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Microstructural Optimization of ZK60 Magnesium Alloy Through ECAP Processing Parameters

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Abstract

This study examines the influence of Equal Channel Angular Pressing (ECAP) processing parameters on the microstructural evolution of ZK60 magnesium alloy, to identify the optimal conditions for achieving optimal grain refinement. Widely ZK60 recognized for its high specific strength its hexagonal close-packed (hcp) crystal structure limits slip activity and reduces ductility at room temperature. ECAP provides an effective means of imposing high shear strain without altering the billet dimensions, promoting dynamic recrystallization (DRX) and refining the microstructure. In this work, ZK60 billets were processed through a single ECAP pass at different temperatures (200, 275, and 350°C) and ram speeds (60, 300, and 480 mm/min). The results showed that low processing temperatures, particularly 200°C, combined with a low ram speed of 60 mm/min, yielded the highest degree of DRX and the finest equiaxed grains, with an average grain size of approximately 8 µm. Increasing temperature or RAM speed reduced the extent of grain refinement due to accelerated recovery and grain growth. Optical microscopy and XRD analyses confirmed that changes in grain size, phase distribution, ECAP temperature, and ram speed strongly control strain accumulation and DRX activity, and that the combination of 200°C and 60 mm/min provides the most effective conditions for microstructural optimization of the ZK60 alloy.

Keywords: *ZK60 magnesium alloy; Equal Channel Angular Pressing (ECAP); Microstructural refinement; Dynamic recrystallization (DRX); Processing temperature; Ram speed; Grain size; Severe plastic deformation (SPD).*

Introduction

Magnesium alloys have gained considerable attention in modern engineering and biomedical fields because of their extremely low density and high strength-to-weight ratio. For lightweight structural components, automotive applications, and biodegradable medical implants, these qualities make them attractive. ZK60 (Mg–Zn–Zr) stands out among the available magnesium alloys because of its excellent combination of strength, resistance, and biocompatibility. Despite these advantages, by their low ductility and formability at room temperature the practical use of magnesium alloys remains limited. According to Wei (2018) and Wang et al. (2021), this limitation mainly stems from their hexagonal close-packed (hcp) crystal structure, which provides fewer active slip systems, leading to uneven plastic deformation and early failure during typical forming processes [1], [2].

A group of researchers has increasingly focused on advanced deformation techniques that can produce refined and stable microstructures to overcome inherent crystallographic limitations. A severe plastic deformation (SPD) methods have proven effective for improving the mechanical properties of magnesium alloys through extensive grain refinement. Equal Channel Angular Pressing (ECAP) has shown particular success, as it imposes significant plastic strain without altering the billet's overall dimensions Among these techniques. This unique feature allows for multiple deformation cycles while maintaining geometric stability. As a result, Hashemi (2023) highlights that ECAP promotes dynamic recrystallization (DRX), alters crystallographic texture, and degrades secondary intermetallic phases, thereby enhancing the strength, ductility, and corrosion resistance of magnesium alloys [3].

depends on the complex interaction between processing temperature and ram speed The microstructural response of ZK60 alloy during ECAP processing depends not only on the magnitude of

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the imposed strain. These factors directly influence the rate of dislocation multiplication, the nucleation and growth of dynamically recrystallized grains, and the competing recovery and grain growth mechanisms. According to He et al. (2010), over-processing temperatures typically promote strain accumulation and prevent excessive grain coarsening, while moderate ram speeds allow enough time for uniform DRX development [4]. Conversely, R. Ding et al. (2010) reported that higher temperatures or very high ram speeds accelerate recovery processes and induce adiabatic heating, both of which reduce the efficiency of grain refinement [5]. Although there is a benefits of ECAP for magnesium alloys, the combined and individual effects of temperature and ram speed on the microstructural evolution of ZK60 alloy still need further, more detailed investigation.

Accordingly, the present work is designed to clarify how controlled variations in ECAP temperature (200–350°C) and ram speed (60–480 mm/min) influence the grain size, dynamic recrystallization behavior, and phase morphology of ZK60 magnesium alloy subjected to a single ECAP pass. were employed Optical microscopy and X-ray diffraction to capture the resulting microstructural changes, to correlate them with the applied deformation conditions [6].

Literature Review

Magnesium alloys have garnered sustained scientific interest due to their exceptional combination of low density and high specific strength, rendering them highly suitable for both structural and biomedical applications. Nonetheless, their mechanical performance is predominantly influenced by their crystallographic characteristics. The hexagonal close-packed (hcp) crystal structure limited available nuber slip systems at ambient temperature, resulting in reduced ductility and non-uniform plastic deformation under conventional loading conditions [1], [2]. Therefore, the development of stable and refined microstructures in magnesium alloys remains a scientific and technological challenges.

Focusing on advanced deformation techniques capable of imposing large plastic strains while controlling microstructural evolution. The SPD has emerged as one of the most effective approaches for producing ultrafine-grained magnesium alloys. Within this category, ECAP has attracted particular attention for its ability to impart extremely high shear strain without altering the billet's external dimensions. Hence, this process of ECAP, which promotes dynamic recrystallization, alters crystallographic texture and fragments intermetallic phases, thereby enhancing strength, ductility, and corrosion resistance. Therefore, according to Hashemi et al. (2023), the general principle of ECAP and the intersecting channel configuration are illustrated schematically in Figure 1 [3].

