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Effect of Equal Channel Angular Pressing on Microstructure and Mechanical Properties of Aluminum Based Composite Materials

Saman AHMADZADEH[1,*], Ömer KELES[1], Necip CAMUSCU[2]

[1]Department of Mechanical Engineering, Gazi University, Ankara –TURKEY

[2]Ministry of Science, Industry and Technology, Ankara – TURKEY

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Abstract

In this study, Equal Channel Angular Pressing (ECAP) of Al-Mg-Zn/SiCp composites was experimentally investigated. Using powder metallurgy (PM), different sets of Al-Mg-Zn/SiCp composites with different concentrations of Mg and SiC were prepared by hot pressing, which are then subjected to ECAP process in a furnace at 373K up to 1 or 2 passes using route C. Post experimental evaluation of mechanical properties showed that material hardness was increased following each ECAP pass. However, no substantial change was observed in wear resistance of the composites. Despite the lack of significant effect on the wear resistance, some small variations in hardness and wear resistance among different sets of samples are observed, which are weakly related to Mg and SiC content in the composites.

1. INTRODUCTION

Ultrafine-grained materials with nanoscale grain sizes have been one of the important goals of producing nanomaterials in recent decades [1-4]. Lack of high efficacy in directly employing nanomaterials into the manufacturing processes has called for the use of innovative alternatives such as severe plastic deformation methods. Equal Channel Angular Pressing/Extrusion (ECAP/ECAE) process is one of the commonly used method and it is first introduced by Segal in 1977 [5-7]. This process is based on applying severe shear strain on the material leading to deformation of grain and grain boundaries. This resulted in the modification of some mechanical characteristics especially an increase in fatigue resistance [8-9].

Some of the factors affecting the plastic deformation are: inner and outer curvatures of ECAP die, number of pressing, pressing temperature, pressing speed, and pressing routes. In the process, each pressing consists of several passes; in which sample might be intentionally rotated around its longitudinal axis. Based on this local rotating angle, these routes are named as A, B_A, B_C, and C. Different local rotations have significant effects on the sample as a result of change in planes and directions of shear which in turn lead to different microstructures and characteristics [10-11].

Based on Hall-Petch equation, yield stress (σ_y) is inversely related with the average grain size. However, this law in nanomaterials and small grains is not observed [12]. Hall-Petch equation is defined as [13]:

$$\sigma_y=\sigma_0+kd^{-1/2} \tag{1}$$

In which, σ_0 is the stress resulted from dislocation movement, k is a material constant, and d is the average grain size. Consequently, transforming the microstructure to ultrafine-grain is an effective way to increase the yield strength of the materials. An important advantage of severe plastic deformation methods, which distinguishes them from commonly used thermomechanical processes, is their ability to keep the sample dimensions intact. Thus, a billet can undergo a severe plastic deformation multiple times which increase its capability to produce fine microstructure and homogeneous grains. The ECAP die geometry used in this study consists of two identical channels connected in series with a defined angle between them (Fig. 1). The inner angle of the channel (the angle between the channel centerlines) shown by Ø is set in between 75 and 150 degrees depending on the initial hardness of the alloy while the outer angle (the angle of the

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*Corresponding author, e-mail: saman.gazi@gmail.com circular sector connecting two channels) shown by φ is generally defined between 0 and 20 degrees. The relation between the inner and the outer curves and also the number of pressing passes for each sample defines the level of overall strain after severe plastic deformation process by the following equation [10]:

$$\varepsilon = {}^{N}\!/_{\sqrt{3}} \left[2ctg\left({}^{\emptyset}\!/_{2} + {}^{\varphi}\!/_{2} \right) + \varphi cosec\left({}^{\emptyset}\!/_{2} + {}^{\varphi}\!/_{2} \right) \right] \tag{2}$$

As it can be inferred from Fig.1, at the meeting location of two channels, a simple shear is applied on billet that leads to change in the microstructure and also mechanical properties.

In recent decades, favorable characteristics of aluminum composites such as their light weight and high corrosion resistance, have led to a significant increase of their applications in many engineering applications. In recent years, performing severe plastic deformation methods on composites in order to obtain desirable characteristics in different industries, have led to a broad application of aluminum composites in different fields [14-15]. Although, significant progress has been made in the development of ECAP in recent years, there has been limited research devoted for the production of ultrafine-grained aluminum based composites. In this study, Al-Mg-Zn/SiC_p composite and Al-Mg-Zn alloy produced by powder metallurgy (PM) were exposed to severe plastic deformation and the corresponding change in their mechanical and structural properties were investigated in detail.

