Impact of ECAP on wear performance of Al–Mn magnesium alloy

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Keywords: ECAP, AM90 alloy, grain refinement, microstructure, wear

Abstract

The current study examines the wear performance of deformed AM90 magnesium alloy using pin-on-disc wear test apparatus under varying load of 30 and 40 N with the sliding distance of 2500 m and 5000 m at constant sliding speed of 3 m s−1. The samples were processed through equal channel angular pressing were subjected to microstructural, wear studies and are characterized by optical and scanning electron microscopy. Vickers microhardness was carried out to evaluate the relation between the wear behavior of homogenized and ECAP processed samples. Increment in number of ECAP passes increased the grain refinement as observed in the microstructure with reduction in grain size from ∼70 μm (homogenized sample) to ∼4 μm (ECAP 4 pass sample). Increase in wear resistance and hardness were observed for processed samples and the worn faces are analyzed by scanning electron microscopy and energy-dispersive x-ray spectrometer. The worn surfaces revealed the occurrence of wear debris, delamination, ploughing in the direction of sliding and the predominant wear mechanism was considered to be the combination of oxidation and abrasive wear.

1. Introduction

Magnesium and magnesium alloys are considered as one of the lightest and attractive materials in the field of automotive, aerospace and medical industries for different applications. They are in need for the society due to the properties like low density, improved thermal and electrical conductivity, high specific stiffness, good machinability and vibration damping characteristics [1–4]. Some of the limitations that are included are poor tribological and corrosion properties which hindered the usage of magnesium and its alloys for different engineering applications [5]. Even though, the magnesium and its alloys are not used in fabricating the components like gears, bearings or seals but they may be subjected to rubbing when they come in contact with other components [6]. Generally the life of the moving components depends on the wear properties of those materials. So, the improvement in wear behavior of magnesium alloys may increase the applications of these materials in automotive and aerospace industries. Being one of the lightest material, it also reduces the fuel consumption [7, 8]. Many researchers have reported different methods which are available under severe plastic deformation (SPD) for improving the wear resistance properties of the materials. Few typical SPD techniques includes accumulative roll-bonding [9–11], multiaxial forging [12, 13], high pressure torsion [14, 15], repetitive corrugation and straightening [16, 17] and equal channel angular pressing [18–20].

The grain refinement by equal channel angular pressing (ECAP) is reported to be the most promising method for attaining the grain refinement for bulk materials, where high strains are induced during processing [18]. Different routes are used in ECAP process for inducing high strains into the material to achieve grain refinement as shown in figure 1. In route A, the sample is pressed frequently without any rotation, route B_A the sample is rotated by 90° in alternative direction among successive passes, route B_C the sample is rotated in the same direction by 90° between each pass and in route C, the sample is rotated by 180° in the same direction between each pass [18]. There are few studies reported by researchers regarding the wear behavior of different material processed by ECAP. Li et al [21] reported that the wear resistance is increased for Cu–Zr alloy with increment in more number of ECAP passes. Wang et al [22] showed that the wear resistance is reduced for an
ECAP processed sample of Al-1050 because of its loss in strain hardening ability. Cheng et al. [23] processed Ti-Ni alloy by ECAP and the processed samples exhibited improved wear resistance with their grain refined microstructure. La et al. [24] stated that wear resistance is decreased for ECAP processed pure titanium due to the increment in hardness and strength of the processed material. El Aal et al. [25] indicated a substantial reduction in wear mass loss of processed Al–Cu alloy with increased number of ECAP passes. Kucukomeroglu [26] conveyed that, although the strength of ECAP processed Al-12Si alloy is increased, there was a decrement in the wear resistance. Gao and Cheng [27] reported that the refined microstructure enhanced the hardness and decreased the wear rate of ECAP processed Cu–10Al–4Fe alloy.

As per above reported works, it was observed that, the obtained results for the ECAP processed samples on wear behavior are not constant. Further research is needed to identify the wear behavior of magnesium and magnesium alloys processed by ECAP technique. Limited work has been reported by the researchers to find the wear performance of magnesium and magnesium alloys treated by ECAP process and fewer work is reported on aluminium-manganese [AM] magnesium alloy. In particular AM series alloys are known for their properties like better damping, impact absorption, elongation and toughness. These properties have increased their application in making automobile parts like seat frames, brackets, clutch and brake pedals [28]. Improvement in wear properties of AM series alloys help in increasing the usage of these materials in the area of automobile and aerospace applications.

In the current work, AM90 alloy samples were subjected to ECAP process and the influence of grain refinement on wear properties were investigated. Unprocessed (homogenized) and ECAP processed AM90 samples were tested to know the wear behavior using pin-on-disc wear test equipment. Worn surfaces were studied by scanning electron microscope (SEM) with energy dispersive x-ray spectrometer (EDS) to recognize the wear mechanism.

