was observed on Mg alloy as represented in Fig.1 (a). From Fig.1 (b), it was evident that coated layer had Zr, Zn, O, Mg, and Al elements. Fig.2 (a) shows the coating thickness of 0.9 µm was obtained on Mg alloy by ZrN material. The elements that presented in the coated material were confirmed by Fig. 2(b).

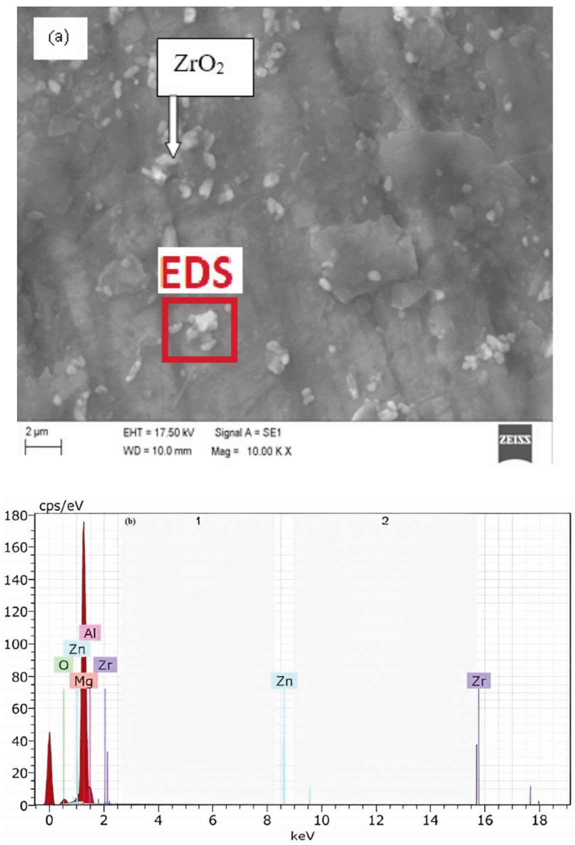

Figure 1. *(a) Cross sectional SEM image of ZrO_2 coated Mg alloy, (b) EDX of ZrO_2 coated Mg alloy*

3.2. Hardness

Fig.3 represents the comparison of the hardness of the coated and uncoated coated Mg alloy. These revealed that the coated surface had higher hardness than the uncoated Mg alloy. This occurred due the addition of ZrO_2 and ZrN particles which enhanced the mechanical properties of Mg alloy. The Zirconium and Zirconium nitride particles are familiar for hardness and wear resisting characters. Therefore they increased the hardness of Mg alloy when they were used as coating material. ZrN coated Mg alloy

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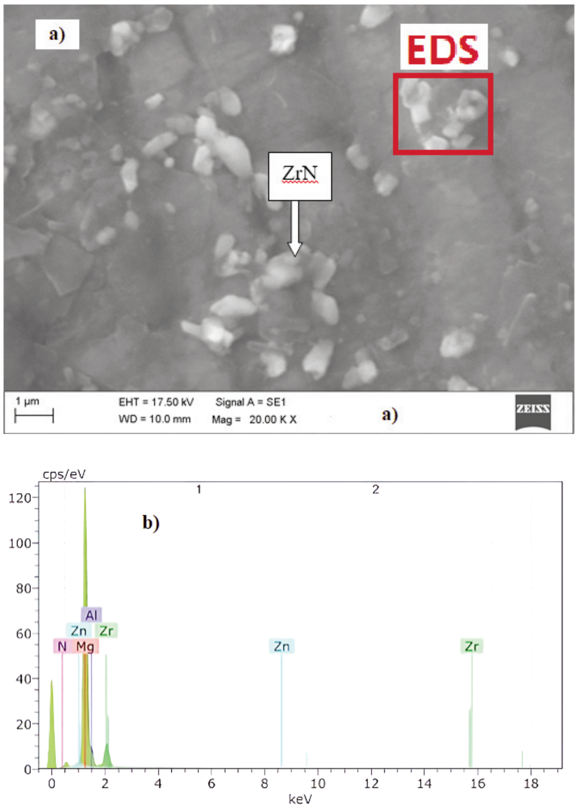

Figure 2. *(a) Cross sectional SEM image of ZrN coated Mg alloy, (b) EDX of ZrN coated Mg alloy*

exhibited better performance than ZrO_2 coated Mg alloy. This was due to the higher performing nature of ZrN than ZrO_2.

