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Research Article

Investigation of Microstructure and Wear Behaviors of AZ91 Alloy Under Different Heat Treatments

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Abstract

This article investigates the effects of heat treatment applied to AZ91 magnesium alloys on wear behavior under different loading conditions. Apart from as-cast AZ91 alloy, AZ91 alloy samples were subjected to both solid-solution treatment (400°C for 16 hours) and three other aging processes (216°C for 4, 8, and 12 hours) under three different applied loads (10 N, 25 N and 50 N). Under various applied loads, microstructural, characteristics, hardness, friction coefficient, and specific wear rate behavior were examined.

In all samples, the specific wear rate increased with the applied load, while the friction coefficient decreased. The highest specific wear rate was observed in a solid-solution treated sample, but the specific wear rate changed inversely with aging time. Furthermore, the microhardness increased in direct proportion to aging time. The results indicated that the sample that was aged for 12 hours wore 13.6% less under 10 N load than the sample that was only treated with solid solution, but the same sample wore 25% less under 50 N load. Microhardness was increased with the increase in β-precipitates, while wear decreased with the increase in hardness.

Keywords: AZ91, microstructure, wear behaviour, hardness, friction coefficient

AZ91 Alaşımının Farklı Isıl İşlemler Altında Mikroyapı ve Aşınma Davranışlarının İncelenmesi

Öz

Bu çalışmada, AZ91 magnezyum alaşımlarına uygulanan ısıl işlemin, uygulanan farklı yük koşulları altında aşınma davranışı üzerindeki etkisini deneysel olarak araştırmayı amaçlamaktadır. Saf-AZ91 alaşımından farklı olarak, AZ91 alaşım numuneleri uygulanan üç farklı yük (10N, 25N ve 50N) altında hem homojenleştirme işlemine (400°C'de 16 saat) hem de farklı üç yaşlandırma işlemine (216°C'de 4, 8 ve 12 saat) tabi tutulmuştur. Uygulanan çeşitli yükler altında mikroyapısal, karakteristik, sertlik, sürtünme katsayısı ve aşınma oranı davranışları birbirine karşı incelenmiştir.

Elde edilen sonuçlar, uygulanan yükle birlikte aşınma oranının tüm numunelerde arttığını, sürtünme katsayısının ise azaldığını göstermiştir. En yüksek aşınma oranı sadece homojenleştirme işlemine tabi tutulmuş numunede görülürken, yaşlandırma süresiyle aşınma oranının ters orantılı olarak değiştiği, ayrıca yaşlandırma süresiyle mikrosertliğin ise doğru orantılı olarak arttığı gözlenmiştir. 12 saat süreyle yaşlandırma işlemine tabi tutulan numunenin sadece homojenleştirme işlemi uygulanan numuneden 10 N yük altında % 13,6 daha az aşındığı gözlenirken, sonuçlar aynı numunenin 50 N yük altında yaklaşık % 25 daha az aşındığını ortaya koymuştur. Yaşlandırma süresiyle birlikte yapıdaki β-fazı çökeltilerin artması mikrosertliği artırmış, sertlikteki artış ise aşınmayı azaltmıştır.

Anahtar Kelimeler: AZ 91, mikroyapı, aşınma davranışı, sertlik, sürtünme katsayısı

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1. Introduction

Among the lightest structural materials, magnesium alloys are 1/3 lighter than aluminum and widely used in the manufacturing industry, automotive, defense, and electronic parts because of their lightness and high specific strengths, damping capacities, corrosion resistance, and electromagnetic shock shielding performance [1-7]. Magnesium alloys need to improve their ability to resist local damage such as cracks, abrasions, and corrosion. Many engineering applications suffer from wear, including transmissions, steering columns, seat frames, fuel tank caps, and so on. In this case, structural failure directly affects the whole component [8]. The heat treatment of magnesium alloys enhances their wear resistance. At different times and temperatures, heat treatment can alter the surface of phases along with their species and amounts [9]. Due to its high Al and Zn content, AZ91 is different from AZ31 and AZ63 in that it has high strength and corrosion resistance. The presence of β-phase ($Mg_{17}Al_{12}$) along grain boundaries plays a role in establishing these properties. AZ91 alloys can be improved in terms of wear resistance by using two heat treatment methods: solid-solution and aging treatment [10].