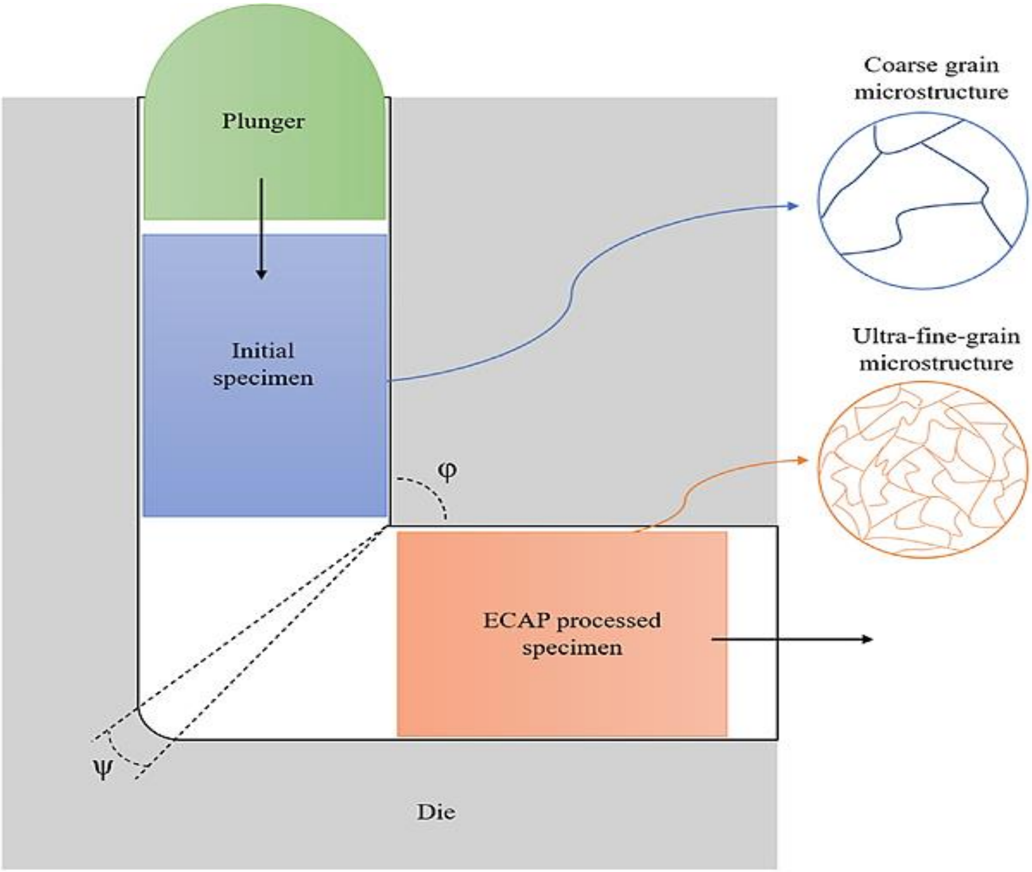

Figure 1 Schematic presentation of ECAP process [3].

Remarkable ability of ECAP to refine grains in magnesium alloys, Figueiredo and Langdon (2008), observed the formation of ultrafine grains in ZK60 alloy, with grain sizes reduced to approximately 0.8 µm after ECAP, influenced by the initial material condition and the chosen processing route. Figueiredo and Langdon (2008), findings highlight the initial microstructure, alloy chemistry, and deformation pathway significantly affect the final microstructural uniformity, with route Bc generally yielding the most consistent refinement. Similarly, Rengen et al. (2010) studied the microstructural evolution of ZE41 magnesium alloy, noting a substantial decrease in grain size from nearly 50 µm to about 2 µm after six ECAP passes. clearly these results demonstrate that the efficiency of grain refinement depends on the magnitude of accumulated strain, deformation temperature, and the interaction between alloying elements and dislocation activity during severe plastic deformation. In particular, the presence of zinc and zirconium in ZK60 plays a crucial role in its response to ECAP. Zinc enhances solid-solution strengthening, while zirconium acts as an effective grain refiner during solidification. The alloy typically features a well-dispersed $MgZn_2$ phase along with fine zirconium-rich particles, both serving as preferred sites for dynamic recrystallization. Consequently, ZK60 tends to undergo significant grain refinement during ECAP, even at relatively low deformation temperatures. A representative microstructure of the as-received ZK60 alloy is shown in Figure 2 below.