3.3. Wear Rate

Fig.4 presents the wear rate of uncoated, ZrO_2 and ZrN coated Mg alloys for different normal loads at sliding speed of 8 m/s and 2 m/s. Fig.4 (a) shows the wear rate of uncoated and coated Mg alloys for various normal loads at a constant sliding speed of 8 m/s. For all these Mg alloys, the wear rate increased with the increase of load. Among the three Mg alloys, the ZrO_2 and ZrN coated Mg alloy showed lower wear rate and both had almost same values. At higher sliding speed, the wear rate reduced due to frictional heating [28]. At lower sliding speed of 2 m/s, the wear rate for uncoated Mg alloy were higher than for the coated Mg alloy as shown in Fig. 4 (b). It can be noticed that at 2 m/s sliding speed the wear rate of all these Mg alloys had higher values than at sliding speed of 8 m/s. The larger wear rates were attained at lower sliding speed which happened due to relative hardness of the material [29]. Also the wear rate variations articulated that various wear mechanism played significant role [30].

Fig.5 shows the coefficient of friction of uncoated

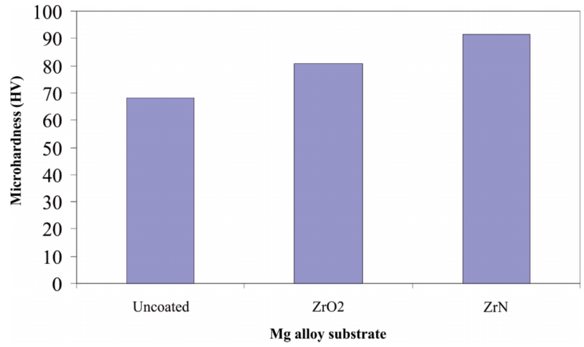

Figure 3. *Microhardness of uncoated and coated Mg alloy*

and coated Mg alloy. Fig.5 (a) represents the coefficient of friction at sliding speed of 8 m/s and the results revealed that ZrN coated Mg alloy showed higher frictional values than ZrO_2 coated and uncoated Mg alloy. Also similar trend was observed in Fig.5 (b). Moreover, from Fig. 5 (a) & (b), it was observed that coefficient of friction at sliding speed of 2 m/s was lower for all these Mg alloy than for the sliding speed of 8 m/s. These changes showed that various wear mechanism were involved at several test conditions.

The contour maps were obtained using Minitab and are shown in Fig.6. These counter maps were developed in order to find the influence of sliding speed and normal load on the wear rate. Fig.6 (a) represents the contour map of uncoated Mg alloy, the results emphasized that at low sliding speed with larger normal load experienced severe wear rate when compared with high sliding speed and low normal load. It was conformed from Fig.6 (b) that lower sliding speed and higher normal load increases resulted in higher wear rate. Also similar trend was observed in Fig. 6(c). Among the three, the wear rate of ZrN coating was the least.

3.4. Wear Mechanism

To study the various wear mechanism that took place on wear samples in different wear conditions were examined by SEM. The wear mechanism like abrasion, adhesion, plastic deformation and oxidation were observed.

SEM of the worn surface of the uncoated and coated samples tested at 2 m/s, 2N are shown in Fig.7. Slight scratches and ploughing were observed in the images. In ploughing, the materials were dispersed on both side of the grooves which led to abrasion wear [31]. The EDS of the worn surface of coated samples showed that wear track depth was within the coating limit.

Fig.8 presents the SEM of worn surface tested at 2 m/s, 10 N for uncoated and coated samples. The semi-solid type of adhesive wear and sticking of debris on

