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Severely Plastic Deformed Magnesium Based Alloys

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Abstract

Magnesium can be replaced with materials which experience strain controlled fatigue in their respective applications. Still, there are infrequent predicaments with utilizing magnesium alloys, comprising lower strength, fatigue life, ductility, toughness, and creep resistant attributes correlate with aluminum alloys. Some recent studies have been affirming that through the severe plastic deformation process, particularly equal-channel angular pressing (ECAP) method promotes very significant ultra-grain refinement in bulk solids, which enhances the mechanical properties. ECAP with a 90° clockwise rotation around the billet axis between consecutive passes in route B_C has improved the ductile characteristics with increased yield strength and rate of elongation which leads to a greater fatigue life because ultra-fine grain refinement can be able to resist the crack propagations. To attain the plasticity at higher temperature magnesium and its alloys are required to undergo extrusion operation before proceeding to the multiple pass ECAP at 200°C because the magnesium alloys exhibit a limited number of slip systems due to its hexagonal crystal structure.

Keywords: magnesium, severe plastic deformation, ECAP, ultra-fine grains, texture

1. Introduction

In today's engineering world, light-weight materials have gained much attention because of demand in low-cost production and increasing efficiency through weight reduction. Correspondingly, Magnesium is one of the materials which attributed in the lightweight material category after aluminum and its alloys. Magnesium and its alloys have some unique application in aerospace and other engineering sectors. But due to some characteristics regarding the manufacturing process such as complex in forming the components and other cold working processes [1]. Particularly in Biomedical application, magnesium has some restriction since Mg dissolves in the body fluid during the restorative process, so degradation of magnesium has to be controlled [2]. One is the low mechanical properties comparing with the bioinert titanium alloys, which makes their application limited in load-bearing implants. Another one is the fast degradation behavior, which involves the gas cavity and high alkaline microenvironment around the implants leading to a poor osteointegration. The mechanical processing of magnesium alloy at elevated temperature influences the grain distribution.

Normally a combination of fine grain structure and coarse grain structure are formed through the various forming operation of wrought materials, and homogeneous grain structures obtained by the cast formed materials [3]. During

cold working, the fine grain formed along with the boundaries in the magnesium wrought materials which through the dynamic recrystallization and the distribution is multi-modal [4]. Normally area of the cross section has been reduced through the total plastic deformation method at certain temperature range. Consequently, the methods mentioned above are not having enough capacity to form homogeneous grain refinement at the initial structure and make the grain structure distribution the multimodal phase [5].

For the last few years, the development of plastic deformation technology, which includes the equal channel angular pressing (ECAP) is emerging [6]. The limitation in the plastic deformation of magnesium alloys will neglect through the SPD technique, so severe plastic deformation of magnesium and its alloys initiate the probability for manufacturing the ultrafine grain materials along with the increased mechanical properties and additionally the superplastic capabilities also experienced by the formed material [7].

Towards the application, ECAP paved a path for refinement of grains in the lightweight materials, particularly in concern aluminum and magnesium alloys. In concern with f.c.c metals, misorientation in the low angles boundaries and alignment of the elongated subgrains in parallel to the primary slip will occur when the sample subjected to severe plastic deformation [8, 9]. Followed by the first pass, the upcoming passes designed to induce further refinement of grains and rearrangement consistent with the dislocation theory for low energy structure [10, 11] after the multiple passes in the ECAP, a set of equiaxial grains which in different angles. The structure changes have been accounted in many metals having f.c.c arrangement, which also constitutes aluminum [12]. On the contrary, by using the ECAP process, UFG can be obtained from the magnesium alloys, and grains get refined by the subsequent passes [13].

During the ECAP process of the magnesium and its alloys, grain refinement depends on the number of passes, channel angle, die angle, and the initial grain structure before to the ECAP. By varying the various parameters of the ECAP process, including the temperature, Mg will exhibit diverse morphology such as grain orientation, distribution, size in multimodal type after SPD [12, 13]. Many studies focused on analyzing the microstructure and the mechanical properties after ECAP processing.

As explained, the pattern specified for the grain refinement channels towards a usual perception of the several articles resembling in the research literature and lead to understanding the ability for the further structure of grain formed under the various condition during SPD of magnesium alloys. In magnesium alloys, grain refinement defined in a simple way [14]. This statement is crucial for highlighting, though, that exact grain refinement mechanism elaborated which authorizes various structure formations based on the experimental circumstances including those primary inferences towards maximum processing steps in SPD of magnesium alloys. Different methods regarding severe plastic deformation consistently engage in developing the process and regarding the nanostructure formation in the samples [15].

