



Microstructure and Mechanical Properties of ZK60 Mg Alloy Processed by Cyclic Expansion-Extrusion (CEE) at Different Temperatures

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HIGHLIGHTS

- The cyclic expansion extrusion (CEE) process was applied to the ZK60 Mg alloy, and the influence of processing temperature was investigated.
- The CEE process at a processing temperature of 190 °C showed significant refining of α -Mg grains to a size of approximately 9 μm .
- The mechanical properties of ZK60 Mg alloy were greatly affected by the CEE processing temperature.
- The tensile test fracture morphologies of unprocessed and CEEed ZK60 Mg alloy revealed the combination of ductile and brittle fracture mechanisms.

ARTICLE INFO

Handling editor: Israa A. Aziz

Keywords:

ZK60 Mg alloy; Cyclic expansion extrusion; Grain refining; Mechanical properties; Tensile fracture surface.

ABSTRACT

In this work, the cyclic expansion extrusion (CEE) process was applied to ZK60 Mg alloy. The correlation between the evolved microstructure and mechanical properties was investigated. The CEE process was performed at a constant ram speed (15 mm/min) and at different processing temperatures (190, 270, and 350 °C). Optical and scanning electron microscopes, X-ray diffraction instruments, Vickers hardness tester, and tensile testing machine were utilized to examine the influence of CEE processing temperature on the characteristics of ZK60 Mg alloy. The XRD analysis showed that two phases were presented in the matrix of ZK60 Mg alloy, namely α -Mg and MgZn_2 , in small amounts. The CEE process reduced the size of α -Mg grains due to dynamic recrystallization, especially at the processing temperature of 190 °C. A slight coarsening of the α -Mg grains was observed with increasing processing temperature to 270 and 350 °C. The hardness value of ZK60 Mg alloy was enhanced by about 11 to 19% using the CEE process compared to the as-extruded sample. The processing temperature greatly affected the mechanical properties, where a significant improvement of about 24% yield strength, 9% ultimate tensile strength, and 38% elongation was observed using a processing temperature of 190 °C. The characterization of the tensile fracture surface of the tested samples indicated that the ductile-brittle fracture mode was responsible for the failure.

1. Introduction

The increasing demand for magnesium (Mg) alloys in various fields of applications, such as aerospace, transmission castings, weaponry, enclosures for electronic equipment, etc., has encouraged researchers to study their characteristics and ways to improve them. Increasing investments by various countries in various sectors around the world and the increasing need for applications of lightweight materials are some of the important factors expected to drive the global Mg alloys market [1]. Plastic deformation of Mg alloys includes changes in their geometry and microstructure. It depends primarily on the material's type and chemical composition and thus on its microstructure and texture. The HCP structure and low stacking fault energy limit the use of Mg alloys [2]. Mg alloys have three active independent slip systems that hinder room temperature deformability. In contrast, low stacking fault energy means that the dominant softening mechanism is dynamic recrystallization (DRX). Thus, the plastic deformation of Mg alloys is applied at high temperatures [3].

Plastic deformation caused by traditional deformation methods can greatly increment the strength of metals. However, this increment is associated with a decrease in ductility [4]. Furthermore, heat treatment theories [5] and alloying strategies [6] have been proposed to enhance the characteristics of Mg alloys. However, an exceptional combination of high strength and high ductility can be produced in metals and alloys by utilizing severe plastic deformation (SPD). Therefore, SPD processes have become an important focus of extensive research in the past decades due to their distinctive benefits, such as the improvement