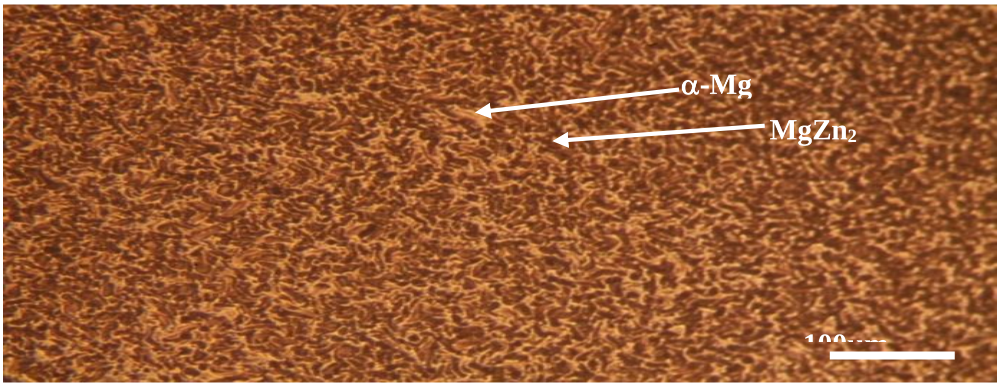

Figure 2 Optical image showing the microstructure of as-received ZK60 Mg alloy

At lower temperatures Processing temperature is one of the most influential parameters controlling microstructural evolution during ECAP, dislocation storage is significantly enhanced by restricted recovery, thus encouraging the nucleation of dynamically recrystallized grains. Temperatures near 200°C are particularly effective in refining ZK-series magnesium alloys, as they generate high dislocation densities and stimulate discontinuous dynamic recrystallization [3]. This is when the deformation temperature exceeds the critical DRX threshold, recovery and grain coarsening become dominant, thereby reducing refinement efficiency and causing texture softening [7]. The ram speed influences the strain rate during ECAP and significantly impacts heat generation. Lower ram speeds allow enough time for the nucleation and growth of DRX grains, resulting in more uniform microstructures. Conversely, higher ram speeds cause increased adiabatic heating, speed up recovery, and often lead to heterogeneous microstructures with incomplete recrystallization. Previous studies have indicated that excessively high ram speeds hinder grain refinement, while moderate speeds support consistent microstructural development [8]. Another important outcome of ECAP processing is Txture evolution. ZK60 magnesium . In the as-received condition alloy has a strong basal texture that greatly limits formability. During ECAP, intense shear deformation reorients the grains, activates non-basal slip systems, and reduces the basal texture. This texture change is crucial for improving ductility and enabling more uniform plastic deformation under service conditions.

Methodology (Rewritten in Professional Academic Style)

Materials

The material used in this study was ZK60 magnesium alloy, supplied as extruded bars with the chemical composition shown in Table 1. The alloy represents a magnesium–zinc–zirconium system with good response to plastic deformation and dynamic recrystallization. The as-received microstructure contained elongated grains and a visible distribution of $MgZn_2$ phases along grain boundaries.

Table 1 Chemical Composition of As-Received ZK60 Mg Alloy (wt.%).

Mg	*Zn*	*Zr*	*Other Elements*
Balance	4.00	0.622	< 0.1

ECAP Die and Equipment

The ECAP experiments were performed using a die consisting of two channels of equal cross-section intersecting at an angle of 90°. This configuration produces simple shear deformation while maintaining the original dimensions of the billet. The die structure and its configuration are shown in Figure 3 and Figure 4 below. Pressing was carried out on a hydraulic universal testing machine of type INSTRON WDW-200E, which provided stable loading conditions and controlled ram-speed operation.

a

Figure 3 Die of ECAP

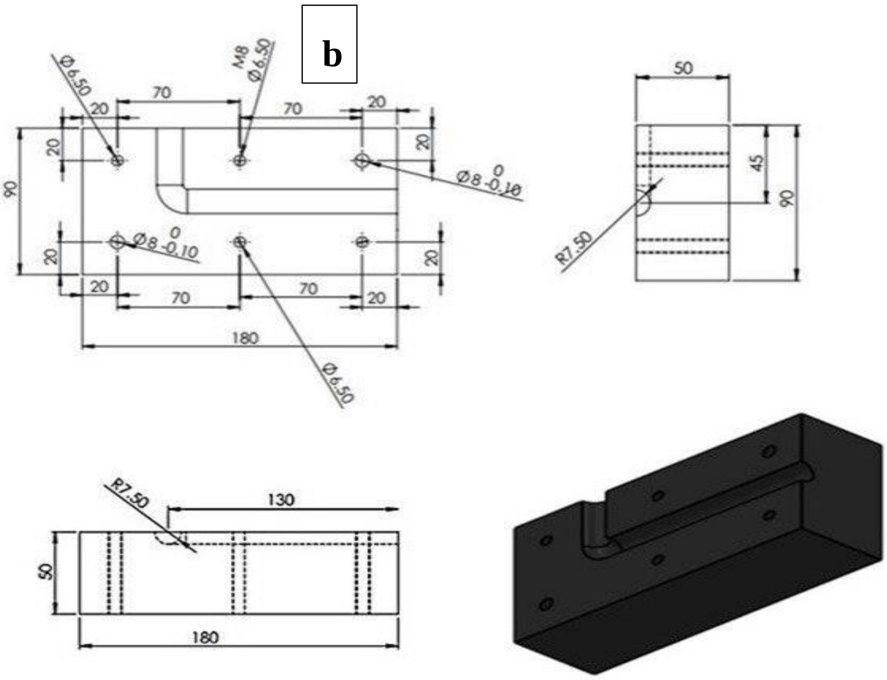

Figure 4 The schematic of die.