Under the certain experimental circumstance getting the ultrafine-grained structure along with predominating boundaries oriented in the high degree of angle and it will be varied according to materials. Furthermore, the nanostructure formation in the alloy during the ECAP process is stable in the entire uni, which required for providing durable characteristics for the metals [7, 13]. Followed by, ensuring the ECAP processed samples for mechanical damages or any penetrated cracks in the material. The other methods of SPD will not meet the requirements as above, such as drawing, hot extrusion, and rolling. Nanostructure formation in the samples is only possible with the mechanical deformations at relatively low temperature as well as optimal parameters for material processing [16].

2. An examination of reports of microstructures produced in Mg and magnesium alloys

To make a competent design, it is essential to analyze different grain structures of magnesium and its alloys, which processed through the ECAP [17]. An initial documented reports organized, and general review presented in **Table 1** for pure magnesium and magnesium alloys, which processed through ECAP.

The outcome in the given **Table 1** indicates the utilization of an ECAP die set up with the 90 internal channel angle, operating with and without back pressure, and the expected outcomes notified in the column number six of **Table 1**. From the table, A and B represent the heterogeneous and homogeneous grain structure,

Alloy	Initial grain size (μm)	Final grain size (μm)	Inter-mediate structure	Structure after multiple passes	Additional information	References
AZ31	48.3	2.5	A	B	—	[18]
AZ31	48.1	1.4	B	B	U = 100 L	[19]
AZ31	2.5	0.7	—	B	U = 110 L	[20]
AZ31	15–22	1	A	B	BP-ECAP (423 K)	[21]
AZ31	5–30	1.9	A	B	Pre-deformed by extrusion	[22]
AZ31	5–30	2.2	A	B	U = 110 L	[23]
AZ31	450	1–3	A	A		[24]
AZ31	20	1–2	—	B	Pre-deformed by hot-rolling	[25]
AZ31	10–20	3.0	A	B		[26]
AZ31	10	3.2	—	B	Pre-deformed by rolling	[27]
AZ31	7–20	2	A	B	Route A	[28]
AZ31	15–22	0.9	A	B	BP-ECAP	[29]
AZ31	28	8	A	—	One pass of BP-ECAP	[30]
AZ61	16	0.62	B	B	Pre-deformed by extrusion	[31]
AZ91		0.5	—	B	Pre-deformed by extrusion	[32]
AZ91	40	1.2	A	B	Route C	[33]
Mg (pure)	400	120	—	B	—	[34]
Mg (pure)	200	20	A	A	—	[35]
Mg (pure)	900	70	A	B	—	[36]
Mg–0.9% Al	100	17	—	B	—	[34]

Table 1.
Comparison of experiments conducted on pure magnesium and a range of magnesium alloy.

respectively. BP indicates the back pressure, and U is for the channel angle within the die. Then ECAP routes defined by route A indicate that ECAP processed without any specimen rotation in-between the two passes, B_A indicates the specimen rotation of 90° in the alternative direction in-between the passes and C denotes the rotation of 180° in-between the passes.

From **Table 1**, we can observe that materials represented in the first column, the grain sizes of the material before and after ECAP provided in the following columns. Then followed by that intermediate stage in the ECAP process is given in the fourth column and the fifth column provided with the additional information regarding the total number of passes. Finally, the references provided in the last column. Additionally, the structure of the grain after processing with the ECAP have given in the notation of Bi-m and Trim to indicate the grain distribution, whether it is tri-modal or Bimodal correspondingly.

From the interference of **Table 1** observation of the distribution of the heterogeneous grain size has done after many passes, particularly while the grain size is large at the initial stage. Certainly, grains were heterogeneous at the initial stage in which the size of the grain ranges from the minimum 45.5 µm for magnesium alloy ZK60 to a higher grain size range of 640 µm magnesium alloy AZ31. From the observation made from the existing investigation, with the minimum number of ECAP passes, the homogeneous grain arrays can obtain with the average grain size of ~40 µm. The high-temperature ECAP processed help to form the homogenization: for instance, the grain size with bi-modal configuration with homogeneity attained with six ECAP passes at the temperature of 423 K in the magnesium AZ31 alloy. In Magnesium-Zn alloy bi-modal distribution is obtained in grain size in which initially the fine and coarse grains have found.

