FSP Pass Number and Cooling Effects on the Microstructure and Properties of AZ31

Akbar Heidarpour[1,*], Saeed Ahmadifard[2], Nadia Rohani[2]

[1] *Department of Metallurgy and Materials Engineering, Hamedan University of Technology, Hamedan, 65155-579, Iran.*
[2] *Department of Materials Engineering, Bu-Ali Sina University, Hamedan 65178-38695, Iran.*

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ABSTRACT

In this study, the effects of passes number and cooling during friction stir processing (FSP) on the microstructure and properties of AZ31 magnesium alloy has been investigated. The process was carried out at tool rotational speed, transverse speed and tool tilt angle of 1100 rpm, 52 mm/min, and 3°, respectively. Cooling was performed by water. The microstructure of FSPed samples was explored by optical microscopy, atomic force microscope (AFM), and scanning electron microscope (SEM). Microhardness and tensile tests were used to characterize the mechanical properties of the samples. In addition, wear and corrosion resistance of processed samples were evaluated. Microstructural investigations showed that the cooling process during FSP led to finer and more homogenized grains because of the suppression of grain growth and as a result, hardness and tensile strength were improved. The best properties obtained after 4 passes by an increase in hardness and tension strength equal to 57 and 21%, respectively. In addition, wear characteristics of this specimen was considerably improved and electrochemical tests showed better corrosion resistance due to the decreasing grain size.

1-Introduction

Magnesium alloys as the lightest structural metal offer a great potential for weight reduction for in the aerospace and automotive industries [1]. Unfortunately, these alloy exhibit very limited ductility accompanied by brittle-like behavior at room temperature. One general way to improve ductility and formability of the magnesium sheets is conducted by refining and homogenizing the grain structure of the sheet [2]. Material processing techniques such as the friction stir processing (FSP) [3-6], equal channel angular pressing (ECAP) [7-9] and accumulative roll bonding (ARB) [10] have been proposed that are capable of creating such fine-grained microstructure with expected mechanical properties. FSP has the potential to become an effective tool for microstructural modification of sheet metals [11]. The FSP compared to other processes, has special advantages, including cheap items, the simplicity of tools, easy operation, economic and technological benefits [12]. The FSP is widely applied to Al alloys and to a lesser extent on Mg alloys. For example, Du *et al.* [13] prepared ultrafine-grained microstructures with an average size of about 300 nm in AZ61 magnesium alloy using FSP combined with liquid nitrogen. Alavi Nia et al. [14] has used

* Corresponding author:
E-mail address: a.heidarpour@gmail.com

overlapping passes FSP to create a fine-grained area on the surface of AZ31 magnesium alloy.
Chang et al. [15] developed an efficient cooling system, in which a copper mold, full of liquid nitrogen, was used as a rapid heat sink during FSP. They successfully produced ultrafine-grained AZ31 magnesium alloys with an average grain size of 100–300 nm. In addition, using second phase particles has a considerable effect on the grain refinement during FSP [16, 17]. Some researchers reported that the corrosion resistance of Mg alloys after grain refinement has increased [18–21] while some studies have mentioned decrease the corrosion resistance [22–24]. Due to the considerable fraction of grain boundary in fine grain structures, the corrosion resistance of the material could be reduced. However, formation of a protective passive layer can be found in metals like Mg alloys which it helps to reduce the corrosion rate of fine-grained metals in the neutral electrolyte. Therefore, grain refinement can be adopted as a promising method in corrosion management of reactive metals like Mg alloys. In addition, the level of secondary phase distribution in the alloy or some dissolved elements in the alpha phase (solid solution), which has been occurred in FSP, also improve the corrosion resistance [25].
In the current study, AZ31 Mg alloy was processed by FSP to modify the microstructure. The effects of the pass numbers and cooling on the microstructure, mechanical properties, wear, and corrosion behavior were investigated.

2. Experimental procedure

The chemical composition of AZ31 magnesium alloy used in this study is shown in Table 1. This composition obtained by spark emission spectrometer. Magnesium specimens with dimensions of 200 × 50 mm and a thickness of 6 mm were cut. Fig. 1 shows the die used to investigate the effect of rapid cooling on the FSP. The die was made of copper. The die was placed under the AZ31 specimen and the coolant run through the grooves embedded within the die. The cooling substance was water at 10 ˚C at a rate of 1.2 L/min. The FSP was conducted at a rotational speed of 1100 rpm and traverse speed of 52 mm/min, and the process for 1 up to 4 passes. Specimens of 10 mm length were cut across the FSPed zone and prepare for metallography. The base metal was etched in the etching agent comprised of 5 g of picric acid, 5 ml of acetic acid, 5 ml of distilled water and 100 ml of ethanol.
The microstructural observations were carried out using an optical microscope (Leica, Germany), scanning electron microscopy (SEM, JEOL JSM-840A) equipped with an energy dispersive spectroscopy (EDS), and AFM (DME DS-95-50E).