of mechanical, corrosion, and physical properties resulting from grain refinement [7]. Mei-yan et al. [8] found that considerable grain refinement in AZ31 Mg alloy was recognized after three cycles of accumulative roll-bonding (ARB) due to the grain subdivision induced by severe accumulated strain. Lu et al. [9] reported that 16 passes of ECAP at 658 K resulted in a fine-grained GZ11 Mg alloy with significant properties that were further improved after rolling at 773 K. Ko and Hamad studied the mechanical properties of AZ31 Mg alloy using a differential speed rolling mill (DSR) [10]. They indicated that fine grains with obtainable homogeneous structures due to intersecting of shear bands during DSR were responsible for enhancing mechanical properties. Sułkowski et al. [11] observed that an increase in the number of extrusion-compression processes cycles greater than 4 resulted in sample failure due to the randomization of the orientation distribution that slowed down the work hardening. The results of Li et al. [12] on the processing of age-treated Mg-Zn-Y alloy with high-pressure torsion (HPT) revealed that the fragmentation of the coarse W ($\text{Mg}_3\text{Y}_2\text{Zn}_3$) into fine particles and reduction of nanosized MgZn and MgZn_2 precipitates were noticed with increasing torsional turns. They also indicated that a grain size of 53 nm was successfully obtained after 7 turns of HPT. Ahmadi et al. [13] disclosed a noticeable increment in the hardness and mechanical properties of AM60 Mg alloy processed by the new SPD process named cyclic extrusion compression angular pressing (CECAP) process due to the formation of fine-grained microstructure and well distribution of refined β -phase at the grain boundaries.

Generally, in all SPD processes, large plastic strains are applied to the samples resulting in the required grain refinement. However, among the SPD processes, Pardis et al. [14] proposed a new method named cyclic expansion-extrusion (CEE) as a modification of cyclic extrusion-compression (CEC). The CEE process has shown the capability to enhance the characteristics of metal and alloys such as hardness, ultimate tensile strength and ductility by refining the grain size through DRX as the strain accumulates [15]. Therefore, various materials such as 1050 Al alloy [14], AM60 Mg alloy [16], WE43 Mg alloy [17], 6063 Al alloy [18], and AZ91 Mg alloy [19] were processed using CEE, and their characteristics were sought. Unfortunately, little information regarding the effect of CEE processing temperature on the characteristics of samples can be obtained from the studies mentioned above. Thus, some light has been thrown on CEE applied at different temperatures, and the evaluation of the evolved microstructure, hardness, and mechanical properties of ZK60 alloy was investigated.

2. Experimental Work

In this study, a 15 mm diameter extruded rod of ZK60 Mg alloy with the chemical composition listed in Table 1 was used as experimental material. A rod (130 mm (length) $\times$ 15 mm (diameter)) was cut from the ZK60 Mg alloy for CEE processing. Figure 1 displays a photograph and diagram of the CEE die with a semi-die angle (α) of 30° . The CEE die was designed and made from hot-worked tool steel. The CEE die consisted of three cavities in which the upper cavity and lower cavity were 20 $\times$ 15 mm in size, and the middle cavity was 20 $\times$ 20 mm. The specimen's theoretical strain (ϵ) of 2.043 was determined as $\epsilon = 4 \ln D_1/D_0$, where D_1 and D_0 represent the final and initial diameters, respectively [14].

The ZK60 Mg alloy rod was subjected to a single pass at different processing temperatures of 190, 270, and 350 $^\circ\text{C}$ using a 20-ton hydraulic press at a constant ram speed of 15 mm/min. The CEE die and the sample loaded inside it were heated using a resistance-type furnace. To keep the required processing temperature at a relatively constant value, the furnace temperature was slightly increased owing to the opening of the top furnace door. The processing temperature was monitored using a temperature measuring device (thermocouple type K connected to a multimeter that displays the temperature in $^\circ\text{C}$). To reduce the friction between the die and the sample, graphite was utilized as a lubricant. Each sample processed using a CEE die was left to cool environmentally.