From **Table 1**, there were two significant limitations found; one is the results illustrated that by considering the process limitations, the initial structure might exhibit the homogenous distribution, tri-modal or bi-modal grain size distribution. And another observed one is when the grain size is larger, or it formed in coarse then the bi-modal distribution in the grain size is preferred. These were the two important consideration which made based on the grain structure.

3. A model for grain refinement in Mg and magnesium alloys processed by ECAP

From **Figure 1**, which shows the patterned design model which created based on the grain structure formation of ECAP processed of magnesium alloys. The design raised based on the assumption of grain size and distribution on the initial stage and mechanism which proposed earlier regarding the grain structure.

The various grain structure at the initial stage, which is before the ECAP is shown in **Figure 1a, d, g, and j**. In which d_c , the critical grain size integrates with the concept which effectively reduced the size, which initiates the nucleation with proper homogenization during the processing of the materials. The structure of the grain and its refinement process is given below.

Particularly, the initial structure of the coarse grain at the initial stage with the size, d and the grain size, d , is larger than d_c ($d \gg d_c$), which is the critical grain size which shown in **Figure 1a, d, and g**. From **Figure 1j**, $d < d_c$, which means the initial grain structure is granular than the critical size. In certain, the grains which attained homogenous nucleation in which entire grain structure is non-basal slips. Furthermore a complete has been yet to be considered to improve the standard of the processing parameters, and it is important to expect that it will rely on kind of alloy and temperature for processing of ECAP and present investigations show that back pressure also a dependent factor for the grain structure.

