Evolution of microstructure, texture, and mechanical properties in a multidirectionally forged ZK60 Mg alloy

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ABSTRACT

An extruded ZK60 magnesium alloy with the nominal composition of Mg–6Zn–0.5Zr was processed by multi directional forging (MDF) for 2, 4, 6 and 8 passes at 473 K. Microstructural studies showed that the uniform microstructure of the as-extruded material transforms to a duplex structure consisting of fine recrystallized grains surrounding some patches of unrecrystallized grains after 2 and 4 passes of MDF. Further MDF passes, however, resulted in a homogeneous equiaxed structure in such a way that the as-extruded grain size of 12.7 μm was refined to 1.9 μm after 8 passes. Evaluation of crystallographic texture revealed that deformation by MDF changed the conventional fiber texture of the extruded condition to a new texture, in which the basal planes tend to align almost 45° to the transverse direction. Mechanical properties of the extruded and MDFed samples were studied by shear punch testing (SPT) at room temperature. The results indicated that the 182.6 MPa shear yield stress (SYS) and 184.9 MPa ultimate shear strength (USS) of the extruded condition were, respectively, reduced to 170.0 and 172.3 MPa after 2 passes of MDF. This drop in strength is attributed to the presence of coarse patches of unrecrystallized grains. Further pressing of the alloy was accompanied by a decrease in the volume fraction of coarse grains and an increase in the number density of fine grains. Textural softening that occurred at higher strain levels of the final MDF passes, however, offset the strengthening effect of these fine grains.

1. Introduction

Magnesium alloys have recently been used in various applications, because of their exceptional combination of excellent machinability, high specific strength, low density and high potential for recycling. Their use, however, is restricted due to the unstable microstructure at high temperatures and low ductility caused by their hexagonal close-packed (HCP) crystal structure [1,2]. The most frequently used magnesium alloys are based on Mg–Al system. Nevertheless, they possess inadequate mechanical properties at high temperatures, due to the thermal instability of the Mg17Al12 phase [3–5]. In addition to aluminum, zinc (Zn) is also considered as an important alloying element in Mg alloys. It is more effective than aluminum in solid solution strengthening at high concentrations. However, Mg–Zn binary alloys suffer from coarse grains and tiny porosities. Since super heating of the melt does not significantly affect the grain size of these alloys, addition of zirconium has been suggested as a proper way to decrease the grain size [6].

Several research works have focused on the influence of grain refinement processing routes on the achievement of superior mechanical properties. In this regard, severe plastic deformation (SPD) methods have been applied to various Mg alloys to obtain fine-, ultra-fine-, and nano-grained microstructures [7–12]. Multi directional forging (MDF) is one of the SPD methods that can induce high strains into bulk materials [13]. The studies of microstructural evolution during MDF process in Mg alloys have shown that grain size distribution at the primary stages of MDF process is inhomogeneous, the microstructure being composed of both fine (1–2 μm) and coarse (> 10 μm) grains [14]. With increasing MDF passes, however, grain size distribution becomes more homogeneous, containing equiaxed fine grains. Various mechanisms have been put forward for explanation of grain size reduction after MDF process. According to the results obtained from microstructural changes during MDF, it has been suggested that dynamic recrystallization (DRX) is the dominant grain refinement mechanism [15].

It is well accepted that severe plastic deformation processes refine the microstructure of Mg alloys, if they are carried out in an appropriate temperature range. The observed grain refinement has been considered as the principal cause for enhanced yield stress after MDF processing of different Mg alloys [15]. Despite the progressive grain refinement at higher strain levels that is obtained after further MDF passes, there has been some softening at finer grain sizes, which is not in agreement with the classic Hall-Petch relationship [16]. The restricted slip systems of
the hcp structure of Mg alloys at ambient temperature results in a strong dependency of mechanical properties on the crystallographic texture. Accordingly, the variation in mechanical properties during MDF processing of Mg alloys, especially at early stages, can be explained by strong crystallographic texture that would be diminished at high strains with change in direction of the applied stress [15].