A few studies have been conducted on the effect of heat treatment parameters on magnesium alloys [11-13]. In die-cast AZ91 Mg-alloy, Chelliah et al. [10] found that the lower coefficient of friction is associated with the load-bearing capacity of the hard β-$Mg_{17}Al_{12}$ phase. The wear rate of homogenized AZ91 Mg alloy is reduced by 6.21 times over that of as-cast AZ91 Mg alloy. In another study, it was observed that the heat treatment time differences affected the distribution and precipitation of the eutectic phase during the T6 process, in this case, the samples kept at low time showed lower wear rates [13]. Incesu and Gungör [14] found in their study that the $Mg_{17}Al_{12}$ phase of the AZ63 alloy was dissolved with solid-solution treatment and subsequently the mechanical properties decreased due to the presence of precipitates along the grain and even grain boundaries. Chen et al. [15] also focused different heat treatments on the wear behavior of AZ91 magnesium alloys. Apart from our study, they selected different times and temperatures at solid-solution and aging treatment and found that the intermetallic phase tended to generate cracks during the high loads (50-100N). In order to eliminate crack propagation, aging time and temperature directly improve the alloy strength and hardness due to the high density dispersed fine intermetallic phase precipitated in the matrix. Lastly, Hong and Zhiwei [16] also investigated AZ91 alloys with Yttrium substitution under five different applied loads (5-70N). They observed that T6 heat treatment conditions show better wear resistance than without Yttrium addition since Al_2Y and intermetallic phase ($Mg_{17}Al_{12}$) facilitate the sliding and reduce the frictional force.

AZ91 magnesium alloys were subjected to a solid solution and three aging treatments to fill a gap in the literature regarding the effect of heat treatment on wear behavior under different loads. In this study, wear properties, morphology, structure, hardness, and morphology were studied under three selected wear conditions (10N, 25N, and 50N).

2. Material and Methods

As-cast AZ91 magnesium alloys were prepared in a round shape with a diameter of 10 mm by Yildirim Casting Trade Limited Company. The chemical composition of as-cast AZ91 alloys is shown in Table 1.

Table 1. The chemical composition of AZ91 alloy (wt. %)

	Al	Zn	Mn	Si	Cu	Fe	Mg
AZ91	8.5	0.7	0.32	0.01	0.001	0.001	Balance

2.1. Heat Treatment Process

Both solid-solution and aging treatment were applied in temperature controlled electrical furnace. Solid-solution treatment was conducted at 400°C for 16 hours at a rate of 3°C/min. and then quenched in the air. This temperature is selected since it is above the solvus temperature of Al-Mg phase diagram [17]. On the other hand, in the aging process, solid-solution samples were kept at 216 °C for 4, 8, and 12 hours, separately, and afterward, quenched in water. Table 2 shows the classification of the prepared sample types.

Table 2. Description of all samples with their identification numbers

Sample ID	Description
A	As-cast AZ91 alloy
B	AZ91 alloy subjected to solid-solution treatment
C	AZ91 alloy subjected to solid-solution and aging treatment at 216°C for 4 hours
D	AZ91 alloy subjected to solid-solution and aging treatment at 216°C for 8 hours
E	AZ91 alloy subjected to solid-solution and aging treatment at 216°C for 12 hours

2.2. Characterizations

To prepare all samples, 120 grit SiC abrasive paper was used first, followed by 180, 320, 400, 600, 800, and 1200 grit. Afterward, samples were polished with Al_2O_3 paste (average size 3μm) and then etched with an etchant solution consisting of 10g HF, 40g HNO_3, and 50g H_2O. Microstructural analysis was taken from both Olympus BX51TRF-6 model optical microscopy and FESEM scanning optical microscopy for observing phase distribution and worn surfaces, respectively. Structural analysis was performed by X-ray diffraction using a Bruker D8 Advance Twin-Twin diffractometer with Cu-Ka radiation for determining the phases of A, B, and E samples. The hardness of all samples was measured using a TTS Matsuzawa micro-Vickers hardness tester. The hardness test was applied with 5 N forces at a 10-second holding time. Each sample was tested at 5 different points and an average value was calculated for comparing each other. Wear tests of all samples were performed on a custom-made block-on-disk wear test device equipped with DIN 1.2842 cold work steel with a 62 HRC surface hardness and a dimension of Ø80 mm x 40 mm. The wear test parameters were chosen in relation to the references [10,15,18,19]. Specimens were conducted on three different normal loads of 10N, 25N, and 50N under a constant speed of 120 rpm (0.5 m/s) and over a sliding distance of 1500 m. The average friction coefficient and specific wear rate were measured at a 300-meter run time and three test repetitions. The specific wear rate was calculated with the following formula [20];

$$\text{Specific Wear Rate } (SWR) = \frac{\Delta w}{\rho x L x d}$$

which Δw=weight loss (grams), ρ is the density of the test sample (gr/mm^3), L is load (N) and d is the sliding distance (m).