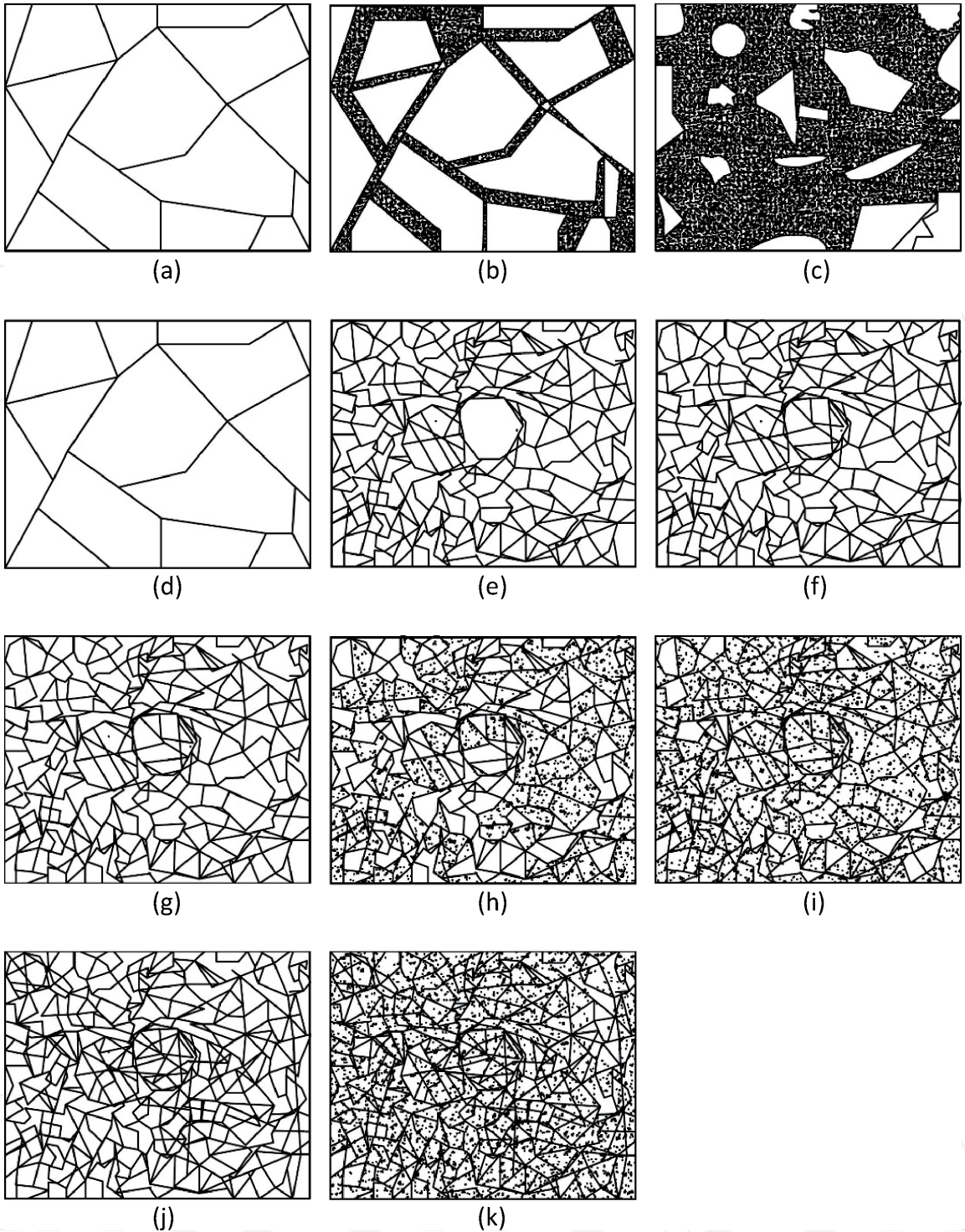


Figure 1. Patterned design on the refinement of grain of the ECAP processed Mg alloys. (a), (d), (g), (j) are the initial grain structure whose grain size can be termed as $d \gg d_c$, $d > d_c$, $d > d_c$ and d_c respectively. (b), (e) and (h) are the intermediate structure at the shear zone during the ECAP. (c), (f), (i) and (k) are the final structure after the pass [8].

From the second column of **Figure 1**, which illustrates the grain structure formed after one pass. Followed by that third column indicated the grain structure formed after more than one number of passes. Where the structure of the initial size of the grain is comparably larger than critical diameter d_c , as shown in the first column of **Figure 1**, the grain structure develops in the initial pass, such as the bimodal or multimodal distribution of the grain as shown in the second column of **Figure 1**. Subsequently, the region occupied by the extended cores of the grains at the initial stage and by the grains which newly formed after refinement have significantly relied on the initial size of the grains which observed during the analysis of

Figure 1. For a better explanation, differentiation among the cores of the initial grains which are usually larger which exist even after one pass of ECAP, the core regions mentioned above illustrated in the second column and the third column of **Figure 1**, in particular recently refined grains which indicated in the dotted region.

In **Figure 1**, the first row illustrates the condition of the grain structure at initial stage which is particularly coarse, because of that even after the one pass of ECAP the initial grains last and hold an extended region which shown in **Figure 1b** and the same grains may get unrefined until multiple passes which are shown in **Figure 1c**. From **Figure 1b**, it can be observed that twinning takes place across the larger grains, so it leads to refinement of grain accompanying with the twinning features. The condition where the critical size of the grain is very smaller than the initial grain size which made bi-modal or multi-modal grain distribution possible which continues even after the multiple passes of ECAP which shown in **Figure 1c**.

In defining the features of the newly developed grains, it is important to explain whether the grain size distribution is multimodal or homogeneous. The d_c grain size variations observed from the first two rows in **Figure 1** were the result of variation in the processing parameters particularly the temperature through the initial structure is similar which illustrated in **Figure 1a** and **d**. The fractional volume of the newly developed grains after one pass is small when compared to the initial structure because it is too coarse which observed from the first row of **Figure 1** and this will be the cause for the formation of bimodal and multimodal distribution among the grain size after the multi passes of ECAP. Subsequently, the same initial structure subjected to the one pass of ECAP with different processing conditions such as lower strain rate and elevated temperature which resulted in the formation of new grains and occupy the extended region which illustrated in **Figure 1e** and also after multiple passes of ECAP the sample exhibits uniform grain structure as illustrated in **Figure 1f**.

After a single pass of ECAP, the grain size distribution becomes bimodal or multimodal by forming reasonably fine structures grains. But the existing grains or grain which not affected by the pass occupied lesser fraction area that the newly formed ones which illustrated in **Figure 1g**. **Figure 1i** shows, by multiple ECAP, passes, the grains get refined and resulted in the homogeneous distribution.