Although there is no report on the softening of extruded Mg alloys after MDF process, there are a few reports on the fluctuations of yield stress after different passes of this process [16,17]. In these reports, yield stress in all MDFed conditions has been higher than that of the as-received material. Nevertheless, the variation of yield stress with MDF passes has not shown any specific trend. MDF processing of an as-cast AZ31 alloy has shown a significant increase in the strength up to 9 passes [16]. In contrast, yield stress has decreased after accomplishing 27 and 36 MDF passes, due to a drop in twin density and an increase in the DRX degree in these conditions. In another research, the yield stress of the WE magnesium alloy increased from 245 MPa in the extruded condition to 425 MPa after one pass of MDF. A drop in yield stress value was observed from 425 MPa to 339 MPa after 3 passes and this value increased again to 378 MPa after 5 MDF passes. This behavior was attributed to the occurrence of texture softening after 3 passes of MDF [17].

This work is aimed at examining the evolution of microstructure, texture, and mechanical properties in a multi directionally forged Mg–6Zn–0.5Zr (ZK60) alloy. The variation in the shear strength of the material during different of deformation stages is discussed based on the grain refinement effects as well as textural changes.

2. Experimental procedure

2.1. Material and processing

High purity Mg (99.9 wt%), Zn (99.9 wt%) and an Mg–30 wt% Zr master alloy were used to prepare the ZK60 Mg alloy having the actual composition of Mg–5.8Zn–0.5Zr. Melting process was accomplished in a graphite crucible in an electrical furnace at 1053 K, using a covering flux to keep molten magnesium from oxidation. The melt was then cast into a mild steel cylindrical mold with 44 mm diameter and 120 mm height preheated to 473 K, using a tilt-casting system to ensure a laminar flow of the melt. As recommended in the literature [18], the cast billets were homogenized at 703 K for 16 h to eliminate the possible compositional inhomogeneities formed during solidification. The homogenized billets were finally extruded to 13 mm × 13 mm bars at 573 K. The extruded bars were cut into 13 mm × 13 mm × 20 mm billets before being subjected to MDF. The samples were multi-directionally forged at 473 K at a pressing velocity of 0.5 mm/min, provided by a universal tensile testing machine. The die assembly was heated to the desired temperature by heating elements incorporated close to the die cavity. A poly tetra fluoro ethylene (PTFE) film was wrapped around the sample to minimize the friction between the sample and the die. A schematic representation of MDF process is shown in Fig. 1, where it can be observed that the initial pressing direction is parallel to the extrusion axis, and between the successive MDF passes, the loading direction is changed by 90°. Based on the geometry of the billets, an equivalent strain of 0.5 can be obtained in each pass, resulting in a maximum cumulative strain of 4.0, after eight passes.

Optical microscopy and field emission scanning electron microscopy (FESEM) were employed to investigate the microstructural evolution of the material during MDF. The images acquired from optical microscopy were analyzed by the Clemex Vision professional image analysis software to determine the grain size and distribution of the MDFed material. After polishing with 0.05 μm Al₂O₃, samples were etched using a solution with chemical composition of 0.4 g picric acid, 2.5 ml acetic acid, 2 ml distilled H₂O and 4 ml ethanol. Microanalysis was carried out by X-ray energy dispersive spectroscopy (EDS) system of the FESEM and X-ray Diffraction (XRD). The intensity distribution of (0002) basal plane was determined by the Schultz reflection method of the plane that is always parallel to the final loading direction of the MDFed samples.

2.2. Mechanical property measurements

Shear deformation behavior of the material was evaluated by the miniaturized shear punch testing (SPT) method. Specimens with the nominal thickness of 1 mm were excised using a wire-cut machine along the loading direction of the MDFed samples, and then ground to the final thickness of 0.7 mm. A shear punch fixture having a 3.075 mm diameter flat-ended cylindrical punch and 3.125 mm diameter receiving-hole was employed in this study. All of the tests were carried out at a constant cross-head speed of 0.25 mm/min at room temperature. The applied load P was recorded as a function of punch displacement by a computerized data acquisition system. The following equation can be used to evaluate the shear stress of the tested material.

\[ \tau = \frac{P}{\pi dt} \]  

where \( P \) is the punch load, \( d \) is the average of the punch and die diameters and \( \tau \) is the specimen thickness. For credibility of the results, the tests were repeated for three times.