Microstructural examinations of the unprocessed and processed samples were performed with a LOM, BLP2000-type optical microscope after surface preparation using grinding, polishing, and etching processes, respectively. The grinding process was carried out using various SiC emery papers with different grit sizes. Polishing was achieved using a 5 μm alumina slurry. Etching was accomplished using an etching solution of 6 g picric acid, 10 ml acetic acid, 10 ml water, and 70 ml ethanol. The phases of the unetched ZK60 Mg alloy sample were identified using Shimadzu XRD 600 employed with Cu-K α radiation. The hardness of the surface of the sample was performed using a Vickers Hardness tester (Hensoldt Wetzlar, Germany) with an applied load of 200 g and a holding time of 10 s. At least five readings were reported at different positions, and the average value was identified. Tensile test samples were prepared according to ASTM E8M-89b, wherein an INSTRON tensile testing machine was utilized for testing to evaluate the mechanical properties. All tensile test experiments were performed at room temperature using a cross head speed of 1 mm/min. The tensile fracture surface of the tested samples was characterized using SEM type VEGA3LM to determine the failure mechanism.

Table 1: Chemical analysis of ZK60 Mg alloy

Elements	Zn	Zr	Fe	Ni	Cu	Sn	Pb	Ag	Mg
Composition (wt.%)	4.94	0.617	0.112	0.006	0.002	0.069	0.0593	0.001	Rem.

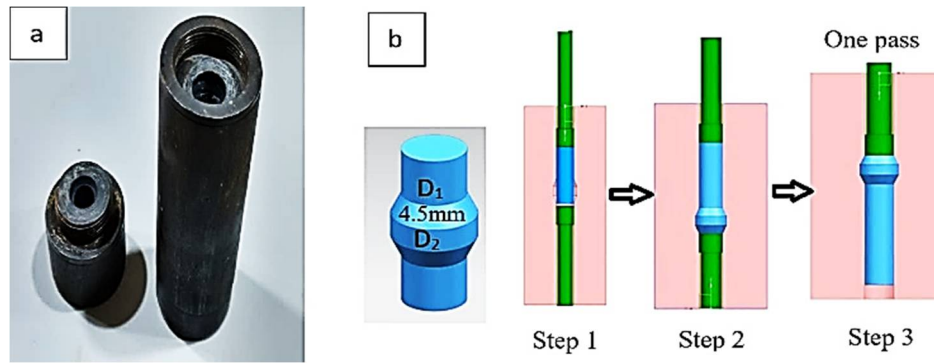


Figure 1: (a) CEE die used in the processing of Zk60 Mg alloy (b) the consecutive steps of CEE

3. Results and Discussion

3.1 XRD and Microstructure

The presence of phases in the as-extruded ZK60 Mg alloy was determined by XRD analysis, as shown in Figure 2. A prominent peak of α -Mg (Mg-doped with zinc) was observed for the ZK60 Mg alloy sample. The magnified view of the XRD graph on the y-axis showed the presence of the intermetallic compound β (Magnesium Zinc, MgZn_2) along with α -Mg. The relatively low relative intensity of the β -phase peaks indicated that the β -phase had a smaller volume fraction that could be related to a lower proportion of Zn (4.94%). The α -Mg peaks were presented at the d spacing of 2.451, 2.605, 2.778, 1.473, 1.900, 1.366, 1.604, 1.343, and 1.302 Å, corresponding to 2θ positions of 36.6, 34.3, 32.1, 63.0, 47.8, 68.6, 57.3, 69.9 and 72.4 degrees, respectively. On the other hand, the peaks of the β -intermetallic compound were presented at the d spacing of 2.184, 2.227, 2.411, 1.466, and 1.710 Å, corresponding to 2θ positions of 41.3, 40.4, 37.2, 63.3 and 53.5 degrees, respectively. Similar phases have been reported in the literature for extruded ZK60 Mg alloy [20]. Moreover, the SPD did not change the phases of ZK60 Mg alloy, either α -Mg or MgZn_2 , as mentioned by Jeong and Kim [21].