ECAP Processing Parameters

A single ECAP pass was applied to all samples in order to ensure that the direct influence of the selected processing parameters could be clearly isolated and evaluated. Two key variables were systematically controlled during deformation: processing temperature and ram speed

Processing Temperature

A Three deformation temperatures were selected according to the known deformation characteristics of the ZK60 magnesium alloy, namely 200°C, 275°C, and 350°C. This temperature range spans the onset of dynamic recrystallization at lower temperatures and extends to the region where recovery and grain coarsening progressively dominate the deformation behavior at higher temperatures

Ram Speed

Three different ram speeds were adopted to investigate strain-rate effects: 60 mm/min, 300 mm/min, and 480 mm/min. The lower ram speed allows for longer deformation time and promotes uniform dynamic recrystallization, while higher speeds increase heat generation and affect the thermal stability of the newly formed grains.

Processing Route

Route A was chosen for all experiments, where the billet is pressed without rotation between passes. Using a single pass under Route A allows for a direct assessment of the individual effects of temperature and ram speed on microstructural evolution, without the additional strain buildup that comes with multiple ECAP passes. The overall pressing sequence is shown schematically in Figure 5 below.

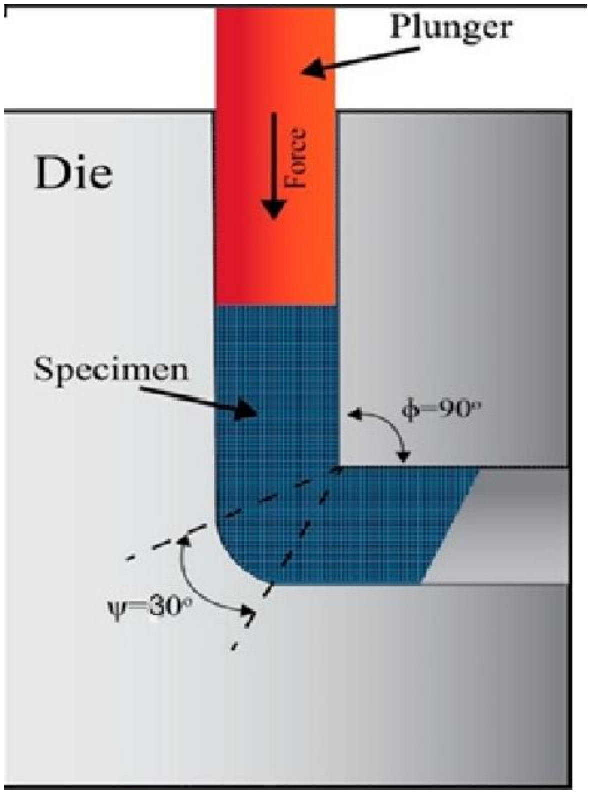

Figure 5 Schematic of ECAP process.

Sample Preparation

From the original ZK60 bars samples were machined to match the dimensions of the ECAP die channels. After deformation, the processed samples were cut along both the length and width for metallographic analysis. Surface preparation involved sequential grinding with silicon carbide papers ranging from grit 320 to 2000. Final polishing with diamond suspensions produced a mirror-like finish, making the grain boundaries clearly visible during microstructural examination.

Microstructural Characterization

Microstructural characterization was carried out using a combination of complementary analytical techniques to accurately identify deformation-induced structural changes, providing a clearer understanding of the material's behavior.

Optical Microscopy (OM)

Optical microscopy was utilized to observe the grain structure and measure the average grain size for each processing condition. The OM images allowed for a direct comparison, both visually and quantitatively, between the as-received alloy and the specimens processed through ECAP.

X-Ray Diffraction (XRD)

X-ray diffraction was employed to identify the constituent phases and to detect any alterations in crystallographic texture subsequent to ECAP deformation. The diffraction patterns acquired were compared with the reference pattern of the as-extruded ZK60 alloy previously illustrated in Figure 2.

Scanning Electron Microscopy (SEM)

Scanning electron microscopy offered higher-resolution views of the microstructure, especially regarding grain boundary features and the spatial distribution of secondary phases within the matrix.

Grain Size Measurement

The grain size was carefully evaluated using the line-intercept technique, following established metallographic procedures. Multiple measurements were taken across several fields to ensure the accuracy and reliability of the data. These steps helped us obtain precise and consistent results. The grain size values, determined under various temperature and ram-speed conditions, are summarized comprehensively in Table 2 below, providing a clear overview of the findings.

Table 2 Average Grain Size of ZK60 Alloy after Single-Pass ECAP

Ram speed(mm/min)	Temperature(°C)	Grain size(μm)
As received	-	35.5
60	200	8
300	200	13
480	200	18
60	275	20
300	275	23
480	275	25
60	350	27
300	350	28.5
480	350	30

Experimental Workflow

The full sequence of the experimental procedure is detailed below, starting with material preparation. This is followed by the ECAP pressing process and concludes with microstructural characterization. To help visualize these steps, we have included an experimental flowchart in Figure 6, this provides a clear overview of each stage, making it easier to understand the overall process.