3. Results and Discussion

Optical images of all samples are illustrated in Figure 1. Figure 1(a) shows the as-cast microstructure, which is made of α-Mg as a main phase and β-$Mg_{17}Al_{12}$ as an intermetallic phase. It can be illustrated that the solid-solution treatment led to the dissolving β-$Mg_{17}Al_{12}$ intermetallic phase and became an α+β eutectic phase which is also reported by Kumar et al [21]. The aging treatment was subjected to three different time conditions at a constant temperature. It can be said in Figure 1(c-e) that the number of precipitates is increased with increasing the duration of aging treatment. It could be also observed that the continuous precipitation like fine needle-shaped β-precipitates within the grain led to strong strength through the dislocation-particle interaction by the Orowan mechanism [22,23]. Lastly, the AZ91 alloy subjected to the aging treatment at a 12-hour sample had the highest microhardness while the solid-solution sample show the lowest value (Table 3).

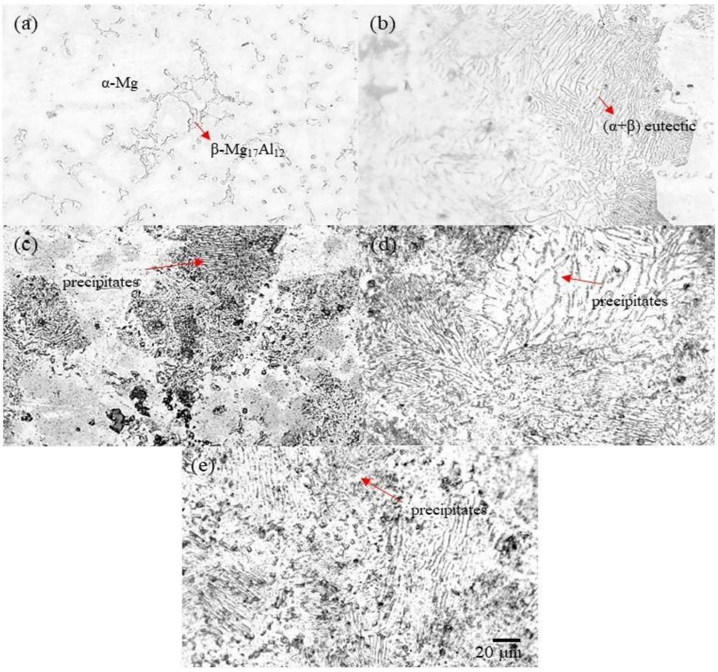

Figure 1. Optical images of (a) As-cast, (b) solid-solution treated, (c-e) 4-, 8- and 12-hour aged samples, respectively.

Table 3. Microhardness results of all samples

Sample Code	A	B	C	D	E
Microhardness (HV)	78.4±1.3	66.8±1.1	88.8±1.5	89.3±1.7	92.9±2.1

The X-ray diffraction analysis of AZ91 alloys with three different heat treatment processes shown in Figure 2 indicates that α-Mg is the primary phase while the second phase is β-$Mg_{17}Al_{12}$. It can be clearly observed that increases in the peak intensity of β-$Mg_{17}Al_{12}$ phases imply that aging treatment gradually promotes the precipitating phase by keeping the temperature below the solidus for an extended period of time (4, 8, and 12hrs). XRD patterns also show that the intensity of α-Mg diffraction peaks in as-cast is lower compared with solid-solution treated AZ91 alloy, suggesting the eutectic phases is retarded. This coincides with the results of Figure 1.