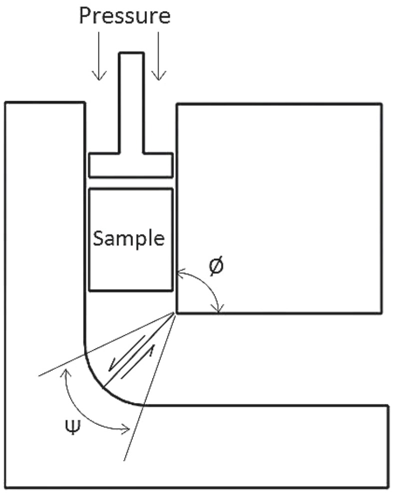


Figure 1. *Mechanism of ECAP process*

2. MATERIALS AND METHODS

Samples were prepared by PM method. In this method, using a small powder size with high surface area is very important in sintering. Mean powder sizes of aluminum, silicon carbide, zinc and magnesium, measured by laser particle size analysis were 10.22µm, 32.67µm, 6.66µm, and 45.08µm, respectively. Furthermore, powders had high purity levels lying within the range of 99.6% and 99.99%. Powders were mixed in Turbula by ZrO_2 balls, and then underwent a hot press-sintering process. In order to increase the density, a liquid sintering mode was used. Therefore, a small amount of Zn powder was also added to the samples. Samples were categorized into two groups which are reinforced (containing silicon carbide as the reinforcement) and non-reinforced. Compositions of the samples in weight percent are presented in Table 1.

Table 1. *Compositions of the samples.*

Al	SiC	Mg				Zn
Bal.	0%	0%	1.5%	3%	4.5%	0.25%
Bal.	5%	0%	1.5%	3%	4.5%	0.25%

Hot pressing was performed at 873K for 25 min. In Fig. 2a, dimension of a sample before ECAP is presented. The ECAP die had an inner angle of 90 degree and outer angle of 37 degree followed by a channel with a 12 mm diameter. ECAP process was carried out at 373K with a constant pressing speed of 5 mm/min. To reduce the friction between billets and die channels, a lubricant based on MoS_2 was applied on the contacting surfaces. All samples were subjected to 1 or 2 passes of ECAP using route C at which samples were rotated around their longitudinal axis by 180° after each pass. An ECAPed sample is shown in Fig. 2b. After completing the ECAP operation, Brinell hardness test (Willson-Wolpart 930N machine) was performed on the samples. In addition, by employing pin on disc method and using ASTM G99-05 standard, wear resistance of the samples was evaluated. In non-reinforced samples, AISI 52100 steel with a hardness of 62 HRC and for the samples with reinforcement elements, sandpaper of #400 grit on disc as the abrasion material was introduced. Wear tests were carried out at room temperature (294K) under a 60% humidity. Moreover, wear tests were performed under a load of 30 N for 1000 meter while disk's speed was maintained at 10 mm/s. The mass loss of samples after 1000 meter was measured by using Shimadzu AUW 320 balance with an accuracy of 0.1 mg. In order to secure a clear image of grains and their sizes, an HF solution was used as an etcher. Finally, sample microstructures were first studied by using an optical microscope **(**Olympus D470) and then by SEM (JEOL JSM-6060LV).