Table 1. Chemical composition of BM.

Mg	Al	Zn	Mn	Fe
95.63	2.92	0.97	0.46	0.015

Fig. 1. The die used for cooling in the FSP.

Microhardness measurements (Buhler, USA) were carried out on polished specimens by applying 100 g load with 15 sec dwell period. Tensile tests according to the ASTM-E8 standard, were carried out at ambient temperature by a SANTAM 150 tensile machine with the strain rate of 0.003 s^{-1}. Electrochemical studies were carried out using 0.9% NaCl solution as the electrolyte using Gill AC potentiostat (Autolab Type III/FRA2). Potentiodynamic polarization was recorded at a scan rate of 1 mVs^{-1} after stabilizing the system to the open circuit condition for 1 hour. Corrosion current density (i_{corr}) and corrosion potential (E_{corr}) were obtained from the polarization plots using Tafel extrapolation method [26]. Frictional and wear properties of the samples were investigated using a reciprocating friction-and-wear test machine, where AISI 52100 steel with the hardness of 63HRC was used as the pin. The tests were conducted at room temperature and at humidity ranging from 35 to 40 pct with a sliding speed of 0.14 m/s under an applied load of 10 N for sliding distance of 500 m. Mass losses of the disk specimens to the nearest 0.1 mg were measured at 50 m intervals in sliding distance using an analytic balance.

3. Results and discussion

3.1 Microstructure

Fig. 2 shows a photograph captured by the optical microscope of AZ31 base metal. It has a coarse microstructure and the mean grain size is about 56 µm. The base metal shows a typical structure with $Mg_{17}Al_{12}$ phase non-uniformly distributed in the Mg matrix. By conducting FSP, the fine-grained structure resulted because of the material has undergone severe plastic deformation and a dynamic recrystallization as well as breaking of the grain due to the intense plastic strain has occurred [27]. Of course, during friction stir process in absence of reinforcing particles, creation a coarse grain was possible, but because of using a cooling system, grain growth suppressed, and then a fine grain structure resulted. By increasing the passes number, a finer granular structure can obtain. During each FSP pass, severe plastic deformation takes place in the stir zone, causing accumulation of dislocation, creating inward gradient boundaries and a series of grain points, which is a good place to sprout the seeds and make more dynamic recrystallization. During friction stir processing the $Mg_{17}Al_{12}$ phase could partially be dissolved into the magnesium matrix and a strongly different structure due to the intense plastic deformation and the thermal cycle could be formed. The solid solution temperature of the $Mg_{17}Al_{12}$ phase is 473 K [28] and it indicated the possibility of the dissolution of the phase into the matrix. The dissolution of the $Mg_{17}Al_{12}$ precipitates is also significantly influenced by the plastic deformation during FSP. Thus, the dissolution of most of the $Mg_{17}Al_{12}$ phase can take place [29]. Fig.3 shows the AFM image of the surface of stir zone after 1, 2, 3 and 4 pass of FSP. It clearly shows that the grain refinement has been happened and the surface layer is composed of nanograins ranging from 386 to 74 nm in 4 passes FSPed specimen. Fig. 4 shows the surface roughness versus the number of passes obtained by AFM, and as it could be seen clearly from the image that the surface roughness has been reduced by increasing the FSP pass numbers.

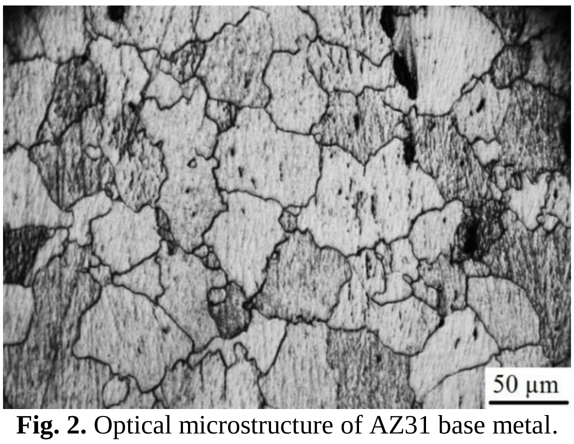

Fig. 2. Optical microstructure of AZ31 base metal.