### 2. Materials and experimentation

Nominal configuration of cast AM90 magnesium alloy is shown in table 1. Cast magnesium alloy (AM series) was procured from venuka engineering private ltd, telangana, India. Material was available in the form of rectangle ingots of $240 \text{ mm} \times 100 \text{ mm} \times 24 \text{ mm}$. Procured materials were cut into $20 \text{ mm} \times 20 \text{ mm}$ square sectioned rods of length $100 \text{ mm}$. Further the samples were turned to $15.8 \text{ mm}$ in diameter and $95 \text{ mm}$ in length. The machined samples were homogenized at a temperature of $400^\circ \text{C}$ for 24 h in a muffle furnace to attain uniform distribution of constituents inside the magnesium matrix.
ECAP die is kept on the base plate of universal testing machine (UTM-40 Ton) and the plunger is positioned to the centre of ECAP die hole (16 mm diameter) with reference to the location where the sample is placed. Split type ECAP die (Hot die steel) designed to a channel angle ($\Phi$) of 110° and an outer arc of curvature ($\Psi$) of 20° as presented in figure 2. ECAP die was allowed to heat by using 4 heating coils that are located in holes provided within the die vertically. Temperature of the die was continuously monitored throughout the ECAP process by the thermocouple fixed at the intersection point of channel angle. Firstly, experimental optimization was carried out to optimize the exact temperature at which the ECAP process can be conducted. Then, the ECAP die was heated to the optimized temperature and is controlled by the temperature regulator available with heating setup. Sample is placed in the die channel and need to make sure that the temperature of the specimen has also reached the required temperature. Initially, ECAP process was conducted for number of samples at different temperatures and optimized the exact temperature as shown in figure 3.

A lubricant of molybdenum-disulphide is added to the die channels to minimize frictional effects between samples and the die channels. Once the required temperature is attained, the sample is pressed by applying a load at the rate of 1 mm s$^{-1}$ using plunger attached to the UTM for deformation of the sample. The channels are intersected to induce the total strain on the sample to get ultra-fine grain (UFG) structure.

Samples of dimension, 15.8 mm in diameter and length of 95 mm (figure 3(a)) were processed at different temperatures varying from 225 °C to 275 °C and the processed samples are indicated in figures 3(b)–(d). Sample processed at 225 °C temperature showed large surface cracks. At 250 °C, surface cracks were reduced and at 275 °C temperature, samples were produced with no surface cracks with a soaking time of 15 min and pressing speed of 1 mm s$^{-1}$ as shown in figure 3(d). Based on the above experimental optimization, 275 °C temperature was considered for processing the samples through ECAP process as it is difficult to deform magnesium at lower temperature because of hexagonal closed packed (HCP) structure with restricted number of slip systems.

The process is repeated by using route B$_C$, where the samples are rotated by angle 90° between two continuous passes. Route B$_C$ is considered to be the effective route for achieving homogeneous microstructures of equiaxed grains in the processed sample with rapid evolution of high-angle grain boundaries [18]. Sample is pressed by plunger attached to the UTM producing corresponding strain of $\sim$0.8 [29] per ECAP pass and the process was repeated till four passes.

Microstructural evolution of homogenized and processed samples of AM90 cast magnesium alloys were investigated. Samples were sliced at a distance of 15 mm away from the tail region of the sample in order to avoid inhomogeneities which may occur because of distortion occurred at tail end region. The samples were polished by using polishing machine with abrasive paper starting from lower grit size to higher grit size and further polished by cloth using diamond paste for obtaining mirror finish surface and was finally cleaned using acetone. For revealing the microstructure, the etchant is prepared with 4.2 g picric acid, 10 ml acetic acid, 70 ml ethanol and 10 ml distilled water [30]. The polished samples were dipped in the etchant for approximately 5 s, so that the sample turns light brown in colour and washed with distilled water and dried thoroughly. Microstructures were observed by using optical microscope (OM) by image analyser (BIOVIS Software) and the average grain size was measured by linear intercept method. Vickers microhardness of the processed samples are measured by using omni tech microhardness equipment. The test was conducted at different locations on the surface of microstructure at equal intervals with an applied load of 25 g for a period of 15 s. Indentations at different locations of mid sectioned region of the sample were recorded to check the uniformity throughout the sample.