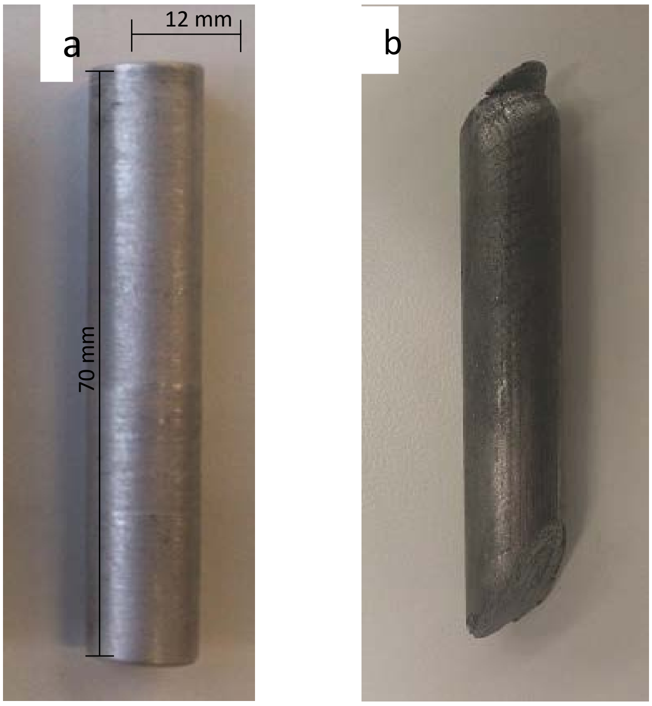


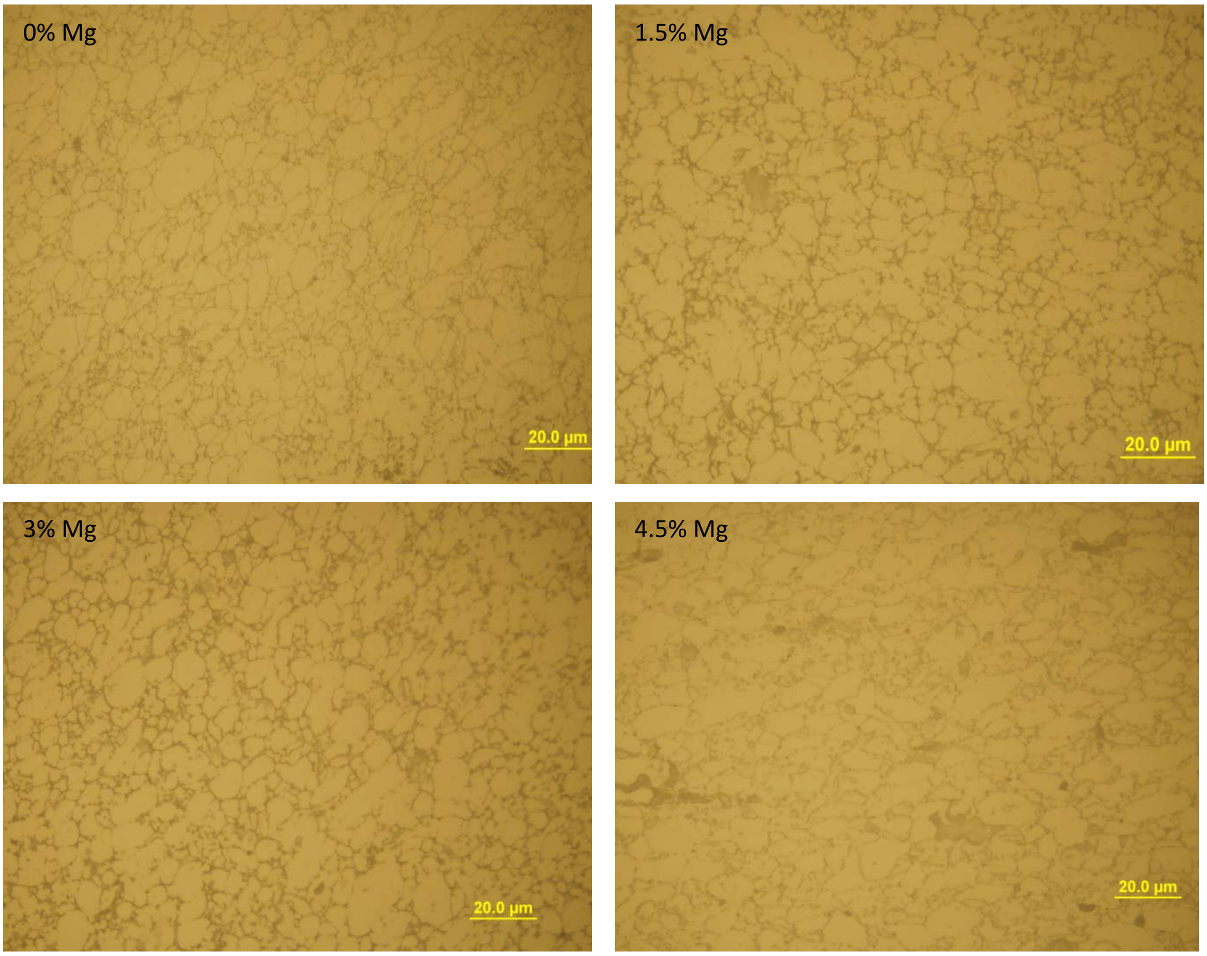
Figure 2. *Sample dimensions used in the experiments:*
a) Before ECAP; b) After ECAP.