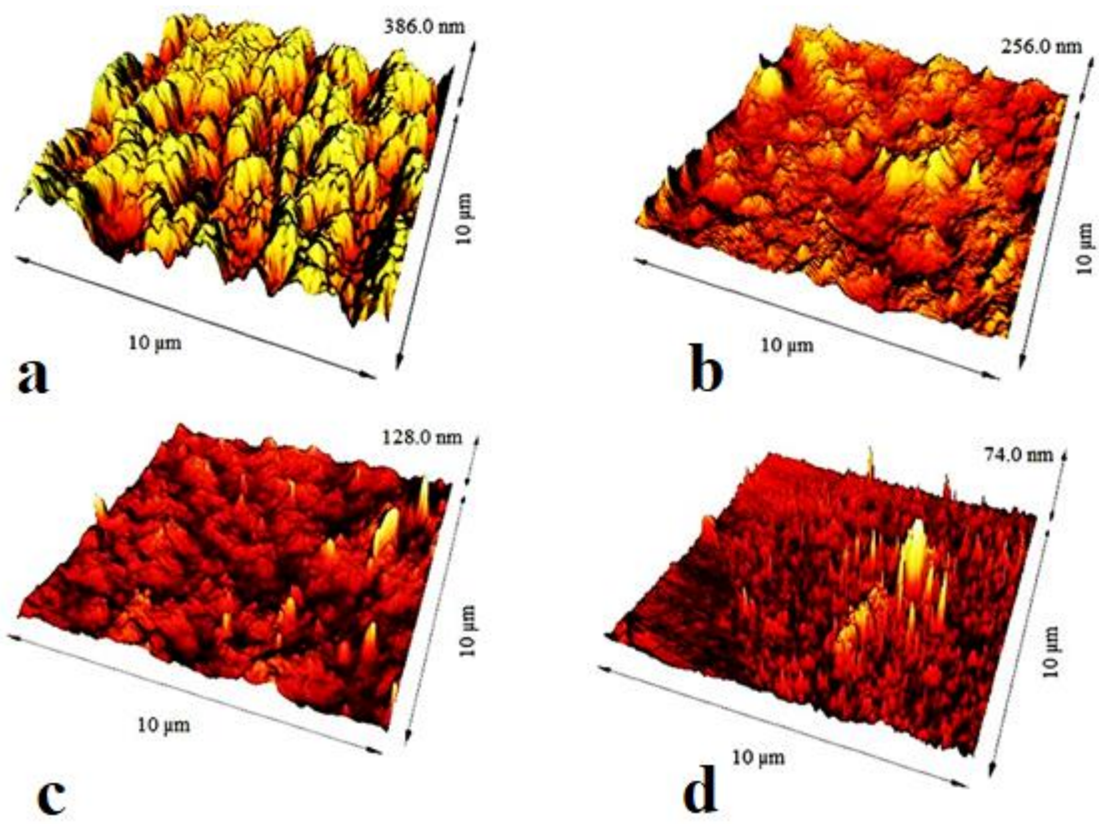

Fig. 3. AFM image of the stir zone (SZ) after (a) 1, (b) 2, (c) 3 and (d) 4 passes.

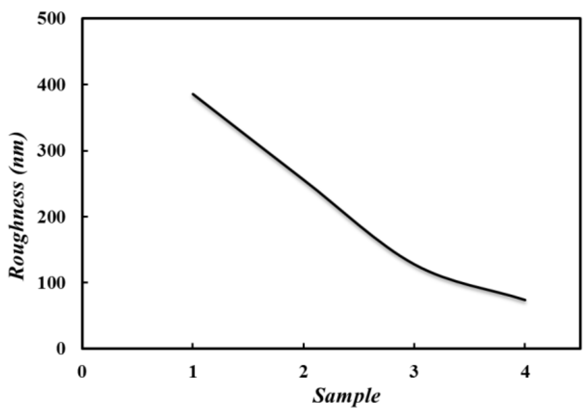

Fig. 4. The effect of ratio of the pass number on the surface roughness of FSP samples.

3.2 Mechanical properties

Fig. 5a shows the results of microhardness profile along the horizontal direction on the cross-section of the FSPed samples. Observation revealed that the microhardness of the friction stirred surface increased with increasing the FSP passes. The evidence shows that there was an obvious change in the microhardness of the stirred structure. Fig.5b shows the hardness profile of a cross-section along the perpendicular direction of the base metal material and FSP specimens. This result shows that FSP can significantly improve the hardness of the base metal and consistent with the Fig.5a. The best result was for the 4 pass sample, which obtained a hardness of about 57% higher than the BM. The highest hardness value obtained for the stir zone due to the reduction in grain size due to the use of the cooling system. According to the Hall-Patch equation, the hardness is reduced by decreasing grain size, i. e. there is an inverse relationship between hardness and grain size [30]. Another reason for increasing hardness after FSP can be attributed to the homogenization of the magnesium matrix and $Mg_{17}Al_{12}$ phase. Moreover, the solid solution strengthening resulting from the dissolution of the $Mg_{17}Al_{12}$ phase into the magnesium matrix also contributes to the improvement of the hardness of the base metal.