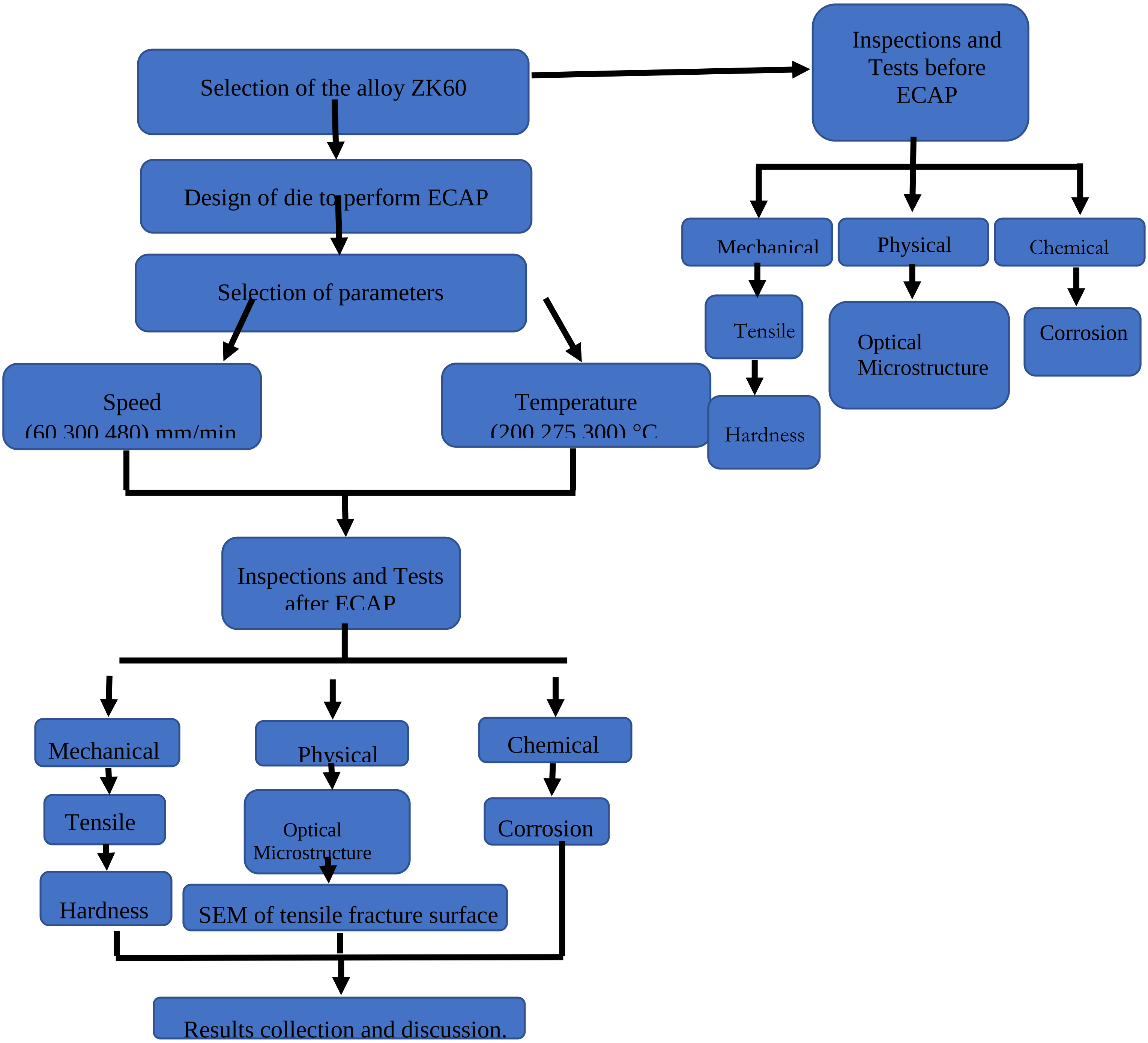

Figure 6 : Flowchart of experimental procedure

Therefore, this methodology offers a consistent framework for evaluating how ECAP temperature and ram speed affect microstructural refinement in ZK60 magnesium alloy.

Results

Microstructure of the As-Received Alloy

The as-received ZK60 magnesium alloy exhibited a heterogeneous microstructure composed of course, elongated α-Mg grains with an average size of 35.5 µm. Secondary $MgZn_2$ intermetallic were distributed predominantly along grain boundaries, contributing to the alloy's initial strength but also producing microstructural non-uniformity. The optical micrograph of the base material is presented in Figure 2 the Optical image of as-received ZK60 Mg alloy. Phase identification by XRD confirmed the presence of α-Mg as the main phase and $MgZn_2$ as the secondary phase, with the diffraction pattern shown in Figure 7 below