3. RESULTS AND DISCUSSION

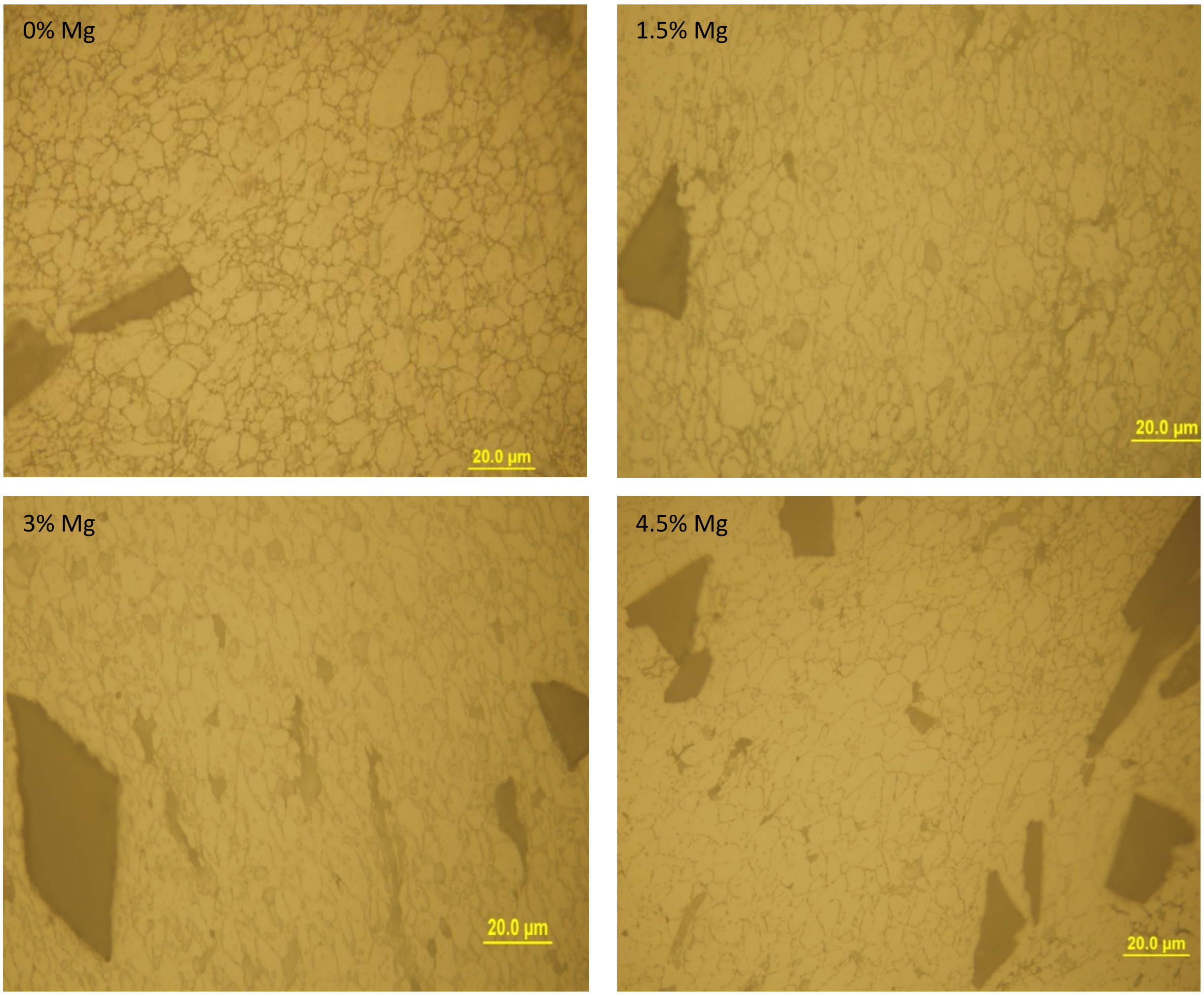
The findings of the present study are provided under the appropriate sub-headings including material microstructure, hardness, and wear tests.

3.1. Material Microstructure Prior to ECAP

Sample microstructures before the ECAP operation captured by an optical microscope are shown in Figure 3a and 3b. In this figure, equiaxed grains with an average grain size of 15 µm are observed. Moreover, SiC particles in reinforced samples are vivid and homogenous. In Al-Mg systems, β phase precipitates are observed on dislocations and also on grain boundaries growing rapidly and turning into incoherent large particles. For this reason, they may not have any significant influence on the mechanical properties of the samples.