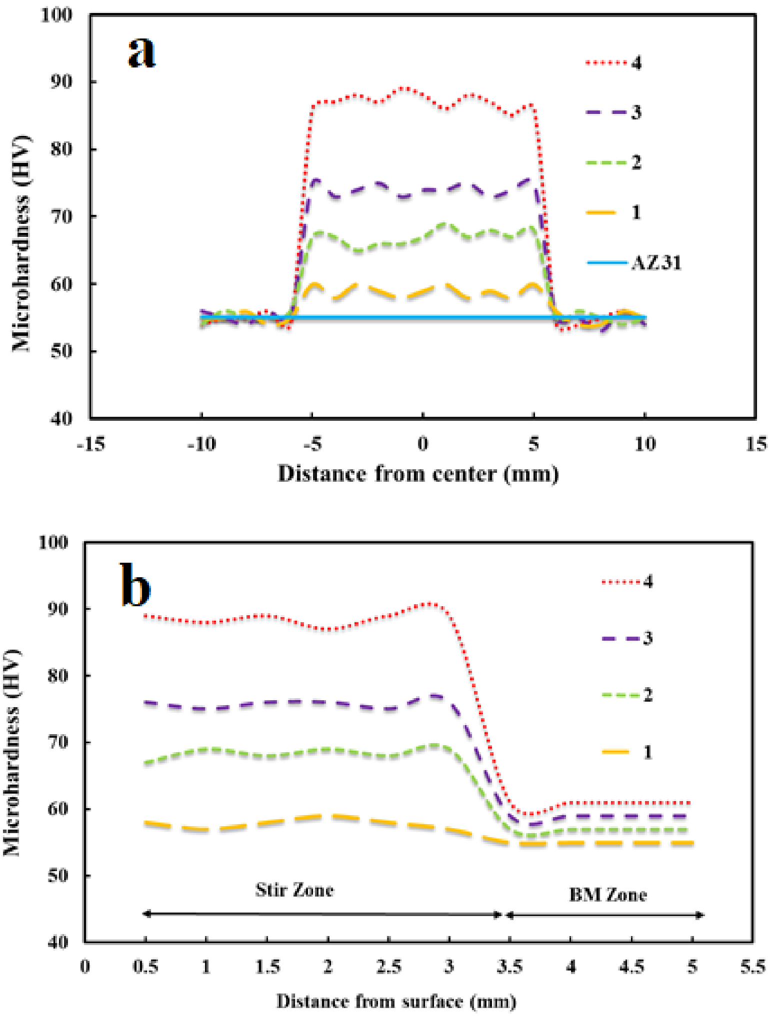

Fig. 5. Microhardness profiles of (a) cross section and (b) depth of FSPed samples.

Fig. 6 shows the stress-strain curves of the AZ31 base metal and FSPed specimens. Table 2 represents the tensile properties of the specimens. As it could be seen, significant changes in the tensile behavior after FSP. Ultimate tensile strength (UTS) of all the FSPed specimens was increased and the maximum tensile strength of 280 MPa was achieved after 4 passes. It is corresponding to a 21% increase in tensile strength of the base alloy. So, this condition was found to be optimum and the achieved hardness, was the maximum hardness value, as mentioned above. In addition, an increase in elongation from 24.43 to 49.53 % was observed. The maximum elongation of 49.53% was achieved for 4 passes FSPed specimen. This considerable improvement of tensile strength is mainly attributed to the significant breakup and the dissolution of the $Mg_{17}Al_{12}$ phase and the remarkable microstructural refinement and homogenization. The dissolution of the coarse $Mg_{17}Al_{12}$ phase can significantly reduce the formation and the growth rate of cracks, and dramatically improve the ductility of the FSP specimens. It is worth noting that the mechanical characteristics of FSPed specimens were enhanced by increasing passes.

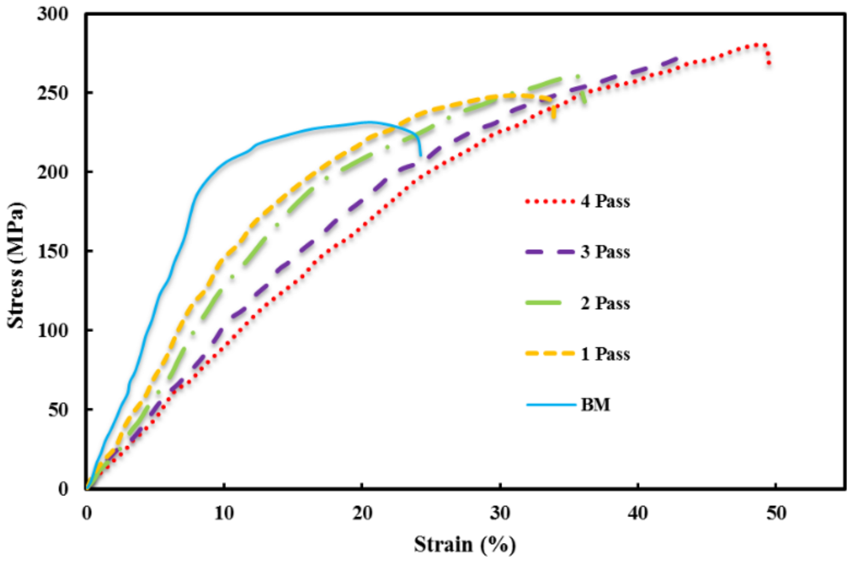

Fig. 6. Stress- strain curve of base metal and FSPed samples

Table 2. Mechanical properties of base metal and FSPed samples

Sample	Total elongation (%)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)
BM	24.23	102.3	231.6
1 Pass	33.85	91.5	248.5
2 Pass	36.2	76.3	260.4
3 Pass	42.89	64.9	272.6
4 Pass	49.53	57.6	280.3