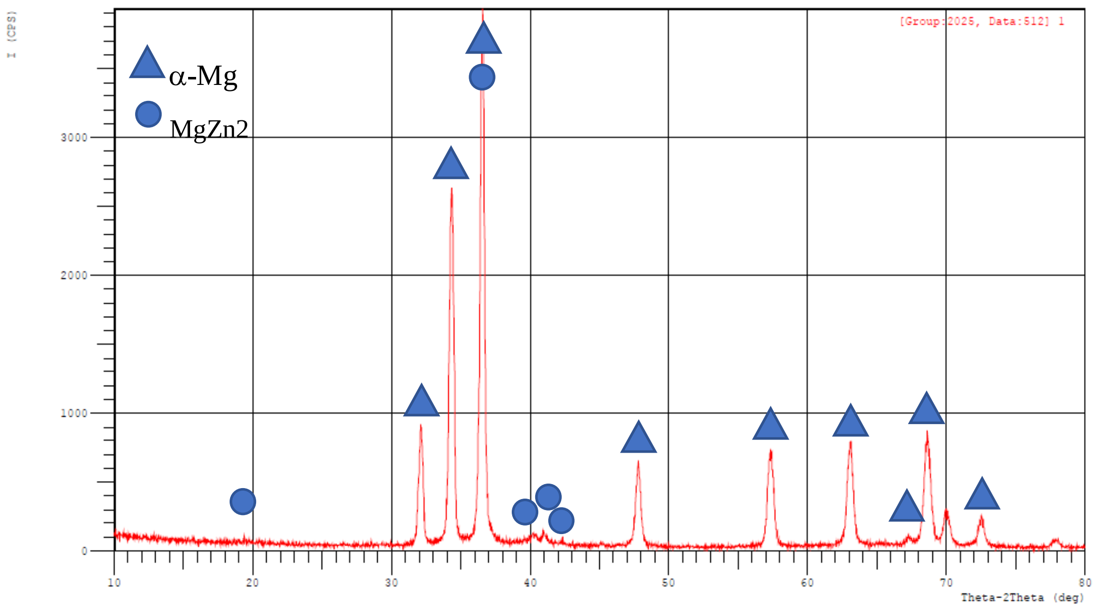

Figure 7 XRD pattern of as-extruded ZK60 Mg alloy.

Microstructural Evolution After ECAP Processing

Substantial microstructural refinement occurred following a single-pass ECAP, primarily due to dynamic recrystallization (DRX). The degree of grain refinement was strongly dependent on both temperature and ram speed. Representative optical micrographs illustrating the combined effects of temperature and ram speed are provided in Figure 8 below.

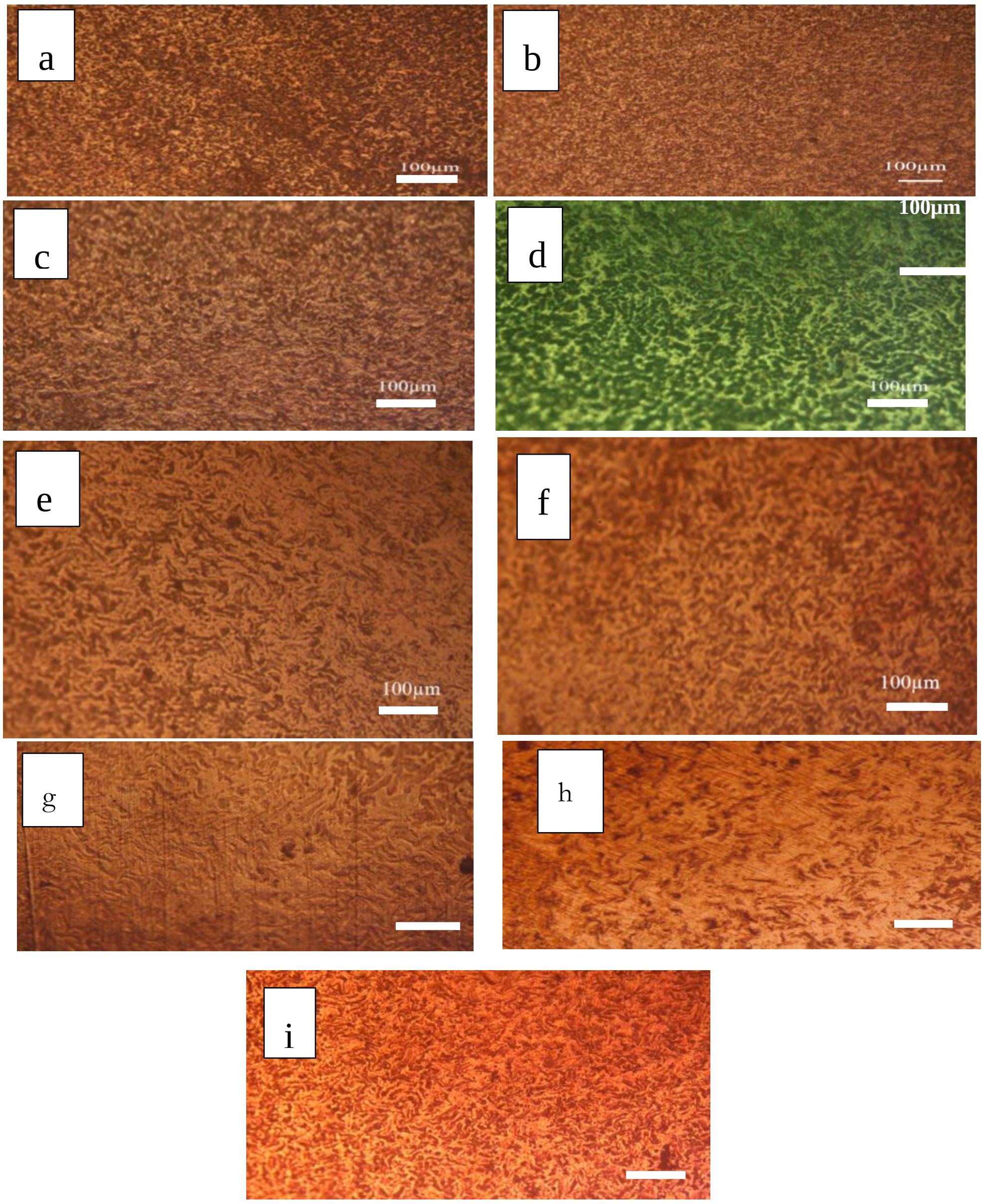

Figure 8 Optical photograph showing the microstructure of ZK60 magnesium alloy after ECAP processes at different parameters: (a) 200°C, ram speed 60 mm/min, (b) 200°C, ram speed 300 mm/min, (C) 200°C, ram speed 480 mm/min, (d) 275°C, ram speed 60 mm/min, (e) 275°C, ram speed 300 mm/min, (f) 275°C, ram speed 480 mm/min, (g) 350°C, ram speed 60 mm/min, (h) 350°C, ram speed 300 mm/min, and (i) 350°C, ram speed 480 mm/min.