The optical microstructure of the cross-section of the ZK60 alloy before and after CEE processing at different temperatures is shown in Figure 3. The microstructure of as-extruded ZK60 Mg alloy exhibited rather heterogeneous, the α -Mg grain size was not uniform, and some α -Mg elongated grains were distributed within the matrix (Figure 3a). Small grains of α -Mg might be formed due to DRX during extrusion. The average α -Mg grain size of as-extruded ZK60 Mg alloy was approximately 38.4 μm . The grain size was determined using the linear intercept method. The CEE processing of ZK60 Mg alloy at different temperatures significantly changed the microstructure. After CEE processing at 190 °C, the microstructure displayed more finely recrystallized α -Mg grains. They were approximately 9 μm in size (Figure 3b). This type of microstructure was produced by DRX. After CEE processing at 270 °C, the α -Mg grains became reasonably homogeneous due to DRX. Slightly grain growth with a grain size of approximately 11 μm was recognized in Figure 3c. Increasing the CEE processing temperature to 350 °C showed slightly coarse equiaxed α -Mg grains. The α -Mg grain size of approximately 16 μm due to DRXed grain growth was identified in Figure 3d.

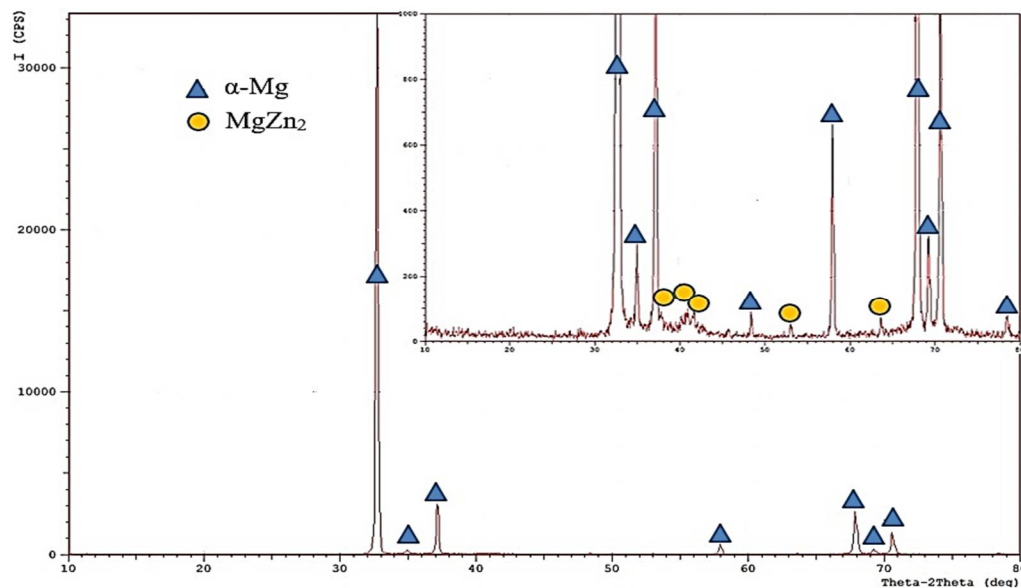


Figure 2: XRD patterns of as-extruded ZK60 Mg alloy (the incorporated graph is the magnified view along the y-axis)