By having the initial grain size as smaller than the critical grain diameter as shown in **Figure 1j**, the homogeneous array distribution achieved through the single pass of ECAP as illustrated in **Figure 1i** and subsequently after many passes homogeneity remains the same.

From the mode, it concluded that bi- and multi-modal grain size distributions which formed through the ECAP were transitional and the distribution of the grains gets altered as ECAP passes the increase.

Spitale et al. processed ECAP in minimal temperature, which is about 250°C with the channel angle of 90° and with 45° of the radius of curvature. The plunger speed for the process is 0.1 mm/s. They observed the evolution of grain structure at different location of the deformation zone. **Figure 2** shows the appearance of the structure entering the deformation zone [37].

The initial structure of the grain exists with the witness of twinning action. Grain boundaries observed with the serration like features. Grains with fine size within the range of 20 μm have witnessed within the region of grain boundaries and twins, which is developed from the initial grain structure, as shown in **Figure 3**. Apart from the certain limits from the grain boundaries and twins, fine grains were not witnessed with then fine grains.

From **Figure 4**, it witnessed that a large area occupied by the fine grains. The fine grains average size is around ~15 μm , which observed from the deformation zone. The fine grain distribution followed the necklace pattern which exists around the area of unrefined grain (>100 μm).

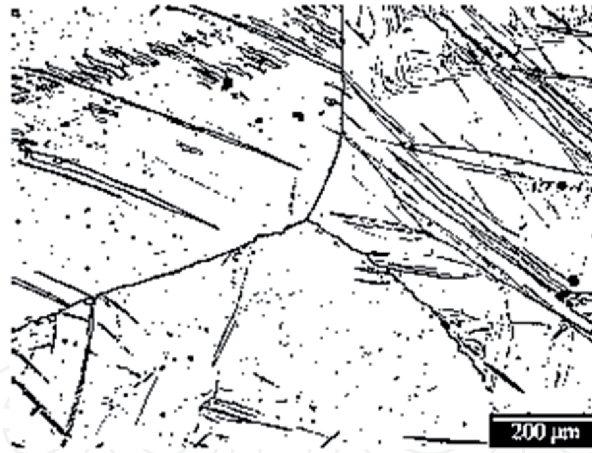


Figure 2.
The grain structure in the region of deformation [37].



Figure 3.
Structure of the grain inside the region of deformation [37].

4. Improved processing routes for ECAP with magnesium alloys

The processing routes influenced the final structure of the grain, failure of billet, and shear localization effect through the grain structure size distribution and refinement mechanism of the magnesium and its alloys. The formation of the shear bands is due to the concentration of the thin layer which belongs to the newly formed grains along with the existing grain boundaries. This shear concentration occurs in a layer due to the nearby regions shear, and it develops damage pile; thus, the failure in the billet takes place. The rise in the initial grain coarse volume leads to the rise in the shear amount, which is in the shear band. The possibilities of the localization of the shear are higher, which is shown in the first row of **Figure 1** successively the growth shown in below rows. This decision is in Correspond with the failure of the coarse grain structure in the magnesium and its alloys.

To rectify the issues in the ECAP process of the magnesium and its alloys, some measures have been improvised and designed such as the processing route along with temperature, die angle and the back pressure range and its usage. To bring down the tenor for shear localization the grain cores along with the boundary extents have to reduce. Thus a primary step of extrusion is made to refine the microstructure which can change the initial grain structure as illustrated as **Figure 1** an into refined grain structure like **Figure 1g** or **j**. As shown in the second row of **Figure 1a**, the grain refinement sequence of the newly formed grains can alter through the rise in the processing temperature. This alteration in the ECAP process



Figure 4.
Grain structure at the end of the deformation zone [37].

can provide a huge volume of formed grains and reduced tensor for localization of shear [14, 24–27, 29]. The accumulation of the damage and the shear bands strain have reduced through the increase in the die angle during the ECAP process. The billet cracking which normally caused by the damage accumulation and the additional grain refinement can achieve by using the back-pressure during the ECAP process of the magnesium alloys.

An alternate method is by subjecting the magnesium alloys to the multiple ECAP as initial pass in the elevated temperature, and the subsequent passes in lower temperature can help to achieve a fine-grain structure with the homogeneous distribution. This process resembles the technique which follows the extrusion process before the ECAP [31].