(a)



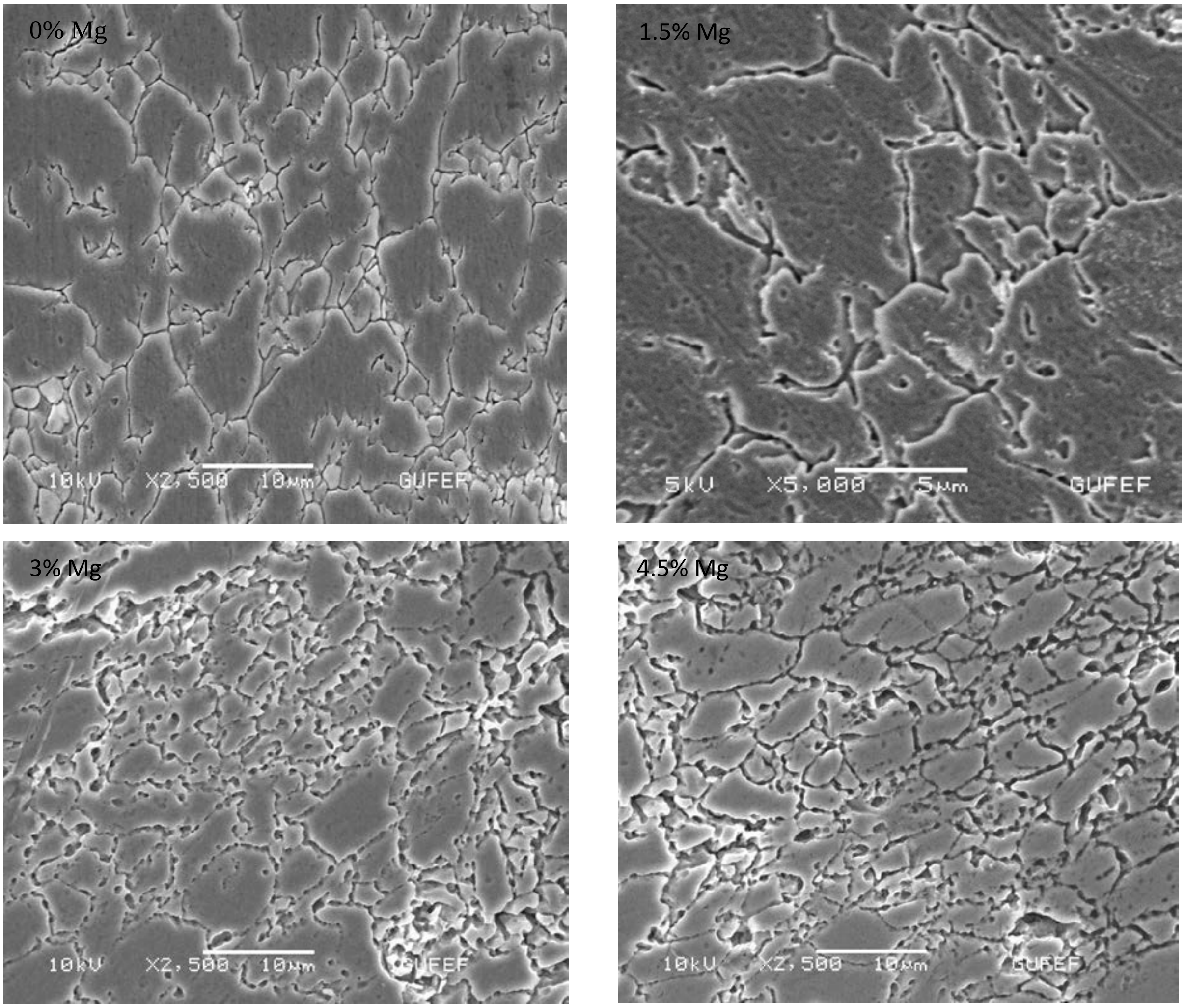
(b)

Figure 3. *Microscopic structure of samples before ECAP:*

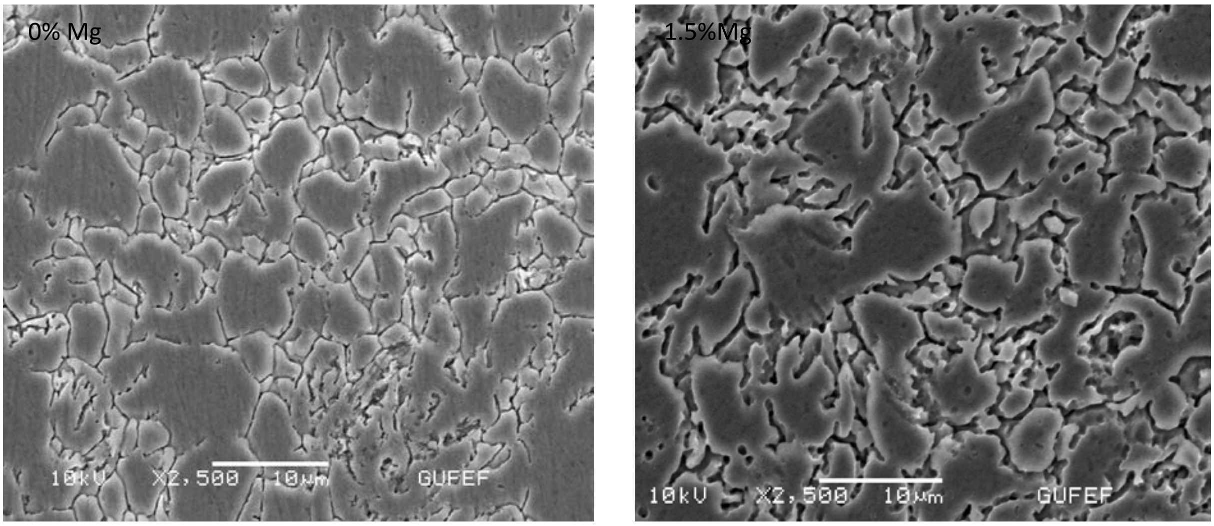
a) non-reinforced samples; b) reinforced samples.