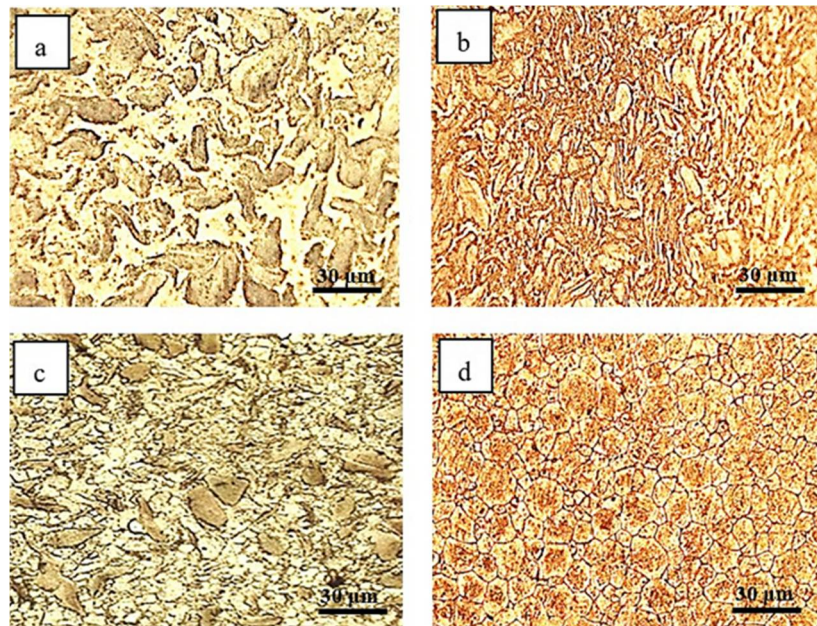


Figure 3: Microstructure of (a) as-extruded ZK60 Mg alloy processed using CEE at different temperatures of (b) 190 °C, (c) 270 °C, and (d) 350 °C

3.2 Hardness

The Vickers hardness test was utilized to reflect the ability of a metal surface to resist plastic deformation. Figure 4 shows the hardness values for the as-extruded and CEEed ZK60 Mg alloys as determined by the hardness test. The average hardness value was significantly affected by the processing temperature. The average hardness value of the Mg alloy processed at 190 °C was increased to 86 HV compared to that of the as-extruded sample (72 HV). This increment in hardness was due to the refinement of the microstructure due to DRX. Increasing the processing temperature to 270 °C yielded a reduction in the hardness value (83 HV) compared to the hardness value at the processing temperature of 190 °C. An additional decrease in hardness appeared with increasing processing temperature to 350 °C (80 HV). The decrease in the hardness value with increasing processing temperature was attributed to the coarsening of the microstructure caused by the growth of the DRXed grains due to the inhomogeneous diffusion [22].

3.3 Mechanical Properties

Figure 5 presents the tensile stress-strain (σ - ϵ) curves of the as-extruded sample and the CEE processed sample at different temperatures. The values of the yield strength (YS), ultimate tensile strength (UTS), and elongation (EL) of the as-extruded sample were 270 MPa, 375 MPa, and 26%, respectively. These values were lower than those of the CEEed ZK60 Mg sample at a processing temperature of 190 °C, which possessed 335 MPa YS, 410 MPa UTS, and 36% EL. It should be noted that as the processing temperature increased from 190 °C to 270 to 350 °C, the YS decreased from 270 MPa to 265 MPa and then to 210 MPa, respectively. In contrast, the UTS of the sample decreased from 410 MPa to 335 MPa and then to 310 MPa when the processing temperature was increased from 190 through 270 to 350 °C, respectively. Furthermore, the EL of the sample decreased from 36% to 30% and then to 28% when the processing temperature was incremented from 190 through 270 to 350 °C, respectively. As mentioned before, the grain size of the sample processed at 190 °C was lower than that processed at 270 and 350 °C. This pointed out that the processing temperature of 190 °C was preferable for DRX than 270 and 350 °C due to the growth of DRXed grains with increasing the processing temperature. During the deformation process, twin formation was inhibited due to crystallographic texture change and grain refinement, thus increasing activation of non-basal slip systems and grain boundary sliding, particularly with increasing deformation temperature [23]. The formation of a strong crystallographic texture resulted in anisotropy in the mechanical properties. In general, the operation of dislocation slip increases with increasing temperature [24]. Therefore, the dislocations of the sample processed at 270 and 350 °C were more likely to slip than the sample processed at 190 °C. Moreover, the sample processed at 350 had a more favorable slip than the sample processed at 190 and 270 °C. The texture is also known to affect the mechanical properties of Mg alloys as weaker texture impairs the mechanical properties [25]. Moreover, the texture strength decreases significantly with increasing temperature [26]. From this, the reason for this significant difference was that YS, UTS, and EL were affected by coarsening of the α -Mg grain size and attributed to the activation of slip systems with increasing temperature [27]. Therefore, we can deduce that the CEE process enhanced the mechanical properties of the ZK60 Mg alloy, especially with the low processing temperature (190 °C). In contrast, the deterioration in mechanical properties was greater with increasing processing temperature (270 and 350 °C) compared to what was observed in the as-extruded ZK60 Mg alloy.