3. Results

3.1. Microstructural observations

The optical microstructure of the tested material in the as-extruded and MDFed conditions is shown in Fig. 2. The microstructure of the as-extruded material comprises fine uniform equiaxed grains, indicative of a fully recrystallized condition with an average grain size of about 12.7 μm (Fig. 2a). In contrast to the extruded condition, the microstructure of the material after 2 and 4 passes of MDF exhibit a bimodal grain structure comprising a very fine-grained recrystallized structure together with patches of unrecrystallized grains (Fig. 2b and c). The volume fraction of these patches, however, decreases with further straining after 6 passes of MDF, as shown in Fig. 2d. It is also evident that after 8 MDF passes, the microstructure is typical of a fully...
recrystallized material, having a uniform grain structure with no sign of unrecrystallized patches of grains (Fig. 2d).

For a more clear observation of the microstructural evolution during MDF, the grain structures of the tested conditions were examined at higher magnification in the FESEM images depicted in Fig. 3. The micrograph of the as-extruded material, shown in Fig. 3a, is indicative of an equiaxed grain structure containing a rather uniform dispersion of second phase particles, having dimensions in the range 0.5–1.5 μm. The micrographs of the material after different passes of MDF indicate that the size of these particles is reduced to less than 1 μm, due to the fragmentation caused by the severe plastic deformation process. As can be seen in Fig. 3b–d, the microstructures of the MDFed material after 2, 4, and 6 passes of MDF comprises fine-grained equiaxed crystallized grains with average sizes in the range 2.2–2.6 μm surrounding patches of unrecrystallized grains. This kind of grain structure is typical of hot deformed ZK60 alloy [19–21].

Concerning the type of intermetallic particles revealed in the microstructure, EDS results of the two fine (A) and coarse (B) particles, demonstrated in Fig. 3a, are given in Fig. 4a and b, respectively. They are both comprised of Mg and Zn. The chemical composition of the finer particle is Mg\text{53.3}Zn\text{46.7} and that of the coarser one is Mg\text{41.5}Zn\text{58.5}, which closely correspond to the MgZn and MgZn\text{2} intermetallic compounds, respectively. The observed overestimation of the Mg content can be due to emission of X-ray from material under the particles. As a support for EDX results, XRD analysis was applied to the extruded material to detect the existing phases. The results exhibited in Fig. 5 indicate that Mg, Mg\text{Zn}, and Mg\text{Zn\text{2}} are present in the ZK60 alloy. The low intensity of the intermetallic peaks is indicative of their low volume fractions in the Mg matrix. The detected particles are expected to contribute to the strength of the alloy at all testing conditions.

To inspect the effect of MDF process on the development of the grain structure, the average grain sizes of the material after extrusion and different passes of MDF are summarized in Table 1. The sizes of the unrecrystallized patches in the MDFed conditions were not considered in the calculation of the given data, for a better comparison with that of the extruded material having a uniform grain structure. It is evident that there is a drastic drop in the grain size of the extruded material after two passes of MDF, where it decreases from 12.7 to 2.6 μm. More passes of MDF, however, cause small reduction in the size of recrystallized grains, in such a way that the grain size of 2.6 μm in the 2-pass MDF decreases to 1.9 μm after 8 MDF passes.

Grain size distribution is an important parameter affecting the mechanical properties of materials processed by the SPD methods. Fig. 6a–d demonstrates the grain size distributions in the recrystallized zones of the microstructures after different MDF passes. It can be inferred from Fig. 6 that an almost normal distribution has been achieved for all of the MDFed conditions. The histograms of all MDFed conditions indicate that the MDF process has been able to develop a well-refined uniform grain structure in the recrystallized zones of the microstructure with reasonably low standard deviations in the range 0.51–0.97 μm.

3.2. Textural evolution

The (0002) pole figure of the extruded ZK60 alloy is illustrated in Fig. 7. It is well accepted that the major deformation mechanism in magnesium, especially at primary stages, is basal planes slip. Accordingly, the basal planes intensity in the measured crystallographic textures is an appropriate way for the identification of operant deformation mechanisms.