3.3 Corrosion

Fig. 7 shows the polarization curve of the BM and FSPed samples. The electrochemical parameters of the samples obtained from potentiodynamic polarization tests are presented in Table 3. Clearly, shift of potential to more positive values and lower the current density for FSPed samples compared to AZ31 base metal was founded. According to Table. 3 corrosion rates decreased by increasing pass numbers. Always the kinetics of oxidation or reduction reactions are controlled by a part of the process that has the lowest rate. By conducting the FSP, while creating of fine grain structure, the passive layer on the surface of samples will be formed. In fact, this passive layer disrupts the electrochemical reaction and ultimately leads to a reduction in the corrosion rate. Similar results of a slight increase in the corrosion resistance for FSPed AZ31 Mg alloy were reported [31]. Actually, dissolution of $Mg_{17}Al_{12}$ phase during FSP decrease the galvanic corrosion and enhanced the corrosion resistance after FSP. In addition, grain refinement has a significant effect on corrosion behavior [18, 19].

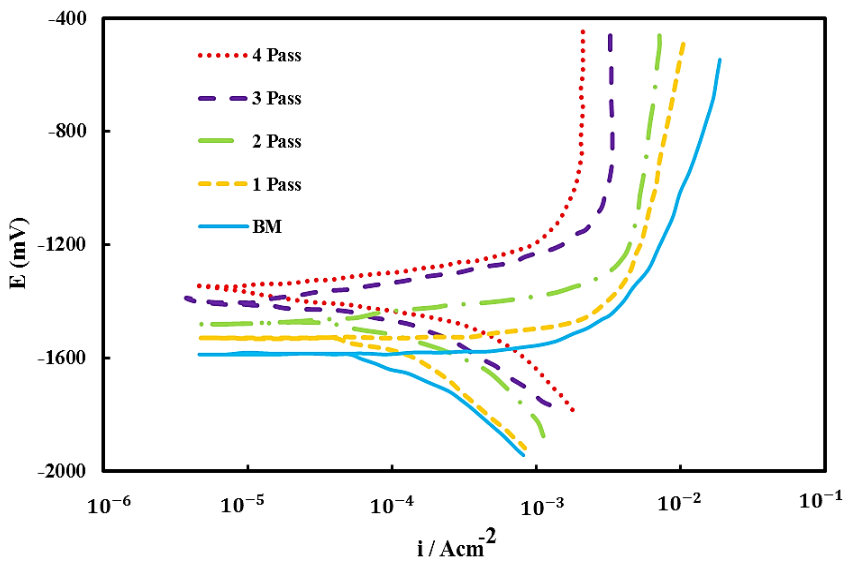

Fig. 7. Potentiodynamic polarization curves of the BM and FSPed samples.

Table 3. Electrochemical parameters and corrosion rates of the samples obtained from Potentiodynamic polarization tests.

Sample	i_{corr} (10^{-5} A/cm^2)	Ecorr (mV)
BM	9.31	-1580.6
1 Pass	9.11	-1530.2
2 Pass	6.32	-1482.4
3 Pass	3.21	-1400.3
4 Pass	2.99	-1349.8

3.4 Wear

Fig. 8 shows weight loss in terms of slip distance for base metal and FSPed samples. In general, the results show that by increasing sliding, the weight loss of specimens is even greater, however, the variation intensity for each sample is different. According to the results, the weight loss of FSPed samples was lower than that of the base metal. Also, by increasing the number of passes, weight loss has decreased, which indicates increased wear resistance of FSPed samples. The lowest weight-loss belongs to the 4 passes sample, which is less than 75% lower than the base metal. Also, the slope of the graphs at the final stages of the test indicates that in the FSPed samples, the weight loss has reached a steady state, but in the case of base metal, the weight loss graph has ascending manner. Fig. 9 shows the variation of wear rate versus the sliding distance of the base material and FSPed specimens. It could be seen that the wear rate is considerably decreased in the FSPed specimens compared to the base metal and by increasing passes number the wear resistance increased. Actually, it is clear that the rate of wear of FSPed samples is lower than the base metal and by increasing pass number the wear rate decreases. According to Archer's law [32], by increasing the hardness of the material, the wear rate decreases and as mentioned above, by increasing the pass number of FSP, the microhardness values increase.