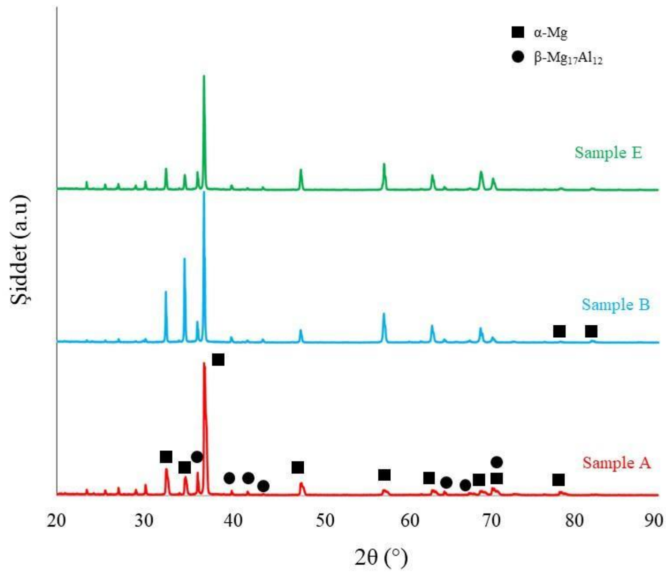

Figure 2. XRD analysis of selected samples indicates two crystalline phases in the crystal structure.

SEM images of worn surfaces of as-cast named Sample A and solid-solution treated samples as Sample B after sliding under a 50N applied load are shown in Figure 3. It can be seen that severe delamination happened during the as-cast AZ91 surfacing alloy wear (Figure 3a). As shown in Figure 3b, the appearance of large amounts of delamination from the contact surface of the sample resulted in an uneven and large amount of wear residue. In addition, the excess of abrasion and plastic deformations in solid-solution samples with low hardness can be interpreted as the reason for the low presence of β-phase. In Figure 3, abrasion and delamination are the two dominant wear mechanisms in the structure. As shown in Figure 3a and 3c, as abrasion usually appears as the main mechanism at low loads like 10 to 50N, this mechanism correspondents to a relatively low amount of mass loss/sliding in the abrasion wear mechanism due to the size of the oxidized particles. While in Figure 3b and 3d, a large amount of irregular lumps and sheets were observed in the load range of 10 to 50N. Since AZ91 alloy has a low cold ductility [24], the continuous deformation of perpendicular cracks causes the release of irregular lumps and sheet-like fragments where delamination can be observed. Especially, solid-solution alloys generated smaller debris than as-cast alloys due to the brittle nature of solid-solution alloys against as-cast alloys.

