International Journal of Engineering Research and Technology. ISSN 0974-3154, Volume 13, Number 11 (2020), pp. 3855-3871
© International Research Publication House. https://dx.doi.org/10.37624/IJERT/13.11.2020.3855-3871

A Comprehensive Review on the Influence of Equal Channel Angular Pressing Parameters on Magnesium Alloys

M.S. Salleh[1,*], A.A. Rahman[1], S.H.Yahaya[1], M.Y.Yuhazri[2] and S. Akmal[2]

[1]Fakulti Kejuruteraan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal Melaka, Malaysia.

[2]Fakulti Teknologi Kejuruteraan Mekanikal dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal Melaka, Malaysia.

**Corresponding author's [1]ORCID: 0000-0003-4800-2470*

Abstract

Equal channel angular pressing (ECAP) is a promising technique to produce an ultra-fine grain microstructure thus improving the properties of the material. Several factors have been acknowledged to have an affect on the microstructure and mechanical properties of the material after an ECAP process which include the operation temperature, number of passes, die angle and also the processing route. Therefore, this review paper gives a concise summary on the influence of ECAP parameters towards the microstructure and mechanical properties of magnesium alloys. Specific attention has been given to the magnesium alloy microstructure evolution and its mechanical properties after ECAP process. It was discovered that refined microstructure with better mechanical properties can be produced at lower temperature, higher number of passes and smaller die angle. It was also noticed that using route B_C of ECAP process may give the best results by contributing about 28% of material hardness compared to the other routes.

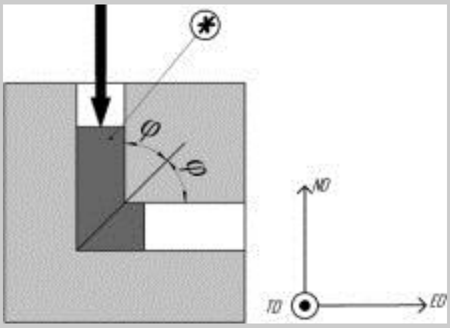
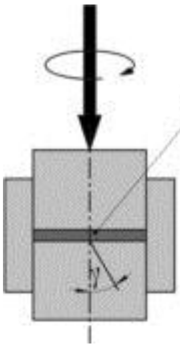
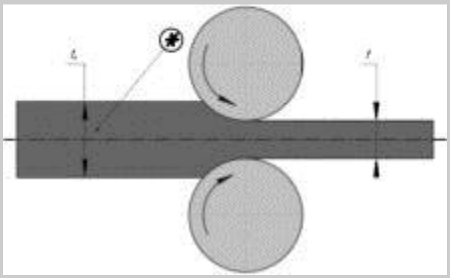
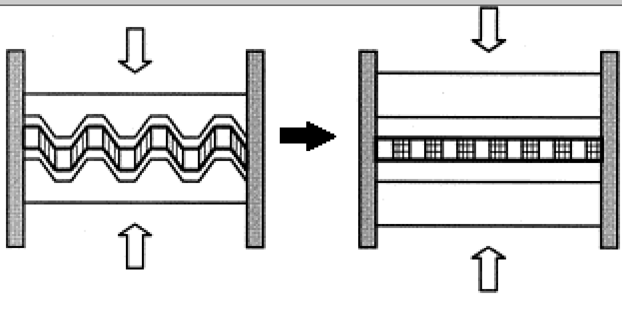
Keywords: Magnesium alloys, ECAP processing parameters, microstructures, mechanical properties.

1.0 INTRODUCTION

Equal channel angular pressing is one of the popular techniques of severe plastic deformation. Severe plastic deformation (SPD) is a viable technique that is commonly used for generating ultra-fine grained metal and nano-grained microstructure as to enhance the mechanical properties and superplastic behaviour of the metals [1-3]. According to Dimic et al., ultra-fine grained metal refers to the grain size produced by SPD technique which is in the range of 100-1000 nm while for nano-grained microstructure, it is less than 100 nm [4]. This method is said to be advantageous in strengthening metals and alloy. Enhancement of high strength is due to their small grains and high dislocation density characteristic [5]. There are various techniques of severe plastic deformation (SPD) that have been developed and applied in various applications. The technique includes equal channel angular pressing (ECAP) [6-8] with high pressure torsion (HPT) [9-10], accumulative roll bonding (ARB) [11-12], constrained groove pressing (CGP) [13-14], accumulative back extrusion (ABE) [15-16] and tubular channel angular pressing (TCAP) [17-18].

In ECAP process, an aluminium sample is pressed through a die with two channels intersect and shear strain is introduced during the process. The strain introduced could cause changes on its microstructure as well as the mechanical properties [19]. HPT technique is a combination of high pressure with torsional straining. The sample size of HPT process which is small and coin-shaped is the disadvantage of this process. The shear strain at the rotation axis is zero and increases in the radial direction if the geometry of the workpiece does not change. It causes the material properties of the sample placed near to the rotation axis to remain unchanged [19]. Another SPD technique is accumulative roll-bonding (ARB). The process of rolling, cutting, brushing and stacking may introduce a large strain value in the sheet. This technique can be applied in the production of metal-matrix composites [19]. In CGP process, the sample is pressed using a grooved dies and followed by a flat dies. At first pressing, the deformed region gives a strain value of 0.58 and after second pressing, the accumulative strain becomes 1.16. The process is continued by rotating the sample for 180° [20]. Table 1 shows the schematic diagram of SPD techniques mentioned above.