Processing at 200°C

At the lowest processing temperature a 60 mm/min resulted in the finest microstructure, with average grain size reduced from 35.5 µm to 8 µm. DRX produced ultrafine grains and dense subgrain boundaries, with clear evidence of slip and twinning accumulation. Increasing ram speed to 300 mm/min increased grain size to 13 µm, producing a less homogeneous microstructure with more regions of elongated, deformed grains. At 480 mm/min, grain size increased further to 18 µm due to adiabatic heating, which accelerated recovery and limited refinement efficiency. These results highlight the sensitivity of ZK60 alloy to strain rate at low temperatures.

Processing at 275°C

At 275°C, microstructural refinement shows sensitivity to ram speed. At 60 mm/min, grain size was about 20 µm with high-angle boundaries. Increasing to 300 mm/min raised it to 23 µm, due to strain-rate heat. At 480 mm/min, it grew to nearly 25 µm, showing adiabatic heating effects. Higher ram speeds favor grain coarsening.

Processing at 350°C

At the highest processing temperature of 350°C, thermal activation primarily influenced the microstructural response. At a ram speed of 60 mm/min, dynamically recrystallized grains with an average size of about 27 µm formed, indicating that grain growth became more apparent under higher thermal conditions. When the ram speed increased to 300 mm/min, the grain size slightly increased to around 28.5 µm, with α-Mg grains remaining evenly distributed but noticeably larger. At the maximum ram speed of 480 mm/min, the largest grains, nearly 30 µm in size, were observed. This clearly demonstrates the combined effect of high temperature and adiabatic heating. Although dynamic recrystallization still occurred at 350°C, grain growth dominated the overall microstructural development. A Two emerge cleared below:

Effect of Temperature

At a constant ram speed, an increase in processing temperature consistently resulted in larger grain sizes. The finest grains were obtained at 200°C and 60 mm/min, where the grain size reached approximately 8 µm. In contrast, the coarsest grains formed at 350°C and 480 mm/min, with a grain size approaching 30 µm. This trend highlights the dominant role of thermal activation in promoting recovery and grain boundary migration at elevated temperatures

Effect of Ram Speed

Lower ram speeds produce finer, more homogeneous microstructures across all temperatures. The smallest grains occur at 60 mm/min, while 480 mm/min results in coarser microstructures due to increased adiabatic heating. The best grain refinement in ZK60 magnesium alloy is achieved by combining low temperature with slow ram speed.

XRD Analysis

X-ray diffraction analysis conducted post-ECAP processing demonstrated that no novel phases emerged under any of the deformation conditions applied. The alloy preserved its original α-Mg matrix alongside $MgZn_2$ secondary phases. However, minor fluctuations in peak intensity were observed, suggesting partial alteration of the crystallographic texture resulting from severe shear deformation. The retention of α-Mg and $MgZn_2$ peaks, consistent with the as-received state, affirms that the microstructural evolution was predominantly driven by dynamic recrystallization and recovery processes, rather than phase transformation mechanisms.

Discussion

The microstructural evolution of ZK60 magnesium alloy under single-pass ECAP conditions heavily depends on both processing temperature and ram speed. The significant grain refinement seen at lower temperatures and slower ram speeds indicates that dynamic recrystallization (DRX) dominates over recovery and grain growth, aligning with earlier research on Mg alloys processed by ECAP [5], [6]. The as-received material consisted of coarse, elongated α-Mg grains with $MgZn_2$ intermetallics along the boundaries, a structure that generally offers favorable sites for DRX nucleation during intense plastic deformation [4].

Influence of Processing Temperature

Processing temperature was found to be a decisive parameter controlling grain refinement efficiency. At 200°C, the imposed shear strain was accommodated mainly through intense dislocation multiplication and twinning activity, while thermally activated recovery remained limited. Under these conditions, grain size was reduced markedly from 35.5 µm to approximately 8 µm, representing the most refined microstructure achieved in this study. This behavior is consistent with the findings of Figueiredo and Langdon, who demonstrated that lower ECAP temperatures promote strong strain accumulation and early-stage dynamic recrystallization in ZK60 alloy [6]. The restricted recovery at this temperature further stabilizes the newly formed fine grains, in agreement with the observations reported by Ding et al. [5].

Increasing the processing temperature to 275°C kept dynamic recrystallization active but reduced its efficiency. Grain sizes grew to 20–25 µm as thermal activation influenced microstructural changes. Higher atomic mobility speeds up subgrain coalescence and boundary migration, partially counteracting

DRX refinement. This aligns with He et al. (2010), who noted recovery begins to compete with recrystallization at moderate temperatures [4].