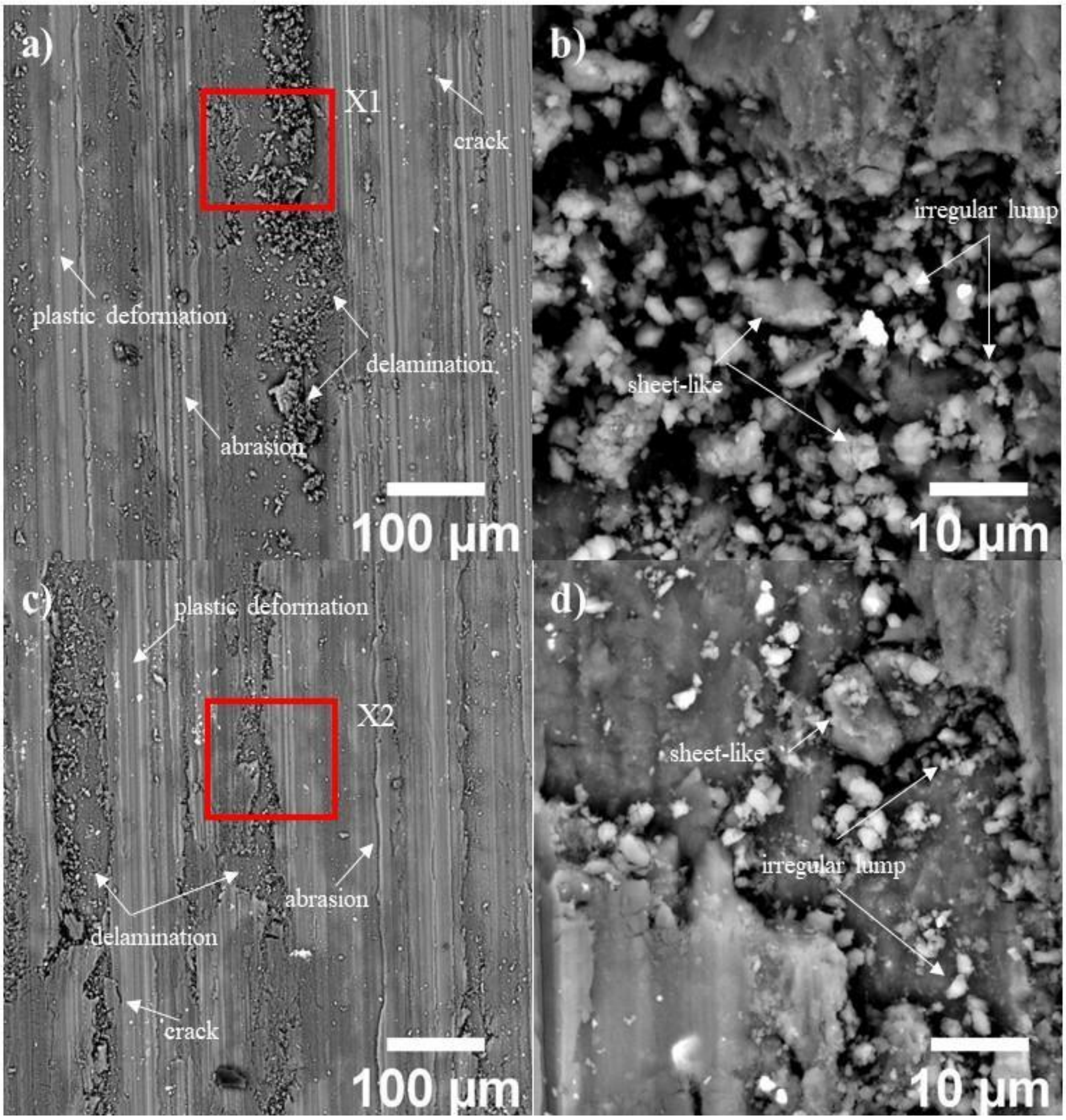

Figure 3. SEM images of worn surfaces under an applied load of 50 N. (a) As-cast, (b) the magnified surface (the red square part of X1 in a), (c) Solid-solution treated sample, (d) the magnified surface (the red square part of X2 in c).

SEM images of worn AZ91 surfaces that were subjected to aging treatment for 12 hours under three different applied load was illustrated in Figure 4. It can be clearly seen that a large number of plastic deformations were observed along the wear direction in Figure 4a-c. These scratches are an abrasive wear behavior caused by the rigid abrasive disc. This is generally observed in wear mechanisms under low loads applied to soft alloys such as magnesium alloys. It is inevitable to see deeper and wider grooves under increasing applied load (Figure 4d-f).

An array of shallow and short cracks appeared on the wear surface when the load was increased to 25 N (Figure 4e), indicating wear delamination. This favored the initiation and propagation of subsurface cracks in the matrix-reinforcement interface area, due to normal and shear stress being transferred through the contacting asperities. With continued wear, cracks grew and eventually caused the surface layer to separate. In addition, the smoothness of the surface throughout the delamination is associated with plastic deformation. Delamination is expected to be even smoother under increased load (Figure 4f). It was also suggested that mild to severe wear changes with specific wear rate variation.