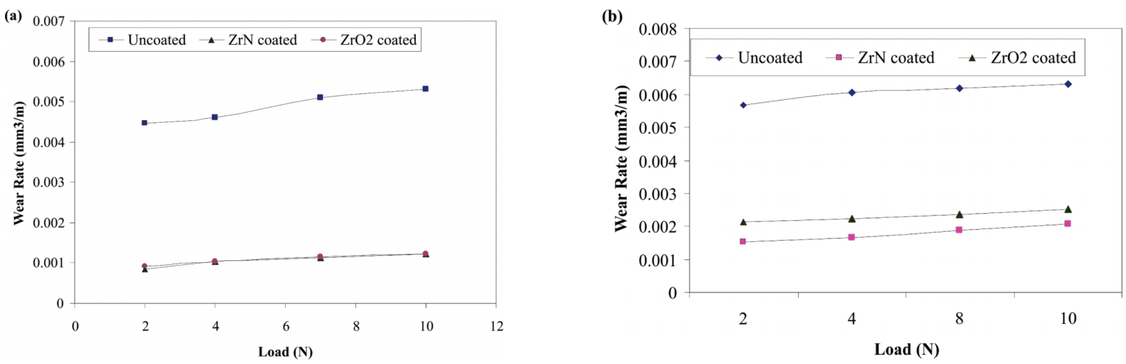

Figure 4. *Wear rate of uncoated and coated Mg alloy (a) at 8 m/sec (b) at 2 m/sec*

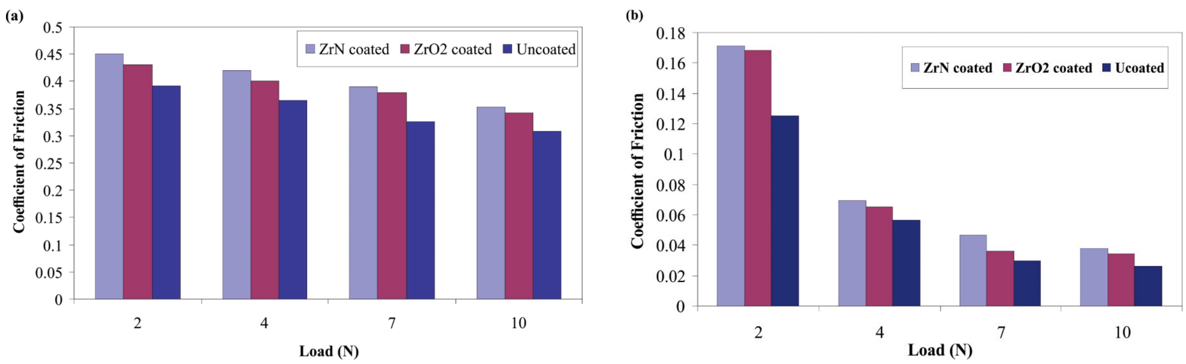

Figure 5. *Coefficient of friction of uncoated and coated Mg alloy (a) at 8 m/sec (b) at 2 m/sec*