Table 1: Schematic illustration of severe plastic deformation technique [19, 20].

Process	Schematic illustration
Equal channel angular pressing (ECAP)	
High pressure torsion (HPT)	
Accumulative roll bonding (ARB)	
Constrain groove pressing (CGP)	

Magnesium was firstly identified by a British chemist, Sir Humphrey Davy in year 1808. In year 1833, Mg metal was produced by his assistant Michael Faraday by electrolysis of fused anhydrous $MgCl_2$ [21]. The first sample of Mg was presented in 1862 at World Exhibition London and was mostly being used in pyrotechnical application [21]. Nowadays, the application of magnesium has become popular and it is being used in many applications especially biomedical, nuclear, and automotive.

Since Mg has low ductility and excellent machinability behaviour, there is great potential application for its in mechanical products. Mg is broadly being used in the automotive industry as its high strength to weight ratio can be used for the reduction of vehicle weight as well as the fuel

International Journal of Engineering Research and Technology. ISSN 0974-3154, Volume 13, Number 11 (2020), pp. 3855-3871
© International Research Publication House. https://dx.doi.org/10.37624/IJERT/13.11.2020.3855-3871

consumption [22- 23]. However, the HCP structure in magnesium has a drawback of limited ductility at ambient temperature.

In recent years, review studies on ECAP process have been reported in the literature. A review on equal channel angular extrusion as a deformation and grain refinement process has been done by Adedokun in 2011 [24]. This article highlighted the review of ECAP process including the advantages and limitations, the parameters and also the material tested. In 2013, Estrin and partner [19] wrote a review on the challenging parts of SPD and some highlights to the potentials and limitations of SPD technologies. Two years later, Kawasaki et al. [25] discussed on achieving superplastic properties in ultrafine-grained (UFG) materials at high temperature which study on the occurrence of superplastic flow in a series of UFG aluminium and magnesium alloys after ECAP and HPT. In 2015, a review on ECAP processing was done by Roodposhti et al. [26] on the effect of ECAP process parameters including pressing route, temperature, pressing speed, channel and curvature angle and number of passes through the die to the commercially pure titanium (CP-Ti) alloys.

Although some reviews have been done on ECAP, there has been little reporting on the effects of ECAP parameters on certain materials. Therefore, this review paper gives a brief description of the effects of ECAP processing parameters on the microstructure and mechanical properties of magnesium alloy. The ECAP parameters discussed include die and curvature angle, number of passes, processing route and temperatures. The explanation on the effects of ECAP parameters on microstructure and mechanical behaviour of various magnesium alloy are also discussed.

2.0 ECAP GENERAL PROCESSING

Since earlier, ECAP has been known as a process that focusses on producing an ultra-fine grained microstructure [27-29]. This process was first introduced in 1972 by Dr. Segal and his co-workers [30] at the Physical Technical Institute Academy of Science of Buelorussia, Minsk. Initially, the aim was to focus on development and application of equal channel angular extrusion (ECAE) technology to different materials science and industrial problems. After about 20 years later, the technique of producing an ultrafine- grain and nano- grain materials was discovered through ECAE process [30-31].

In 2005, Mishra and co. worker did a research on microstructure evolution in copper processed by severe plastic deformation [32]. They found that the grain refinement was the most effective at the first few passes and became extra equiaxed as the number of passes increased. They also discovered that the process of obtaining ultra-fine grain microstructure involved five stages as illustrated in Fig. 1.

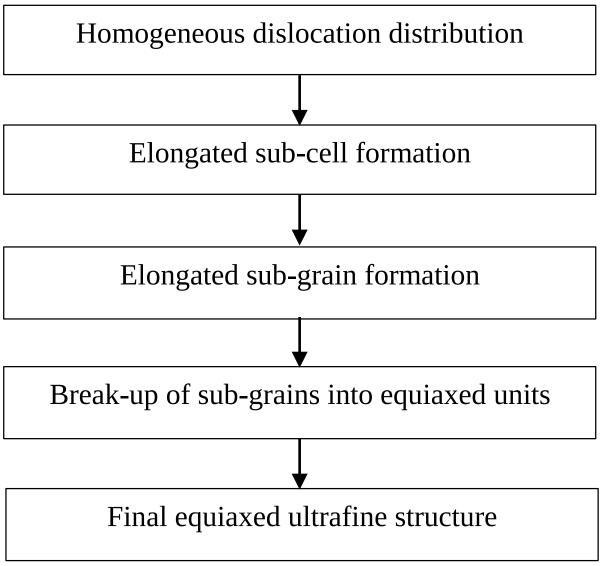


Fig. 1: Stages of grain refinement.