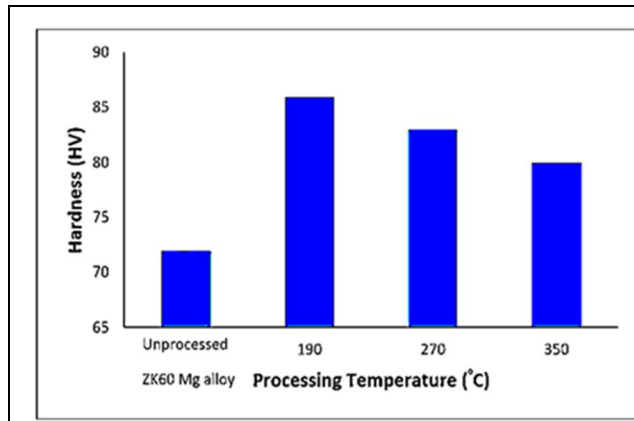


Figure 4: Hardness versus CEE processing temperature of ZK60 Mg alloy

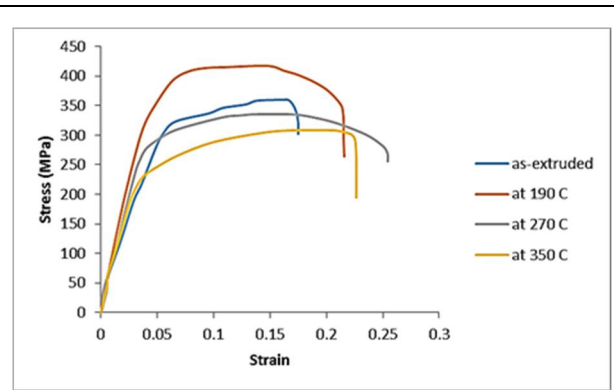


Figure 5: σ - ϵ curves of as-extruded and processed ZK60 Mg alloy using CEE at different temperatures

The differences in fracture morphology between as-extruded and severely deformed ZK60 Mg alloy using CEE at different processing temperatures can be seen from SEM fractography in Figures 6a to d. The fractography images, Figure 6a,b, mostly showed the cleavage fracture. The cleavage steps pointed out a typical brittle fracture in ZK60 Mg alloy in both the as-extruded or CEE-processed state. However, dimples were recognized along the fracture surface, indicating that the fracture was ductile (Figure 6a-d). Increasing the processing temperature increased the number of dimples, reduced their sizes, and made them more uniform on the fracture surface, indicating a significant improvement in the plasticity (Figure 6b-d). The transition in the fracture mechanism from brittle fracture to ductile fracture was mostly affected by grain refining and texture modification [28]. Therefore, the fracture mode of ZK60 Mg alloy was a combination of brittle and ductile fracture.