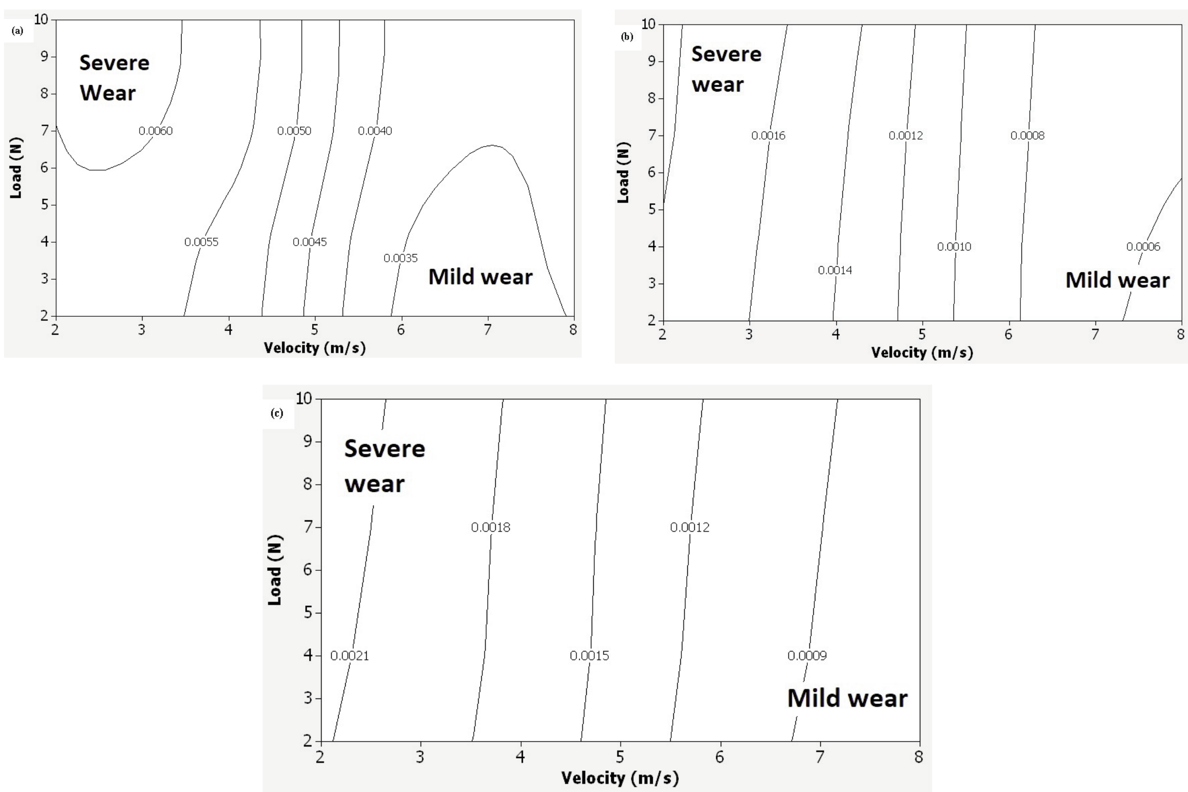

Figure 6. *Contour maps of the wear results of (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

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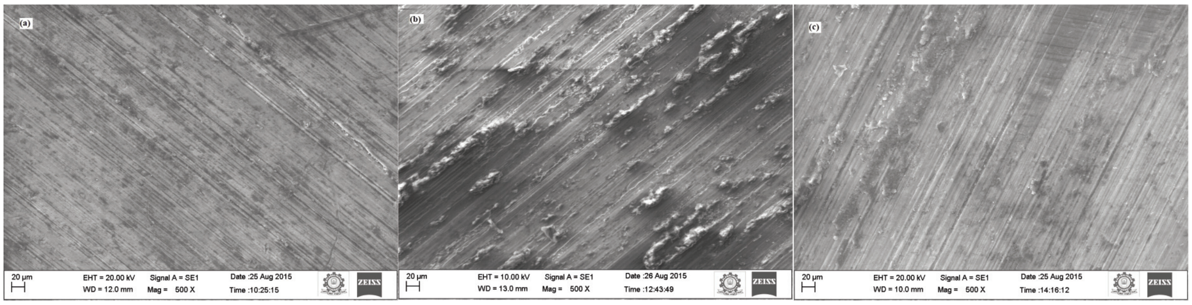

Figure 7. *SEM of the worn surface at 2m/s, 2N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

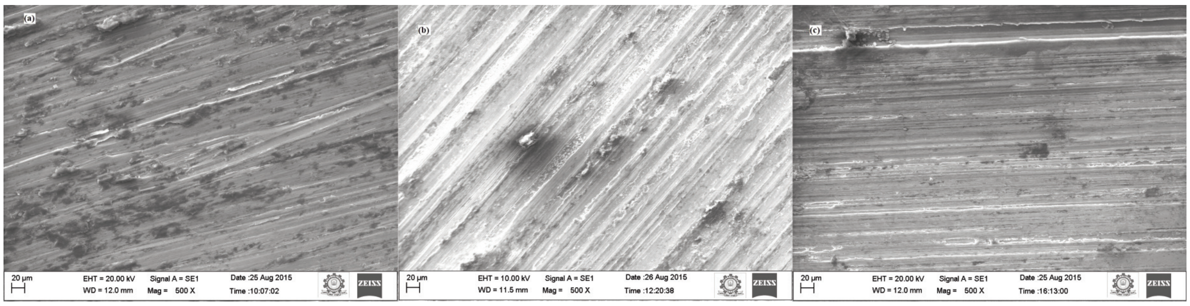

Figure 8. *SEM of the worn surface at 2m/s, 10N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

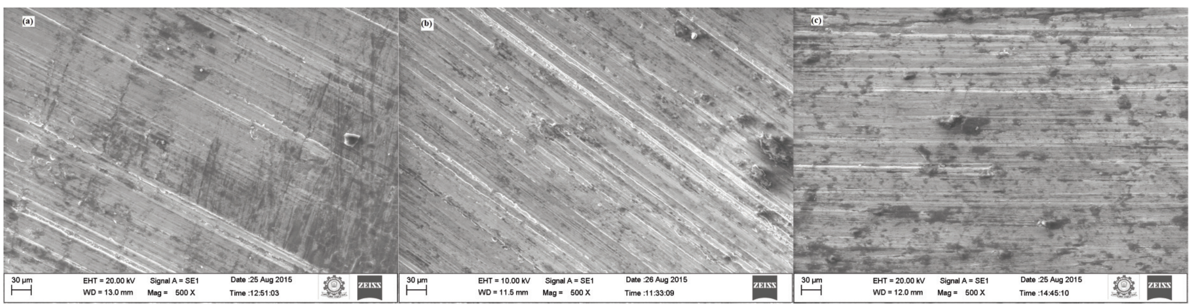

Figure 9. *SEM of the worn surface at 4m/s, 4N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