The customizing of the grain size distribution in the magnesium alloys is associated with the grain structure refinement mechanism of the alloys, which processed through the ECAP process. By changing the ECAP process, it is desirable to alter or control the grain structure and its fine or coarse refinement structure. To increase the certain mechanical properties of the material such as ductility and strength concept of manipulating the material grain boundary and grain refinement was introduced many years ago in the basis of the grain boundary engineering, the same concept have been followed and approached in the process of ECAP. The recent investigations showed that through the ECAP process, the alteration of the grains structure and its distribution achieved in the selected materials. This merit region in the process mentioned above is, for instance, developing a grain distribution in the bimodal configuration in nanostructured Cu, a combination of enhanced ductility and sufficient strength showed up during the tensile testing at ambient temperature which presented in research work. To obtain the sufficient strength and altered elongated grains to increase the stability during the tensile deformation, ECAP method paves the way for these through the forming ultrafine grains and altering the grain size distribution. Among the Grain size distribution, the bimodal configuration considered as the efficient thing for rising the ductility characteristics during the cyclic loading and deformation which assure mechanical properties can improve through the grain size engineering [23–27].

5. Effect of texture

The influence of texture is not straightly integrated with any developed model for the refinement of the grain structure. Unless the developed model is expected to be effective for ECAP processing route and initial texture. This typical thing

supposed due to the forming mechanism of the grain structure along with the grain boundaries has witnessed in the magnesium and its alloys which are having the distinctive initial structures while processing in the various temperature range where ECAP is typically carried out [17]. With the uniform procedure, a recent research work analyzed the texture of magnesium AZ31 which processed through the rolling process and ECAP process and they concluded that texture did not influence the size of the grain which formed after the process meanwhile texture influences the chance of formation of new grains and deformation amount which needed to achieve the deformation.

With the recent proposed modal of ECAP, the progression in the structure of the grain refinement mechanism is witnessed [29]. The bimodal distribution of grain structure observed due to the fine grain refinement and grain nucleate around the boundaries of the grain and twins in the presence of area which does not get affected by the process. A research work [8] provides a technique in which grain which subjected to the recrystallization is nucleate throughout the existing grains in the materials and progress until the structure reaches the homogenous structure equiaxial. The variation among the grain structure refinement modal relies upon the region of nucleation, which tends to form the new grain and feasibility of forming the heterogeneous distribution of grains. Some research delivered that heterogeneous grain size distribution in the magnesium and its alloys.

Because of the coarse grains at the initial stage, the predicted criteria from the developed grain refinement model is getting disapproved with the homogeneous distribution of grain sizes. Possibly grain growth and altered grain structure had observed in the material after subjecting it to the complete ECAP process. In the different stages of the grain refinement, fine grains get nucleated throughout the existing grains and in the region of twin boundaries which happened as expected by the grain structure refinement modal which leads to the distribution of grain size in a heterogeneous manner. The temperature for the intermediate process is likely to prevent the growth of grain and influenced the grain structure to form the bimodal distribution, which witnessed after the ECAP process.

6. Effect of mechanical behavior

The maximum tensile strength is recorded through the compressive tests and in various ways as possible for the material which subjected for three passes of ECAP process which given in **Figure 5**. The data of the received material included for better understanding [30]. It witnessed that the ECAP processed specimen shows elevated flow stress in multiple directions when compared with processed ones.

The material which undergone the ECAP process display an improved yield and maximum yield stress along with improved behavior in the work hardening towards the different compression direction. Significantly the ECAP processed material which tested along the direction of the Y-axis which is perpendicular to the die channel of the ECAP setup shows a concave up like feature with the strain range of 0.02–0.05. Another major witness is that processed sample demonstrates a constant hardening strain range from 0.01 to 0.03 towards the direction of Z-axis and in the direction of X axis it shows 0.01–0.06 in which the hardening rate is slightly get decreased from the initial stage. The processed material showed up a rise in hardening rate in the range of 0.015–0.3045 in the direction of Y-axis.