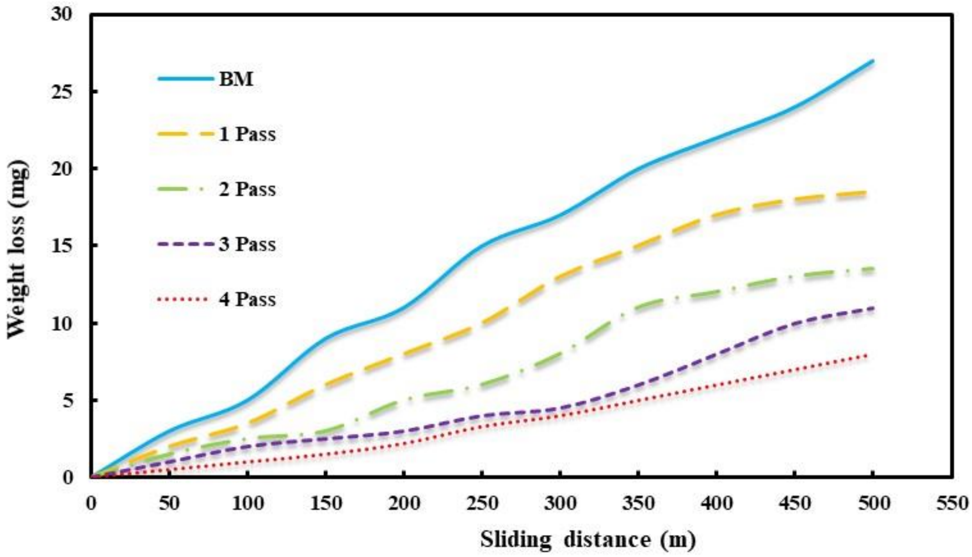

Fig. 8. Variations of wear rate with the sliding distance for base metal and FSPed specimens.

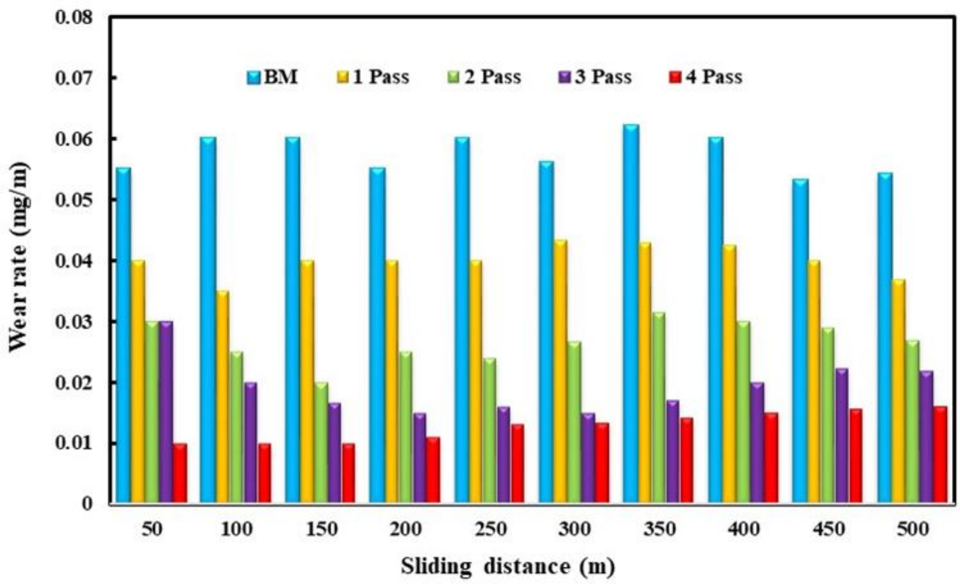

Fig. 9. The average wear rate (weight loss / slip distance) versus sliding distance for base metal and FSPed specimens.

Therefore, the minimum wear rate observed for 4 passes FSPed specimen. The major factors that contributed to the reduced wear rates for 4 passes FSPed alloy could be attributed to the microstructural refinement resulting in higher hardness, greater work hardening capability and improved ductility. It can be concluded from these results that the wear resistance of the AZ31 Mg alloy significantly was enhanced by the friction stir processing due to the intensive grain refinement during the process [33].

Fig. 10 shows the variations of the friction coefficient for the base metal and the FSPed samples as a function of sliding distance. The results of wear characteristics of base metal and FSPed samples were presented in Table 4. The friction coefficient of base metal was 0.74 and decrease after friction stir processing. Minimum friction coefficient obtained after 4 pass FSP, which is about 87% lower than the base metal. This values show the impact of FSP and confirmed that microstructural evolution and hardness changes directly affected the wear behavior. The large changes in friction coefficients are caused by the periodical accumulation and elimination of wear debris on the worn track and repeated banding structure in the tool traveling direction [34-36]. Fig. 11 shows the SEM of worn surfaces after the wear test of the base metal and FSPed samples. This figure reveals deep grooves scratching parallel to each other in the sliding direction. The abrasive wear features such as micro-cutting, plowing and grooves are noticeable on the surfaces.