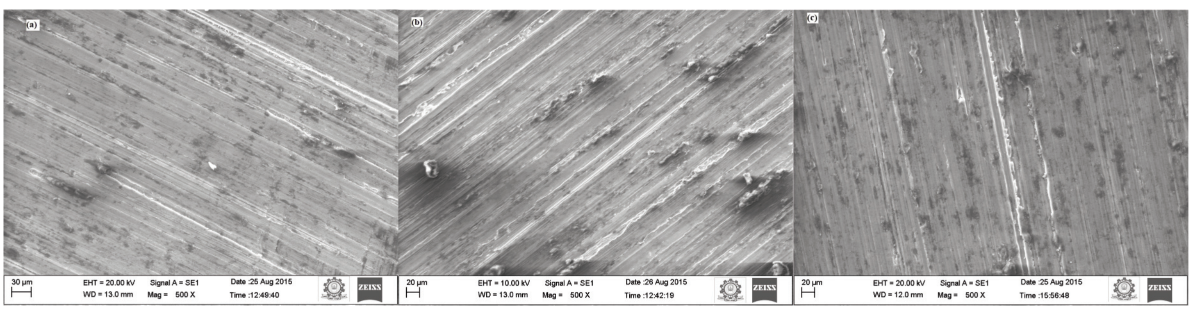

Figure 10. *SEM of the worn surface at 6m/s, 8N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

the surface can be noticed in Fig. 8 (a). Fig. 8 (b) & (c) show debris and abrasive wear mechanism. This articulated on increasing the load to 10 N at low speed of 2 m/s which separated the oxidized particles from the surface in the form of debris [32, 33]. Fig. 9 shows the SEM of worn surface of uncoated and coated samples tested at 4 m/s, 4N. For this speed and load, all the samples had small size debris and abrasive wear.

The worn surface of uncoated and coated samples tested at 6 m/s, 8 N were examined by SEM as shown in Fig.10. The uncoated sample Fig.10 (a) revealed