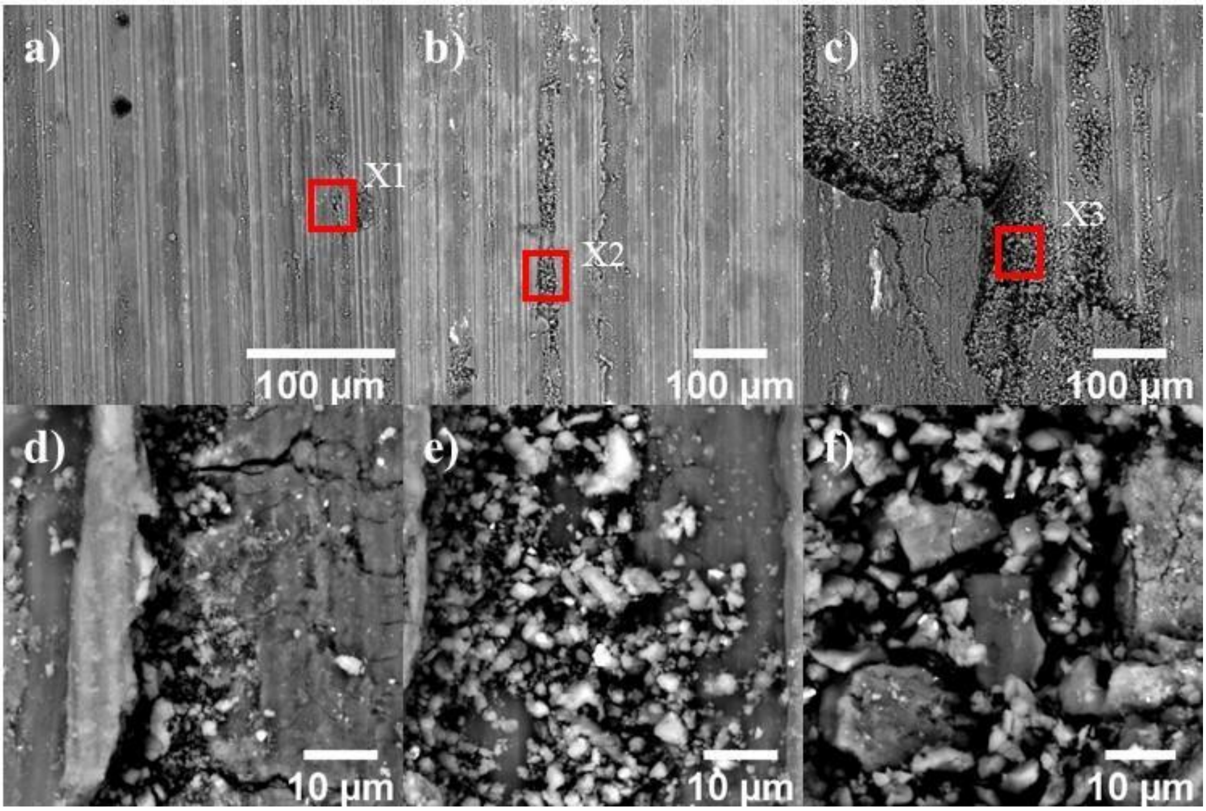

Figure 4. SEM images of aging treat samples at 12 hours worn surfaces under an applied load of (a) 10 N, (b) 25N and, (c) 50 N. (d-e) the magnified surface (the red square part of X1 in a, X2 in b and X3 in c, respectively).

The friction coefficient and specific wear rate of as-cast and four heat-treated samples under different loads are given in Figure 5. In Figure 5a, since the dominant wear mechanisms are abrasion and delamination, this causes a lower coefficient of friction in the load range of 10 to 50N. With increasing applied load up to 50N, grooves parallel to the sliding direction appear on the worn surface and cracks appear on the worn surface, indicating that abrasion and delamination are the dominant wear mechanisms on the surface [25-26].

The friction coefficients of these samples, which were applied with different heat treatment parameters, showed a similar trend as the load increased. It is seen that the change in the friction coefficient between 10N and 25N loading has a very sharp decrease compared to the loading between 25 N and 50 N.

Solid-solution heat-treated AZ91 alloys with Si particles are known to wear rapidly [21], as demonstrated by energy dispersive spectroscopy (EDS) in Table 1. The presence of $Mg_{17}Al_{12}$ precipitates reduces the contact area of the grinding surface. Therefore, the low actual contact area led to the abrasive slipping, resulting in lower friction coefficient values. In other words, with increasing load applied to the samples, thermal softening and subsequent melting of the contact surface at wear also allowed the high frictional heat to slide more easily at the interface. Thus, a higher plastic deformation effect on the wear surface contributed to the continuity of the applied pressure. This situation is also seen in Figure 4 from the width of the fragment rupture areas on the wear surface depending on the applied load.