Table 4. Wear characteristics of base metal and FSPed samples.

Sample	Weight loss (mg)	Wear rate (mg/m)	Friction coefficient
BM	15.3	0.057	0.74
1 Pass	11.1	0.04	0.66
2 Pass	7.55	0.026	0.52
3 Pass	5.2	0.019	0.31
4 Pass	3.85	0.012	0.26

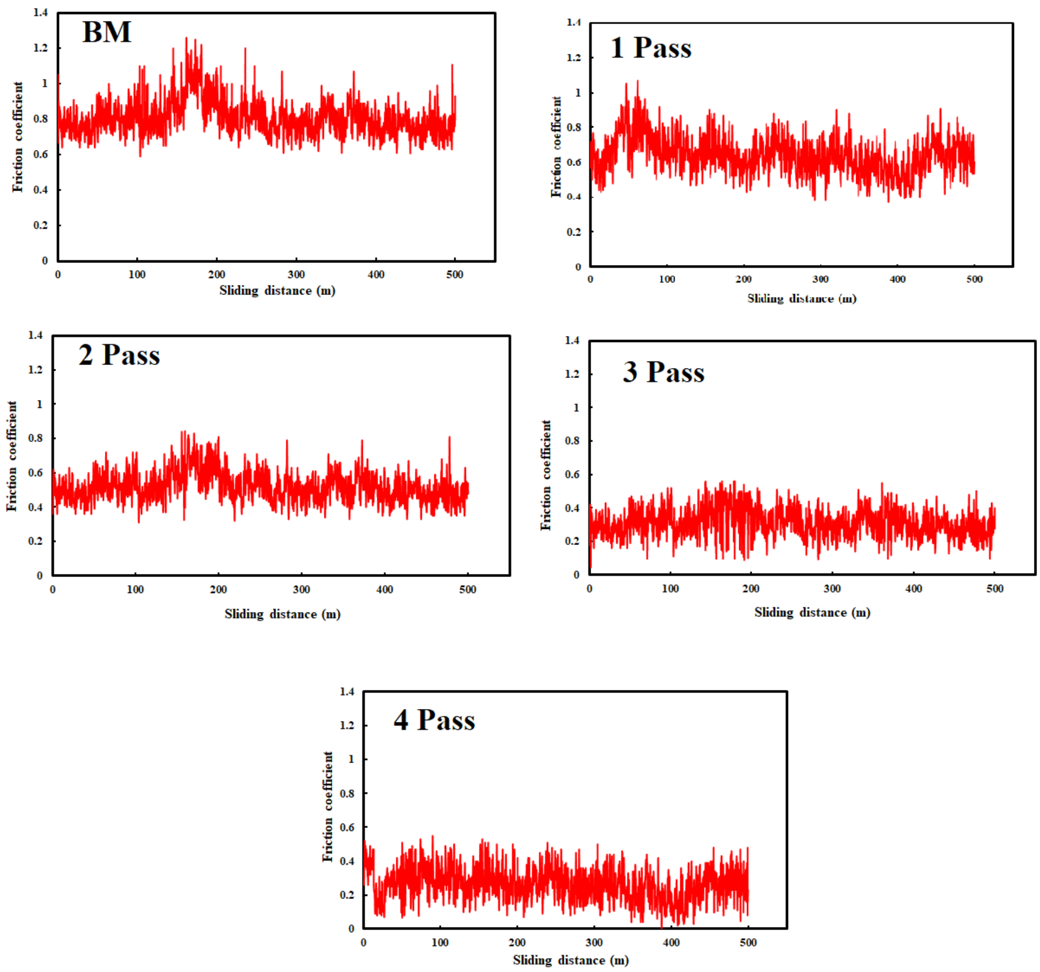

Fig. 10. Friction coefficient versus sliding distance for different specimens.

These features can be observed to be more intense at base metal. In addition, the presence of plastic deformation indicated by the lip formation was detected. By increasing the pass number of the FSP, the extent of these features was diminished. The presence of fine grain structure (in 4 passes FSPed sample) may have contributed towards grain boundary strengthening in accordance with the Hall–Petch relation. This mechanism is the major phenomenon responsible for enhanced mechanical properties, such as hardness and wear of the FSPed alloys [32-34].