![Figure 2. ECAP die of channel angle ($\Phi$), 110° and outer arc of curvature ($\Psi$), 20°.](image-url)
Wear test samples are machined by selecting the mid region of homogenized and ECAP processed samples. Sample dimension were selected based on ASTM G99-05 with a dimension of 28 mm in length and 6 mm in diameter. The tests were conducted at room temperature condition by using pin-on-disc tribometer (DUCOM-TR-20LE) wear test equipment. The test equipment is fitted with a hard and polished disc made from EN31 steel with the hardness of 65 HRC. Before the sample is subjected to wear test, the surface is grinded using 1500 grit abrasive paper and cleaned with ultrasonicator followed by drying. During each test, the disc surface is cleaned with abrasive paper and the acetone to remove worn out particles on the disc surface.

Samples were tested at two different loads of 30 N and 40 N with two different sliding distances of 2500 m and 5000 m with constant sliding speed of 3 m s⁻¹ and track of 110 mm diameter. Initial weight of the sample is measured before the test and final weight is measured after the test. The difference in weight loss showed the wear resistance of that particular sample. Surfaces of tested samples (homogenized and ECAP processed) were analysed by SEM with EDS attached to observe the wear mechanism.

3. Results and discussion

3.1. Microstructural behavior

Microstructures of unprocessed and ECAP processed samples were analyzed with optical microscopy as shown in figure 4. The grain size of as-cast sample was found to be 60 ± 10 μm. The as-cast samples were exposed to homogenization at a temperature of 400 °C for 24 h to ensure uniform distribution of secondary particles inside the magnesium matrix. The homogenized sample possessed a grain size of 70 ± 10 μm and were subjected to ECAP process up to 4 passes. Initially, the sample is subjected to wear test, the surface is grinded using 1500 grit abrasive paper and cleaned with ultrasonicator followed by drying. During each test, the disc surface is cleaned with abrasive paper and the acetone to remove worn out particles on the disc surface.

Samples were tested at two different loads of 30 N and 40 N with two different sliding distances of 2500 m and 5000 m with constant sliding speed of 3 m s⁻¹ and track of 110 mm diameter. Initial weight of the sample is measured before the test and final weight is measured after the test. The difference in weight loss showed the wear resistance of that particular sample. Surfaces of tested samples (homogenized and ECAP processed) were analysed by SEM with EDS attached to observe the wear mechanism.
mechanisms and dynamic recovery [33]. High orientation grains possessing lower schmid factor finds difficulty in deforming but the low orientation grains consists of larger schmid factors undergo effortless deformation [34]. Lin et al [35] conveyed that, the higher value (0.27) of schmid factor for processed magnesium alloy condition and zero value schmid factor for unprocessed condition has caused non-uniform microstructure. Homogeneity in microstructure has increased with increment in number of ECAP passes.

The grain size achieved for ECAP processed 3 pass sample and 4 pass sample was 10 ± 3 μm and 4 ± 2 μm, respectively. Uniform and equiaxed microstructure could be because of dynamic recrystallization (DRX) which occurred during ECAP process and static recrystallization prior to the beginning of ECAP process during preheating of the die [36]. Extensive plastic deformation that took place in the samples with increased ECAP passes lead to grain refinement. Grain sizes of homogenized and ECAP processed 1 pass sample to 4 pass samples are plotted in figure 5.

3.2. Hardness

Microhardness test was carried out on homogenized and ECAP processed samples by considering a load of 25 g with load application time of 15 s. Hardness was checked at different points on the surface of the sample to recognize the uniformity of hardness distributed above the surface of the sample. Microhardness values were obtained for homogenized and ECAP processed samples and are highlighted in figure 6. The hardness of homogenized sample was around 85 Hv and with increase in number of ECAP passes, the hardness values increased to 90 Hv for ECAP 1 pass sample and 102 Hv for ECAP 2 pass sample. An enhancement in microhardness may be due to increased dislocation density and development of sub microcrystalline structures that has occurred during the severe plastic deformation.

With refined microstructure, hardness has increased due to the restriction of dislocation movement imposed by high dislocation density, misorientation of grains and low angle grain boundaries (LAGBs) [37]. As the ECAP passes increases further, hardness value decreased for ECAP 3 pass (95 Hv) sample because of development of newer grains that hindered the strain hardening effect. For ECAP 4 pass sample, microhardness value has slightly reduced to 91 Hv, which may be due to variation in texture. Decrease in hardness after 3 pass could be also due to decrease in dislocation density due to dynamic recrystallization and grain growth [38]. Increment in the homogeneity of microhardness was observed with increase in number of ECAP passes. From
the figure 6, it can be interpreted that, hardness of processed ECAP 1 pass sample and 2 pass samples are less homogeneous in comparison to ECAP 3 pass sample and 4 pass sample. Uniformity in hardness over the surface of ECAP 3 pass sample and 4 pass sample is due to homogeneous microstructure obtained because of increased number of ECAP passes [39].