At the highest processing temperature of 350°C, grain growth became the dominant mechanism controlling microstructural evolution. The average grain size increased to approximately 27–30 µm, indicating that recovery and boundary migration overshadowed DRX activity. As noted by D. Orlov (2011), thermally induced grain coarsening has been widely documented in ZK-series magnesium alloys processed under SPD conditions at elevated temperatures [9], confirming that 350°C lies beyond the optimal thermal window for effective grain refinement.

Influence of Ram Speed

The ram speed significantly influenced the strain rate and the level of adiabatic heating during ECAP. At all tested temperatures, the lowest ram speed of 60 mm/min consistently yielded the finest and most uniform microstructures. The relatively slow deformation rate provides enough time for DRX nucleation and grain boundary formation while reducing excessive heat buildup. This behavior aligns closely with previous studies on magnesium alloys processed under low strain-rate ECAP conditions [6]. When the ram speed increased to 300 mm/min, the resulting grain sizes became moderately larger. This change indicates partial suppression of grain refinement caused by the higher deformation rate and the shorter time for grain boundary formation. Similar strain-rate effects have been reported by Ding et al. [5], who found that increasing deformation speed decreases refinement efficiency in ZE41 magnesium alloy.

At the highest ram speed of 480 mm/min, adiabatic heating took center stage, causing incomplete dynamic recrystallization and noticeable grain growth. Orlov et al. (2011), also highlighted how sensitive the ZK60 alloy is to thermal rises caused by strain rate, observing similar microstructural coarsening during rapid, severe deformation [9].

Interaction Between Temperature and Ram Speed

The interaction between temperature and ram speed demonstrates the balance between deformation kinetics and thermally activated processes. At low temperatures, DRX efficiency is highly sensitive to heat generated by the ram speed, making refinement strongly dependent on strain rate. At higher temperatures, however, the effect of ram speed diminishes because thermal softening and recovery dominate the deformation behavior. These observations coincide with the microstructural interaction patterns described in earlier magnesium ECAP studies [6], [10].

The micrographs in Figure 4.3 show that although grain refinement decreases with increasing temperature, microstructural uniformity improves. This agrees with findings by Yunbin He et al. who showed improved homogeneity but reduced refinement at elevated deformation temperatures [4].

XRD Observations and Phase Stability

XRD results confirmed the stability of α-Mg and $MgZn_2$ phases throughout all ECAP conditions. No new phases were detected, indicating that microstructural evolution was driven primarily by DRX and recovery rather than phase transformation, consistent with previous reports on ZK60 alloy subjected to SPD [6] ,[4]. The slight changes in peak intensity point to moderate texture evolution, which is a well-documented phenomenon in ECAP-processed Mg alloys [57].

The presence of $MgZn_2$ at grain boundaries may have contributed to DRX nucleation and restricted grain growth, particularly at lower temperatures, supporting similar conclusions drawn by Ding et al. [5] and Orlov et al. [9].

In summary, the experimental results clearly demonstrate that a processing temperature of 200°C and a ram speed of 60 mm/min are the most effective ECAP conditions for producing an ultrafine-grained microstructure in ZK60 magnesium alloy. Under these optimized conditions, intense strain accumulation, extensive dynamic recrystallization, and restricted recovery act in concert to yield the smallest average grain size, approximately 8 µm.

As either the processing temperature or the ram speed increases, the refinement efficiency gradually declines. This decline is due to the increasing influence of recovery mechanisms, grain coalescence, and adiabatic heating, all of which oppose the grain subdivision process. These interconnected thermo-mechanical interactions align closely with the ECAP deformation mechanisms commonly reported in the literature for magnesium alloys [6], [9]. Therefore, the current findings highlight the existence of a narrow and highly sensitive processing window for achieving optimal grain refinement in hcp magnesium alloys. The observed behavior supports established DRX-controlled deformation models and confirms the high sensitivity of ZK60 alloy to both thermal input and strain-rate variations during severe plastic deformation.

Conclusion

The results of the present investigation clearly indicate that the microstructural characteristics of ZK60 magnesium alloy are highly responsive to the processing parameters applied during single-pass ECAP. A pronounced refinement of the grain structure was obtained under low deformation temperature and slow ram speed, which confirms the dominant role of dynamic recrystallization under these conditions. The optimum condition was identified at 200°C and a ram speed of 60 mm/min, where the smallest average grain size of about 8 µm was achieved as a result of efficient nucleation of new grains and the limited influence of recovery mechanisms.

As the ither the processing temperature or the ram speed increased, a progressive decline in refinement efficiency was observed. Grain growth became more pronounced, with grain sizes reaching approximately 30 µm at 350°C and 480 mm/min. This behavior demonstrates that thermal activation, recovery processes, and adiabatic heating increasingly govern the microstructural response at higher deformation conditions, thereby weakening the effectiveness of the imposed ECAP shear strain .

The phase stability confirmed by XRD analysis further supports that the microstructural evolution is driven by deformation-induced mechanisms rather than phase transformations, as the α-Mg matrix and $MgZn_2$ secondary phases remained unchanged throughout all processing conditions. Overall, these findings highlight the crucial importance of precise control over temperature and strain rate in optimizing grain refinement in ZK60 magnesium alloy. Accordingly, single-pass ECAP offers an efficient method for improving the microstructural properties of this alloy, provided that deformation occurs within a narrow and carefully optimized processing window.

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