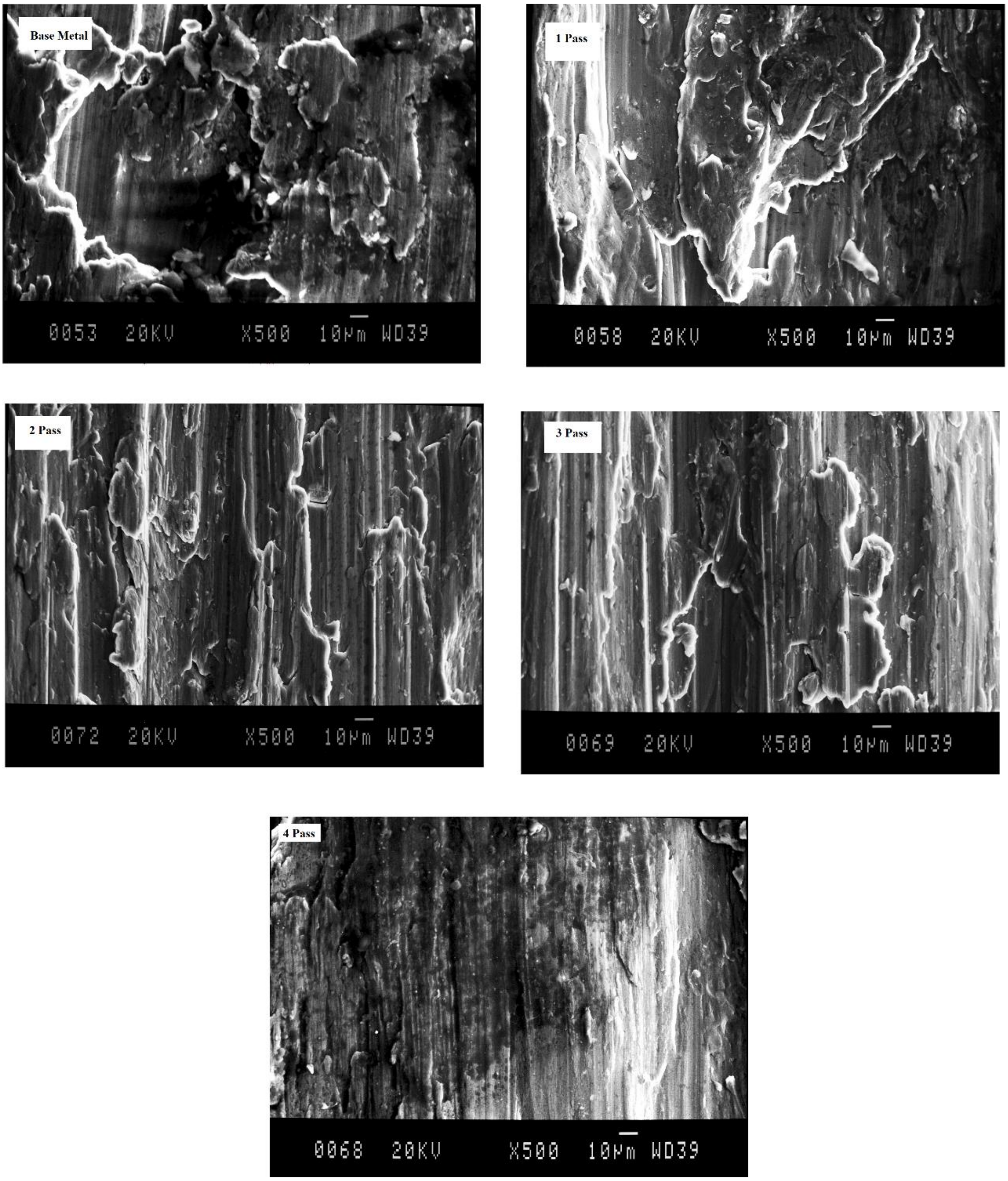

Fig. 11. SEM micrographs of worn surface of base metal and FSPed samples.

Conclusions

In the study, the effect of pass number and cooling rates on the microstructure, mechanical properties, corrosion and wear behavior of AZ31 Mg after FSP was studied and the following results obtained:

1- By using the cooling system, the FSP can be done in the number of passes more and more strain forward, and then, it reduced the process time. In addition, by using a cooling system and performing a process in the number of passes, a very fine-grained structure obtained.

2- The results of hardness and tensile strength showed that by increasing the number of passes, more favorable properties could be obtained. The best result obtained for the 4 passes sample, so that the hardness and tensile strength, increased by 57 and 21%, respectively, compared to the BM.

3- The corrosion results showed that by increasing the number of passes, the corrosion resistance increased due to decreased grain size, which increases the speed of the formation of the passive layer. Also, according to the results, the current density and corrosion potential in the BM, decreased from 9.31 (10^{-5} A/cm^2) and -1580.6 (mV) to 2.99 (10^{-5} A/cm^2) and -1349.8 (mV) for 4 passes FSPed specimen, respectively.

4- The major factors that contributed to the reduction in the wear rates for FSPed specimens were found to be microstructural refinement resulting in higher hardness, greater work hardening capability, and improved ductility. It was found that maximum mass loss/sliding distance (wear rate) occurred for the base metal. Moreover, the wear rate was found to decrease with increase in pass number. Abrasive wear was found to be dominant wear mechanisms.

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