From the above results, it is clear that ECAP processing ensures the mechanical anisotropy behavior of the commercially pure Magnesium. The appearance of twinning action in the material during the compression test leads to the anisotropy. The incident of increased hardening rate is clearly because of the twinning action

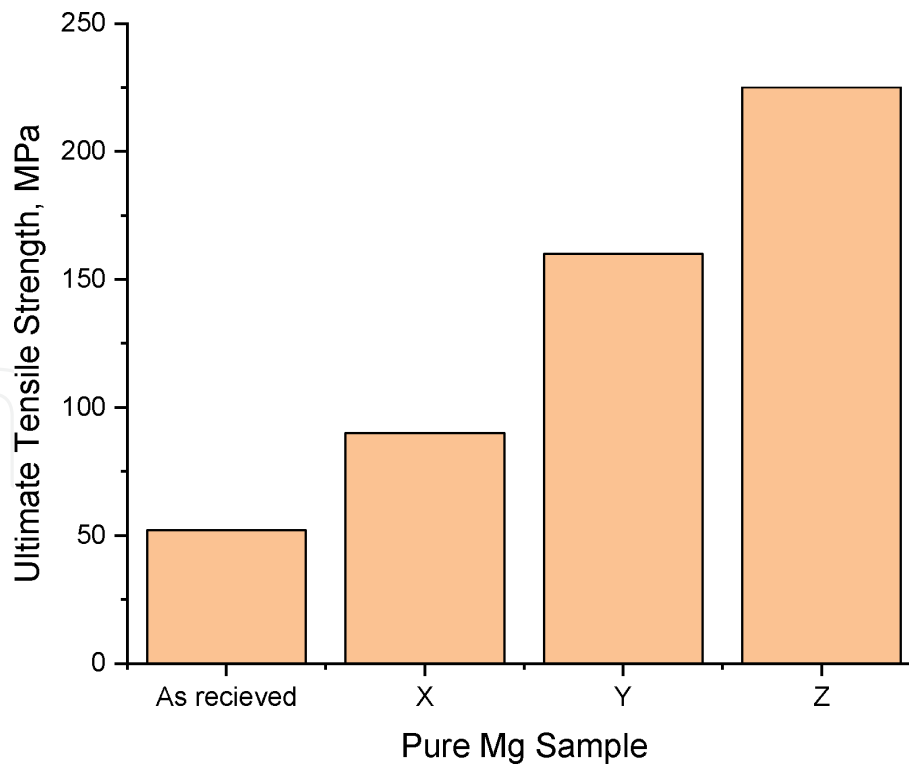


Figure 5.
Ultimate tensile strength obtained for the CP-Mg before and after ECAP processing [30].

which occurred in the direction of Y-axis. Furthermore, the same twinning action indicated in the ECAP processed AZ31 alloy during the compressive test along the direction of the y-axis, which ensured through the microscopic examination. Along with that observation notable amount of twins were witnessed in the magnesium alloy after subjecting it to the strain of only 0.04 (in approx.) in the compressive test along the direction of Y-axis. The twins obtained after a certain level of strain in the corresponding direction was notably lower. Finally, the outcomes represent that after ECAP processing of commercially pure magnesium, the material shows an improved anisotropic behavior and notably twinning action also takes place while the material compressed in the direction of the Y-axis which is perpendicular to the die channel angle. The tensile characteristics of the rolled magnesium are in the good range and lowered elongation when compared to the ECAP processed commercially pure magnesium. These factors indicate that ECAP processing slightly reduces the yield stress and improves the ductile characteristics of the material. And also fine grain refinement was witnessed through the microstructural study of the ECAP processed Magnesium. Thus, the decreased yield stress after the rolling process, along with ECAP contribute to the texture effect of the magnesium.

7. Summary

Magnesium has plenty of tones and choice when equaled to the other metals or nonmetals which have utilized under the category of lightweight materials in automotive, aerospace and biomedical application. Certain advancements are transpiring in magnesium development, and that will engage a fine pathway to the prospect. Among the Severe Plastic deformation (SPD), one of its type Equal Channel Angular Pressing (ECAP) emerged as the technique which can be able to extend the magnesium alloys to the vast application. Among the science group, there is mindfulness about to witness the transcendent application of magnesium and its alloys. Furthermore, enough research and the experimental establishment is

needed for the unique evolution of deformation behavior of the magnesium alloys to achieve aspired microstructures which influence the mechanical properties plus fine shapes for avoiding post processes and further greater transformations of the phase which compromise to permit improvement of magnesium alloys. This very low density concerning magnesium and its alloy collectively among attractive characteristics such as castability is traversing to extended transportation business. The progressed application can begin from an expanded design along with many perceptions of the basic features of magnesium behavior and also the rise of cost-affordable magnesium alloys.

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