3.2. Microstructure of the Samples After ECAP

Microstructure of the samples after two passes of ECAP using route C at 373K are shown in Figure 4a and 4b. It is evident that after 2 passes; microstructures are not transformed into the homogenous ultrafine-grained form and the process needs more passes. However, SEM micrographs depict that the average grain size throughout all samples is in the order of 2 µm. Noting the participation of sliding plates during deformation process, the feature of deformed structures is inhomogeneous. As a result of severe strain the dislocation density through the material has increased and these dislocations move during the deformation and cause sliding of the planes in the grains. Thus, the dislocations move out of the grains and settle in the grain boundaries. As a result of increased dislocation density on grain boundaries, optical microscope cannot spot grains and grain boundaries simultaneously and so the SEM micrographs are used. The decrease in the grain size is associated with a severe plastic deformation during ECAP process, which is resulted from internal energy consumption to produce sub-grain boundaries. Moreover, newly generated dislocations are located close to each other and they act like grain boundaries. High angle grain boundary is the dominant phenomenon in these microstructures, which is consistent with the previous findings [16].


0% Mg
10kV X2,500 10µm GUFEF
1.5% Mg
5kV X5,000 5µm GUFEF
3% Mg
10kV X2,500 10µm GUFEF
4.5% Mg
10kV X2,500 10µm GUFEF

(a)


0% Mg
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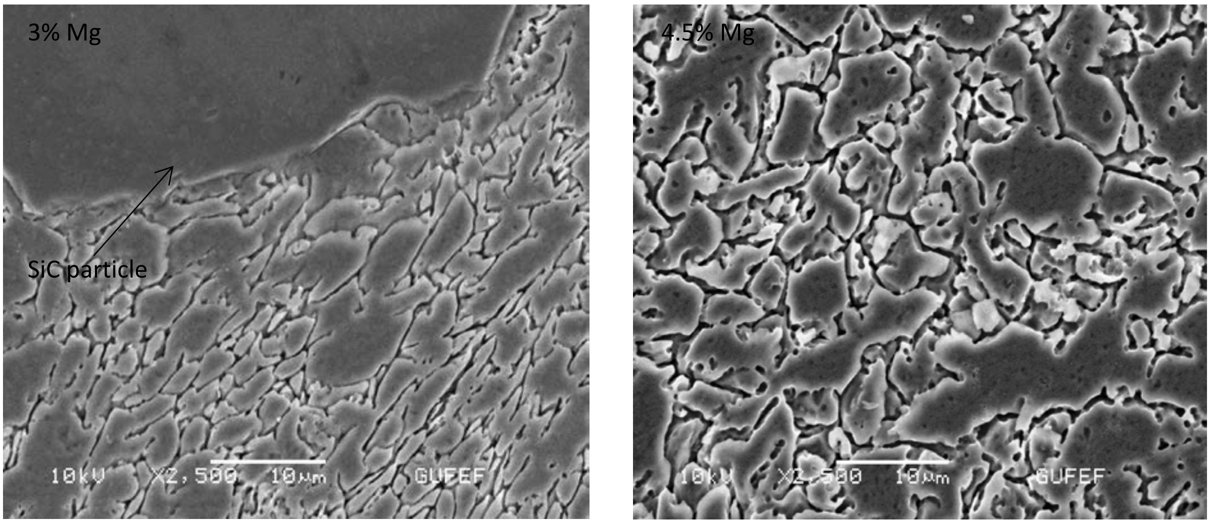
1.5%Mg
10kV X2,500 10µm GUFEF