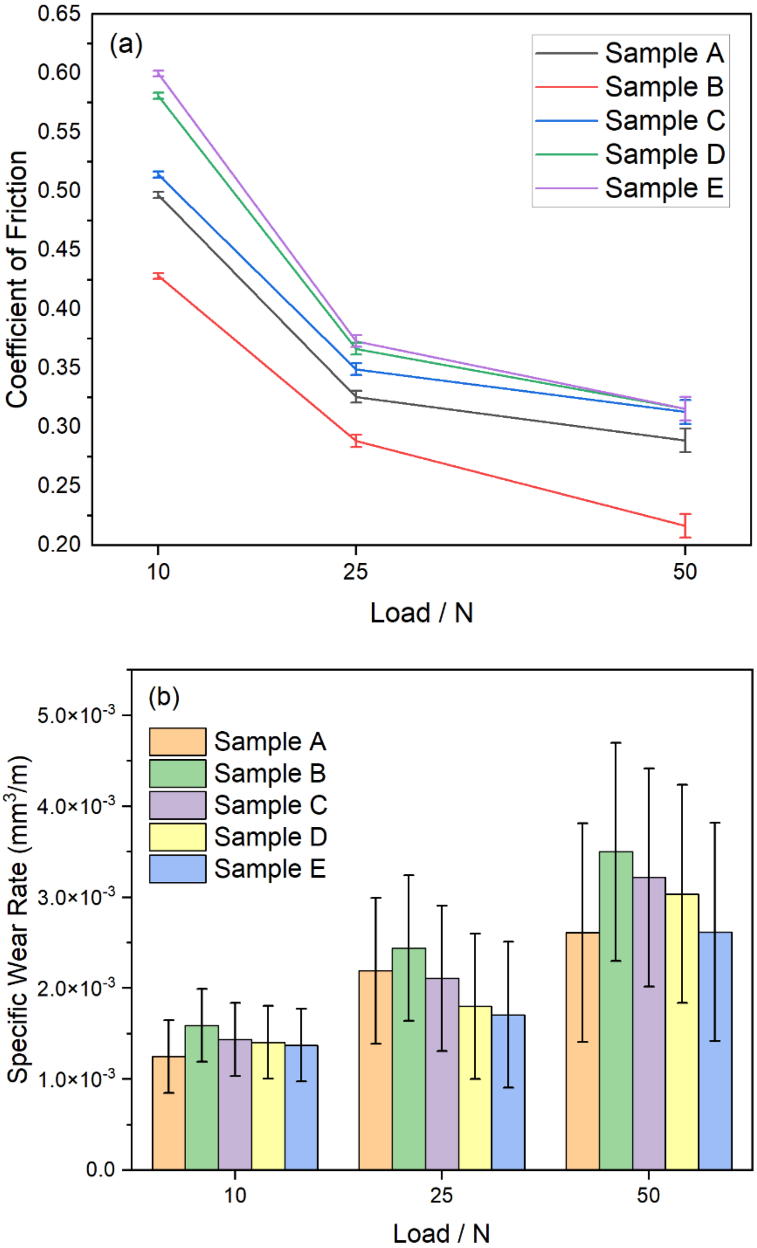

Figure 5. (a) Friction coefficients and (b) specific wear rates of all samples under three different (5 N, 25 N, and 50 N) applied loads.

It is well-known that the hardness is inversely proportional to the wear rate [27]. Specific wear rate behaviors of five different samples under three different applied loads (10N, 25N, and 50N) are given in Figure 5b. According to the Achard equation [27], the specific wear rate was calculated in mm^3/m by dividing volume loss with hardness by the sliding distance with applied load. It was observed that the specific wear rates increased in a linear form with increasing applied load. It can be said that the transition from light wear to severe wear occurs while an increment of the applied load. These results also showed a close follow-up effect with results reported in other studies [28,29].

4. Conclusion

Under different load conditions, this study explored the impact of heat treatment on the wear behavior of AZ91 magnesium alloys. As-cast, solid-solution, and aging samples were compared for microstructures, characteristics, hardness, and wear behaviors. Wear behavior is mainly determined by the β-$Mg_{17}Al_{12}$ phase in the microstructure. The amount of precipitate increased with time in the aging process, which is also confirmed by XRD. Solid-solution samples with low hardness values showed a higher specific wear rate and lower friction coefficients. Moreover, samples subjected to aging improved in wear resistance with time. AZ91 alloys with heat treatment have remarkable properties for automotive engineering parts.

Ethics in Publishing

There are no ethical issues regarding the publication of this study

Author Contributions

Conceived and designed the experiments: GA, DB, MFS. Performed the experiments: GA, MFS Analyzed the data: GA, DB. Wrote the article: GA, DB, and MFS. All authors read and approved the final manuscript.

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