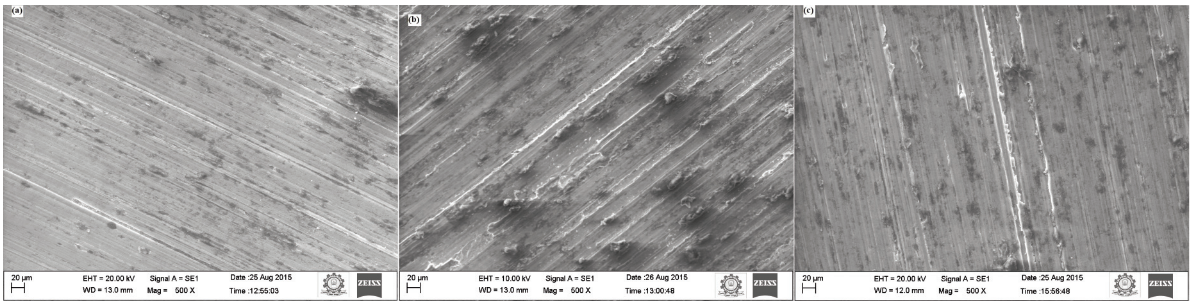

***Figure 11.** SEM of the worn surface at 8m/s, 2N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

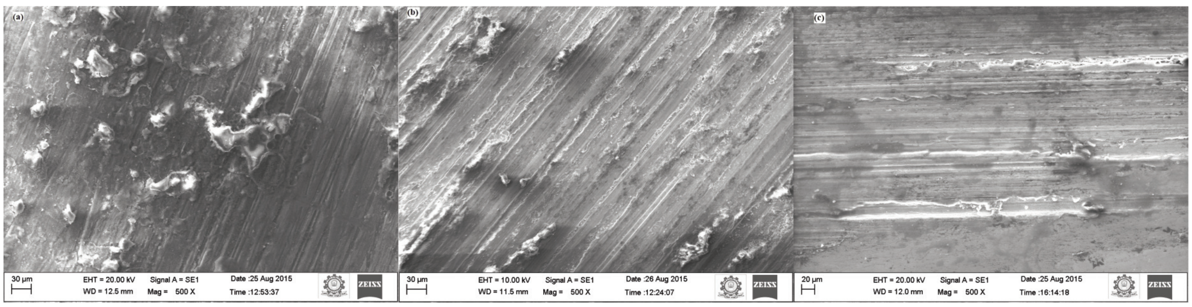

***Figure 12.** SEM of the worn surface at 8m/s, 10N (a) uncoated Mg alloy (b) ZrO_2 coated Mg alloy (c) ZrN coated Mg alloy*

slight scratches and abrasive wear. Moreover, the delamination and abrasive wear could be observed in Fig.10 (b) & (c). Fig. 11 shows the SEM of worn surface of uncoated and coated samples tested at 8 m/s, 2 N. It was noted that increasing the speed from 4 to 8 m/s led to detaching of more debris in all samples. Further, abrasive wear was observed and it was produced by the surface irregularities created due to the removal of particles from the surface [32].

Fig.12 presents the SEM of worn surface of uncoated and coated samples tested at 8 m/s, 10 N. The uncoated sample shows thermal softening and melting as represent in Fig.12 (a). This occurred when surface temperature of the samples was increased due to frictional heating [33]. Fig.12 (b) & (c) show that delamination, abrasive wear and oxidation were produced on the surface of the coated samples. For higher speed and load, the frictional heating increased and led to forming of oxidation [31].

4. Conclusions

- The wear behavior of ZrO_2 and ZrN coated AZ91D Mg alloy were investigated successfully using pin-on-disc wear tester.
- The microhardness of ZrN coated alloys showed higher hardness than ZrO_2 coated and uncoated alloy.
- The ZrN coated alloy had lower wear rate at lower sliding speed of 2m/s. The ZrO_2 and ZrN coated alloy had almost similar wear rate at high speed of 8 m/s.
- The coefficients of friction of ZrN coated alloy had higher value at all sliding speeds. The worn out surfaces revealed abrasion, adhesion, delamination, oxidation, thermal softening and melting wear mechanisms.
- The wear map was developed for uncoated and coated AZ91D Mg alloy.

Acknowledgements

We thank who helped us learn a lot about this project.

Author Contributions

Research concept and drafted the article [A. Haiter Lenin]. Supervised, data analysis and interpretation [M. Sivapragash]. Data collection and edited the paper [P. Kumaradhas, S. C. Vettivel]. All authors read and approve the final manuscript.

Data availability

All data generated or analyzed during this study are included within this article

Competing Interests

Authors declare no conflict of interest.

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ISPITIVANJE KARAKTERISTIKA AZ91D Mg LEGURA PREVUČENIH SLOJEM ZrO_2 I ZrN

A. Haiter Lenin [a*], P. Kumaradhas [b], M. Sivapragash [c], S.C. Vettivel [d]

[a] Univerzitet WOLLO, Institut za tehnologiju u Kombolči, Fakultet za mašinsko inženjerstvo, Kombolča, Etiopija

[b] Univerzitet za tehnologiju i primenjene nauke, Fakultet za mašinsko inženjerstvo, Salalah, Oman

[c] Inženjerski fakultet Narayana Guru, Odsek za mašinsko inženjerstvo, Manjalumoodu, Tamil Nadu, Indija

[d] Fakultet za inženjerstvo i tehnologiju u Čandigaru, Odsek za mašinsko inženjerstvo, Čandigar, Indija

Apstrakt

AZ91D Mg legura je u stanju u kojem je primljena prevučena slojem industrijske keramike, ZrO_2 i ZrN, postupkom fizičke parne depozicije (PVD). Ispitivanja habanja pri suvom kliznom trenju izvedena su u uređaju za habanje tipa igla-na-disku pri brzini klizanja od 2-8 m/s i normalnom opterećenju od 2-10 N. Primećeni su mehanizmi habanja kao što su abrazija, delaminacija, termičko omekšavanje i oksidacija. Rezultati su pokazali da je PVD prevlaka povećala otpornost na habanje AZ91D Mg legure. Habana površina je pregledana pomoću skenirajućeg elektronskog mikroskopa (SEM).

Ključne reči: *AZ91D Mg legura; PVD prevlaka; Suvo klizno habanje*