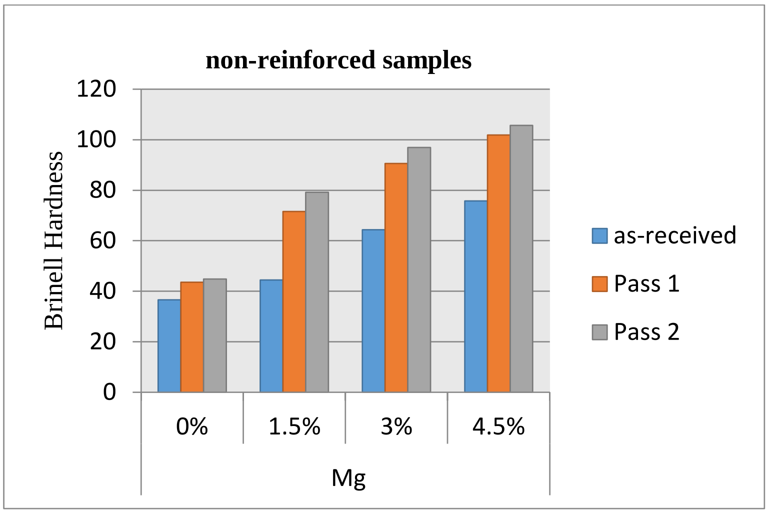


(b)

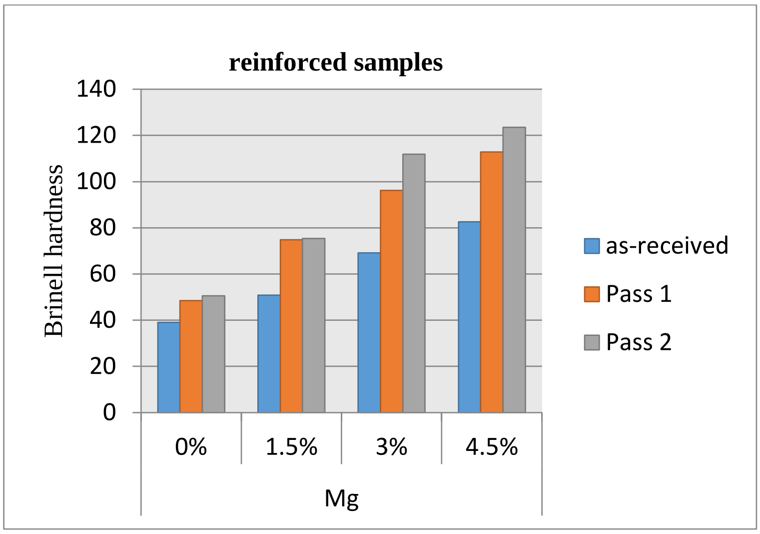
Figure 4. *Microscopic structure of ECAPed samples (by SEM):*
a) non-reinforced samples; b) reinforced samples.

3.3. Hardness Results

Brinell hardness results of all samples prior to and after ECAP are given in Fig. 5. As seen in this figure, in comparison to as-received condition, hardness of samples increased substantially. The increase in the hardness is attributed to two main reasons. One is the mechanism of the hardness strain system and the other one is the increase in produced dislocation density both of which are relatively higher in the first pass as compared to the subsequent passes. Consequently, the major part of hardness increase during ECAP takes place in the first pass. [17]. ECAP results in an increased density of dislocations, which is the perfect location for precipitations. Thus, there will be improved distribution of precipitations throughout the material. It is also worth noting that addition of Mg boosts the hardness by increasing the amount of precipitation. It is also evident that the samples containing reinforcement elements are approximately 15% harder than non-reinforced samples with the same amount of Mg. The greatest hardness value (123 BHN) occurs for the sample consisting of Al-4.5% Mg-0.25% Zn-5% SiC; in which case, it shows a 45% improvement compared to the primary mode (as-received).



(a)



(b)

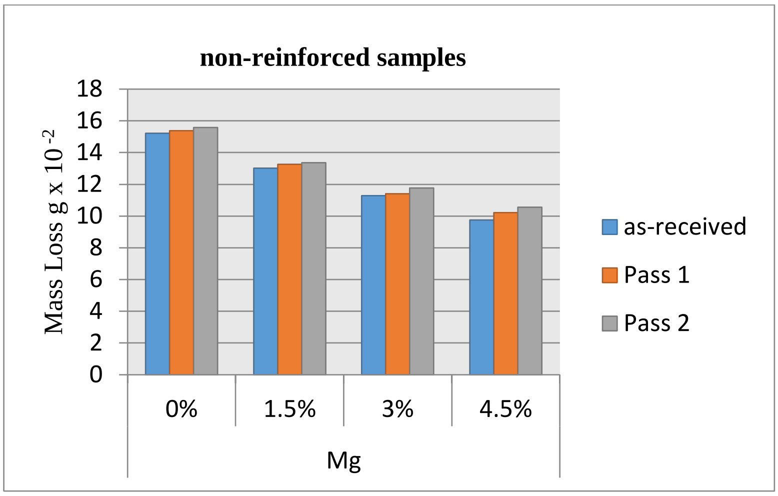
Figure 5. *Brinell hardness of the samples:*
a) non-reinforced samples; b) reinforced samples.