Fig. 8 shows the textural evolution of the material after different passes of MDF. This can be manifested by the effect of MDF process on the location of maximum texture intensity on (0002) basal plane pole figures. The pole figures of the MDFed materials indicate that most basal planes start rotating in such a way that they are placed at angles that are lower than 90° to the direction normal to the last forging.
Comparison of Figs. 7 and 8a shows that there is a substantial decrease in texture intensity after 2 passes of MDF process. However, 4 passes of MDF leads to an increase in texture intensity (Fig. 8b), followed by intensity drop with further passes of MDF (Fig. 8c and d).

3.3. Mechanical properties

Shear punch testing technique was employed to study the shear yield stress (SYS) and ultimate shear strength (USS) of the material under different processing conditions. In this test, the variation in shear stress is usually plotted as a function of normalized displacement of the punch [22–25]. Fig. 9a illustrates typical room temperature SPT curves of the ZK60 alloy after 4 MDF passes. As can be seen, the results have a high degree of reproducibility in terms of strength and displacement. It is worth noting that 1% proof stress is usually taken as SYS, and the stress at the maximum stress is defined as USS. SPT has frequently been used to study the mechanical properties of magnesium alloys deformed by severe plastic deformation [8–10,26]. It has been shown that there is usually a linear relationship between the SPT stress values and tensile test data [27].

Fig. 9b depicts SPT curves for the extruded condition and MDFed ZK60 samples. The extruded specimens were taken perpendicular to the extrusion axis, and MDFed samples were parallel to the LFD. All SPTs were carried out at room temperature at the strain rate of $8.3 \times 10^{-2}$ s$^{-1}$. As can be seen in Fig. 9b, the extruded alloy possesses the highest strength levels among all tested conditions. In other words, the overall levels of the curves after MDF are lower than the extruded condition. The variations in SYS and USS values with number of passes are summarized in Fig. 9c. The 182.6 MPa SYS of the extruded alloy decreases to 170.0 MPa after 2 MDF passes. The variation in strength during further passes, however, is trivial with that of the 6th pass (173.1 MPa) being slightly higher than that of the 4 passes (171.6 MPa) and 8 passes (170.4 MPa). The same trend is observed for the variation of USS with pass number, shown in the same figure.

4. Discussion

4.1. Microstructural evolution

Microstructural observations (Figs. 2 and 3) indicate that during initial passes of the MDF process, a bimodal microstructure consisting of fine dynamically recrystallized grains surrounding coarse patches of unrecrystallized grains are formed as a result of partial dynamic recrystallization. Similar bimodal grain structures have also been reported in Gd-containing Mg alloys after severe plastic deformation [28,29]. The presence of alloying elements, which can retard the nucleation of recrystallization, has been introduced as the prime cause for the formation of colonies of unrecrystallized grains. Therefore, it seems that the amount of strain imposed by 2, 4, and 6 passes of MDF and the deformation temperature of 473 K in the present investigation were not sufficient to attain a fully recrystallized structure in the ZK60 alloy. After the 8th pass of MDF, however, the higher accumulated strain has resulted in a uniform structure with no unrecrystallized grains (Fig. 3e). The driving force for recovery and recrystallization during MDF process is provided by the strain accumulation in an appropriate range of temperature. Accordingly, dynamic recrystallization occurs during MDF, in which numerous fine equiaxed grains are formed in almost all of the MDF passes. It seems, however, that this dynamic recrystallization has a discontinuous nature, because the number density of fine grains gradually increases with increasing MDF pass numbers. This
results in a homogenous and fine-grained structure after the completion of dynamic recrystallization [30].

Similar trends in the grain refinement of other Mg alloys by MDF have been reported in the literature [14,16]. A final grain size of 3.3 μm has been attained for the ZK60 alloy after extrusion with the high extrusion ratio of 100 at 583 K [31]. In addition, finer grain sizes of 0.7, 2.9, 1.4 and 2.9 μm have been achieved for the same material after different severe plastic deformation methods of friction stir processing [32], high pressure torsion (HPT) at room temperature [10], 8 passes of ECAP at 433 K [33], and two passes of ECAP at 473 K [34], respectively.

In addition to the size of the microstructural constituents, the uniformity of grain size distribution in the materials experienced severe plastic deformation is essential for attaining homogeneous mechanical properties [26]. It is evident that the material processed by 8 passes of MDF has the most uniform distribution with the lowest standard deviation of 0.51 (Fig. 6). This is due to complete dynamic recrystallization throughout the microstructure, which can be achieved only after 8 passes of MDF.