3.3. Wear behavior
Two loads and two sliding distances were selected as varying parameters to conduct wear test using DUCOM pin-on-disc wear testing machine. Load of 30 and 40 N and sliding distance of 2500 m and 5000 m was selected. For each condition, 3 trials were conducted and the average coefficient of friction (COF) values were obtained for varying conditions and are plotted in figure 7.

It is observed from graph that as the ECAP passes increases, COF starts reducing compared to homogenized samples indicating wear resistance behavior of processed samples. Fluctuation in the COF values for 40 N load is less compared to 30 N load as shown in figure 7, because of increased surface contact between the sample and the rotating disc [40]. Reduced fluctuation shows increased wear resistance property of the ECAP processed samples.
Wear mass loss follows the trend similar to COF. Samples are subjected to varying loads and sliding distances for evaluating the wear behavior. ECAP samples showed reduced wear mass loss in comparison to homogenized condition. This may be due to enhanced microhardness distribution which avoids loss of material due to sliding motion of the sample with the disc preventing the development of delamination and wear debris. Wear mass loss for different conditions are shown in Figure 8. ECAP 2 pass sample showed higher wear resistance because of higher hardness value due to grain refinement. Grain refinement due to ECAP process has increased the hardness of the sample and accordingly increased the wear resistance property of the sample which is related by Archard equation [41].

Delamination was observed on the worn surface with wear debris and small grooves are formed due to ploughing action caused by debris present on the worn surface as identified in figures 9–12. It was noticed that, the wear mass loss of the sample has increased with increase in load and sliding distance which had led to the formation of delamination and wear debris on the sample surface. With increase in number of ECAP passes, the wear resistance increased reducing the formation of delamination and wear debris. The increased hardness in ECAP processed samples restricts the formation of cracks. Therefore the formation of delamination and wear debris is reduced during sliding thus by increasing the wear resistance property. Smooth surfaces appeared with increase in number of ECAP passes compared to initial conditions. With these surface morphologies, the mechanism of wear is noted to be an abrasive wear.

EDS analysis revealed the presence of oxygen element along with magnesium. Since magnesium is more prone to oxidation when comes in contact with oxygen it can be considered as one of the reason for wear mass loss.
loss. EDS was carried out on worn surfaces of homogenized and ECAP 2 pass sample by considering best among ECAP processed samples under 30 and 40 N loads. Magnesium and oxygen elements are identified on the wear surface in the areas of delamination, ploughing and wear debris as shown in figures 13 and 14. Based on the above discussion we can also consider the possibility of oxidation wear.

**Figure 9.** SEM micrographs of worn surfaces (a) Homogenized sample and ECAP (b) 1 Pass, (c) 2 Pass, and (d) 4 Pass samples with 30 N load and sliding distance 2500 m.

**Figure 10.** SEM micrographs of worn surfaces (a) Homogenized sample and ECAP (b) 1 Pass, (c) 2 Pass, and (d) 4 Pass samples with 40 N load and sliding distance 2500 m.
4. Conclusions

Equal channel angular pressing of AM90 magnesium alloy was conducted up to 4 passes. The processed samples were subjected to microstructural studies and are tested for tribological behavior using pin-on-disc apparatus and the obtained outcomes are summarized:

![Figure 11. SEM micrographs of worn surfaces (a) Homogenized sample and ECAP (b) 1 Pass, (c) 2 Pass, and (d) 4 Pass samples with 30 N load and sliding distance 5000 m.](image)

![Figure 12. SEM micrographs of worn surfaces (a) Homogenized sample and ECAP (b) 1 Pass, (c) 2 Pass, and (d) 4 Pass samples with 40 N load and sliding distance 5000 m.](image)
Grain size of homogenized AM90 magnesium alloy was reduced from $70 \pm 10 \mu m$ to $4 \pm 2 \mu m$ after ECAP 4 passes and lead to the formation of homogeneous grain structure due to dynamic recrystallization occurred during ECAP process. COF found decremented for ECAP processed samples under different load and sliding distances in comparison to homogenized sample that showed reduced wear rate. Improvement in wear behavior was witnessed for ECAP processed samples due to increased hardness and uniformity over the cross section of ECAP processed samples. Occurrence of delamination and wear debris witnessed by SEM showed abrasive wear mechanism and the existence of oxygen elements with magnesium represented oxidation wear mechanism.

Figure 13. EDS of worn surfaces under 30 N load (a) Homogenized sample and (b) ECAP 2 Pass sample.

Figure 14. EDS of worn surfaces under 40 N load (a) Homogenized sample and (b) ECAP 2 Pass sample.
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