3.4. Wear Test Results

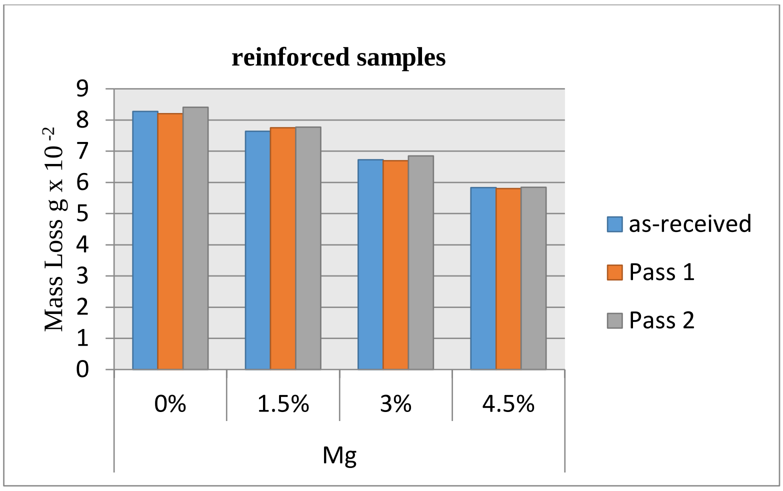
Mass losses during the wear tests for all samples before and after ECAP are illustrated in Fig. 6. From the Archard equation, the mass loss of the material can be estimated theoretically as follows [18]:

$$V = K({}^{SL}/_{3H}) \tag{3}$$

Where H is material hardness, L is the normal load, S is the total distance, K is the dimensionless wear coefficient, and V is the worn volume of the sample. According to Eq. 1, since the ECAPed samples have a greater hardness, they suffer from a smaller mass loss. However, this finding contradicts to that is shown in Fig. 6. In this case, the ECAPed samples wear resistance are observed to be lower than their as-received condition and also sample mass loss increases as the number of passes increase. Overall, it can be stated that ECAP has an adverse effect on the wear strength of the samples, though it may not be significant in all cases. The contradiction between theoretical results obtained from the Archard equation and the wear test results could be associated with two main reasons as consistent with the early work [19]. As it is shown, grain size decreases and also grain refinement occurs in microstructure after ECAP. Due to their inherent properties during the wear test, small grains can be easily separated from the materials surface as compared to the large grains and, consequently, mass loss of ECAPed samples becomes higher than that of the non-ECAPed ones. On the other hand, because of ECAP, material ductility decreases which is an effective factor on weight reduction during wear test. The reason for why Archard equation fails to explain the changes in material characteristics during ECAP is that it ignores the influence of the refined grain size microstructure and introduction of high energy non-equilibrium grain boundaries after ECAP and finally increase in the grain boundary mis-orientation angle as the ECAP passes are increased [20-23]. These results are in accordance with the findings of a recent study [24]. It is also shown that the samples with SiC reinforcement having 45% are more resistant to wear and they have less mass loss than the non-reinforced samples.



(a)



(b)

Figure 6. *Mass loss of samples,*
(a) non-reinforced samples (b) reinforced samples

4. CONCLUSION

In this work, ECAP was presented as an effective procedure in producing ultrafine-grained materials with high angle grain boundaries while improving the precipitations and dislocation distribution in the material microstructure. After two passes of ECAP, average grain size dropped from 15 µm in the as-received condition to 2 µm. In addition, the precipitations generated block the movement of dislocations while further increasing the material strength and hardness. Increasing the number of ECAP passes resulted in increased hardness of the samples. In all ECAPed samples with two passes, materials hardness increased by approximately 45% as compared to the as-received ones. All sets of samples, which are subjected to wear testing, show that ECAP decreases the material wear resistance as contradicted to Archard equation. Moreover, samples mass loss increased as the number of ECAP passes is increased. The reason is the presence of sub-grains and also grain refinement in the samples microstructure while the material ductility decreases during ECAP.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors

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