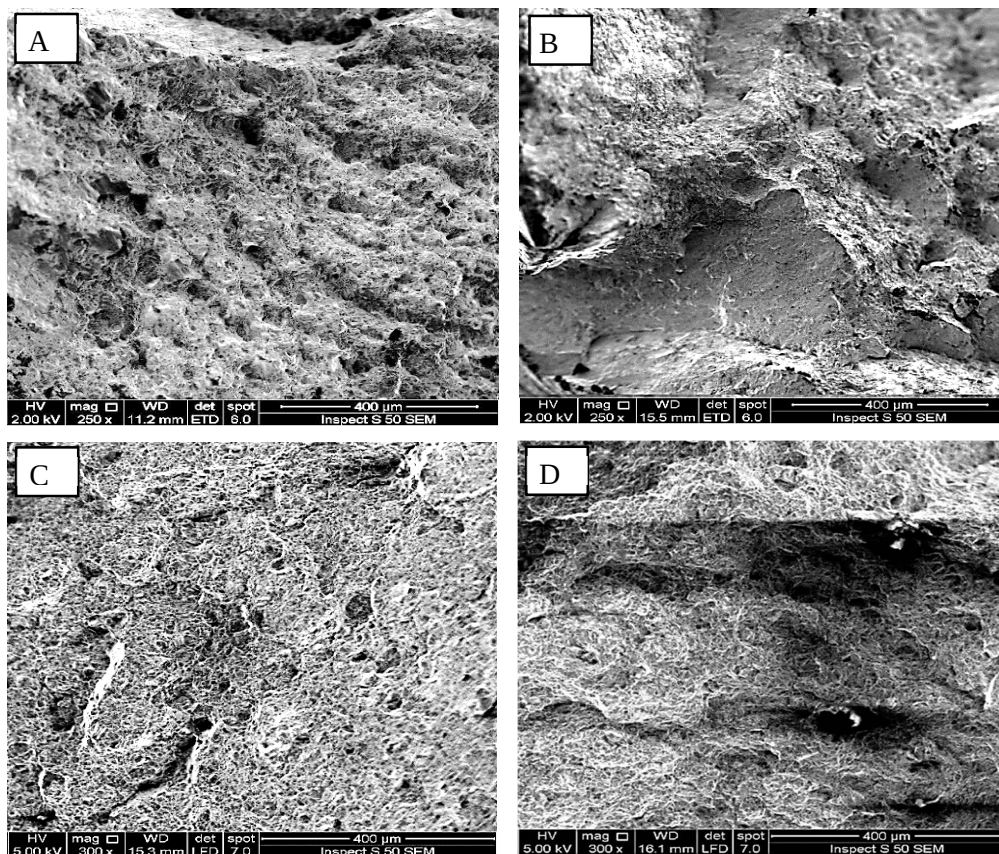


Figure 6: SEM fractography of as-extruded ZK60 Mg alloy (a) and CEEed ZK60 Mg alloy at a processing temperature of (b) 190 °C, (c) 270 °C, and (d) 350 °C

4. Conclusion

The conclusions based on the present experiments can be summarized as follows.

1. The microstructure of the as-extruded ZK60 Mg alloy was heterogeneous with generally coarse and elongated α -Mg grains and an average size of 38.4 μm . Whereas the microstructure of ZK60 alloy processed using CEE was refined at a processing temperature of 190 °C as the average grain size of α -Mg was reduced to about 9 μm due to DRX. Augmenting the processing temperature yielded a slight coarsening of the α -Mg grains to 11 and 16 μm at processing temperatures of 270 and 350 °C, respectively.
2. The hardness increased from 72 HV for the as-extruded Mg sample to 86 HV for the Mg sample processed at 190 °C due to grain refinement as a result of DRX. However, increasing the processing temperature to 270 and 350 °C reduced the hardness to 83 HV and 80 HV, respectively, compared to the hardness value at the processing temperature of 190 °C. This is attributed to the growth of the DRXed grains.
3. The mechanical properties of ZK60 Mg alloy were improved after the CEE process. The optimum properties of 335 MPa YS and 410 MPa UTS and 36% EL were obtained due to grain refining at a processing temperature of 190 °C.
4. Macroscopic fracture modes in as-extruded and CEE-processed ZK60 Mg alloy samples tended to be a combination of ductile and brittle fracture, irrespective of the condition of the Mg alloy sample.

Acknowledgment

The authors would like to express their sincere appreciation to the Department of Production Engineering and Metallurgy, the University of Technology-Iraq, which allowed us to complete this work.

Author Contribution

Methodology, Ahmed Mousa Marheb; Akeel Dhahir Subhi, Writing-Original Draft Preparation, Akeel Dhahir Subhi; Writing-Review & Editing, Ahmed Mousa Marheb; Akeel Dhahir Subhi. "All authors have read and agreed to the published version of the manuscript".

Funding

This research received no external funding.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of Interest

The authors declare that there is no conflict of interest.

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