4.2. Textural evolution

According to Fig. 7, an expected fiber texture in basal planes can be seen after extrusion. This has been caused by the fact that (0002) basal planes are mostly oriented parallel to the extrusion direction. In such a situation, basal planes tend to line up the ⟨1010⟩ direction with the extrusion direction [35]. By applying MDF process, most basal planes are rotated toward the angles lower than 90° respect to the TD (Fig. 8). This behavior has been also observed in the MDF processing of an Mg-Gd-Y-Zr alloy [14]. During subsequent steps of MDF, the original fiber texture is gradually replaced by a newly formed texture.
component with basal planes preferentially tilted toward 45° to the TD (Fig. 8d). In such a condition, most of the grains take an orientation that is favorable for dislocation glide on the {0002} slip planes. As will be discussed in the next section, when the original fiber texture with higher shear strength is replaced by the new dominant texture, developed after different passes of MDF, the strength is reduced.

Concerning the intensity of the texture components, it is deducible that the fiber texture observed in the extrusion condition is weakened after two passes of MDF, as shown in Fig. 8a. The observed drop in the texture strength from 5.3 in as-extruded condition to 2.7 after 2 MDF passes can be ascribed to the formation of partially recrystallized grains. The dynamically recrystallized grains produced by hot deformation have a crucial role in weakening of the overall texture by opposing the strong deformation texture of the unrecrystallized grains [36,37]. As can be seen in Fig. 8b, the basal pole intensity increases to 7.0 after 4 passes of MDF. In this condition, only few new grains have formed in the microstructure, while a higher level of strain has been accumulated, in comparison with the material after 2 MDF passes. These two concurrent events have resulted in the observed increase in the texture intensity of the basal planes. As the deformation precedes, the volume fraction of the DRXed grains increases, resulting in lower intensities of 4.0 and 3.7 after 6 and 8 passes, respectively (Fig. 8c and d). Our findings are in agreement with those in the literature, reporting that in multi-directional forging of magnesium alloys the basal pole intensity did not follow a specific trend with increasing MDF passes [38–40].

4.3. Mechanical properties

According to the classic Hall-Petch equation, one would expect higher strength values at smaller grain sizes, achieved at higher MDF passes. This is not the case in the present work, where there is a significant strength drop after the first 2 passes of MDF (Fig. 9). Although it is well established that severe plastic deformation of Mg alloys results in a significant grain refinement, the strength may not necessarily increase after deformation. This has also been observed in the ECAP of ZK60 [41] and AZ31 [27] alloys. The main cause of such a behavior has been related to the textural softening that occurs during the SPD processing [42,43]. It is well-known that slip on the basal planes is the dominant deformation mechanism of magnesium alloys at ambient temperature. Based on our textural results, exhibited in Fig. 8, it can be deduced that the MDF process has the ability to modify the fiber texture of the extruded condition. Polar images of the examined samples confirmed that the MDF process rotated the basal planes toward the TD. Accordingly, after different passes of MDF, the maximum texture intensity deviates from its initial position, getting closer to 45° respect to the TD. This displacement results in an increase in the Schmid factor that increases the resolved shear stress (RSS) on the basal planes and facilitates an easier flow of material.

In addition to the textural softening effects, the influence of unrecrystallized patches on the final strength should be taken into account. The presence of the unrecrystallized patches could have contributed to the strength drop observed in the primary stages of MDF. In the final stages of the MDF process, where the microstructure is mainly composed of fine recrystallized grains, it seems that textural softening overcomes the grain refining effects. This can justify the lower strength of the material after 6 and 8 MDF passes, despite having finer grain sizes with respect to the extruded condition. The small variation in the strength of the material after 6 and 8 passes of MDF can be ascribed to the fact that the variation between their grain sizes and textures are

Fig. 8. {0002} Pole figures of the alloy MDFed for: (a) 2 passes, (b) 4 passes, (c) 6 passes and (d) 8 passes.
TD after different passes of MDF. This texture component resulted in textural softening, decreasing the strength of the alloy after MDF.

4. Investigation of the mechanical behavior by shear punch tests revealed that grain size effects were dominant in the initial MDF stages, while textural softening became more important in the final stages of the MDF process.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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References