Previously, there have been many approaches on metals and its alloy that have undergone ECAP process effectively, including aluminium and its alloy [33-35], magnesium alloy [36-37], copper and its alloys [38-40], and steel [41-42]. Most of the research focuses on the effect of ECAP processing especially on the microstructures and mechanical properties. Table 2 shows several studies that have been successfully done by numerous researchers on the characteristic and performance of materials after undergoing ECAP process.

Table 2: List of research done on ECAP process.

Authors	Title	Objectives
Y. G. Kim, Y. G. Ko, D. H. Shin, S. Lee [33]	Effect of channel angular pressing routes on high strain rate deformation behaviour of ultra-fine grained aluminium alloy	• To investigate the fracture behaviour and HSR deformation of ultra-fine grained aluminium alloy after 1P and 8P ECAP at different routes. • To examine the factors that affect tensile and torsional properties and the potential for adiabatic shear band formation.
J. Suh, J. Victoria-Hernandez, D. Letzig, R. Golle, S. Yi, J. Bohlen, W. Volk. [37]	Improvement in cold formability of AZ31 magnesium alloy sheets processed by equal channel angular pressing	• To investigate the microstructure development and mechanical properties at room temperature. • To correlate the results with the cold forming behaviour.

F. D. Torre, R. Lapovok, J. Sandlin, P. F. Thomson, C. H. J. Davies, E. V. Pereloma [38]	Microstructures and properties of copper processed by equal channel angular extrusion for 1-16 passes.	• To investigate the microstructure evolution from 0 to 16 passes of ECAE by using TEM and XRD analysis. • To study the mechanical properties of the material after the process.
M. Eddahbi, M. A. Monge, T. Leguey, P. Fernandez, R. Pareja [42]	Texture and mechanical properties of EUROFER 97 steel processed by ECAP	• To study the capability of warm ECAP for developing a stable grain structure and texture in tempered EUROFER 97 that can enhance its mechanical behaviour in its operational temperature range.

Fig. 2 shows the general principle schematic diagram of ECAP process. The process is performed by using a die equipped with bent channel that goes through a sharp angle near the centre of the die. Channel angle, φ and curvature angle, Ψ are the angles between the two channels which are joined together. The first sample is machined to determine the diameter and length to fit the channel diameter. A plunger is used to push the billet through the die with high pressure, P. Since the cross-sectional dimensions are fixed, repeating process is carried out to get a very high strains. The pushing process is repeated for several times to get the desired microstructure [30, 43].

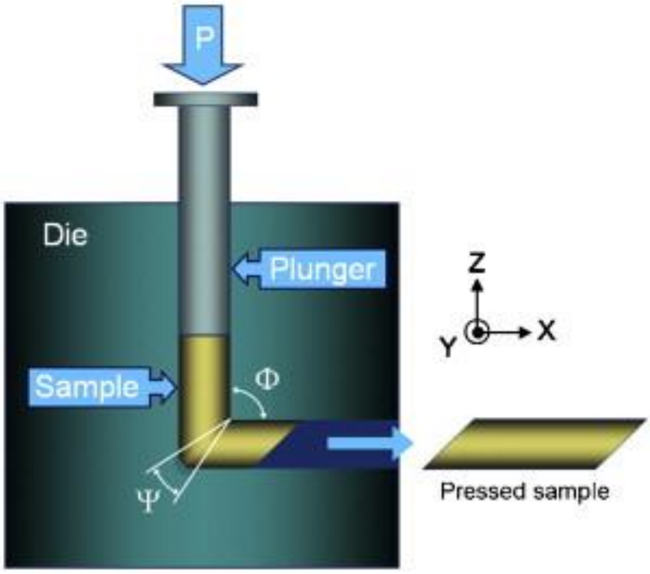


Fig. 2: General principle of ECAP process [31].

The parameters of ECAP process play an important role in changing the microstructure, mechanical properties, corrosion resistance and also other properties. The effects of ECAP parameters to the microstructure of the materials and its properties have been studied by several researchers [44-49]. The parameters that have been considered for investigation are temperature, number of passes, processing route, channel angle and curvature angle. These experimental parameters are controlled as to obtain the desired microstructure and mechanical properties.

2.1 Channel angle and curvature angle

In ECAP process, a billet is pressed through a die by a plunger that consists of two channels with equal cross sections, intersecting at certain angle [50]. The angle that exists between the two channels plays an important role in defining the strain. Typically, the channel angle used is between 90°-120° [51]. However, some literature reported that the angle can be as small as 60° and a maximum of up to 135° [52]. It was reported that the channel angle, ϕ and curvature angle, Ψ affected to the strain present in each passes of ECAP. Iwahashi et al. [53] and Vincentis et al. [54], proposed the strain accumulated, ε_N during the process which can be denoted as:

$$\varepsilon_N = (N/\sqrt{3})\ (2\cot(\Phi/2 + \Psi/2) + \Psi\ \mathrm{cosec}(\Phi/2 + \Psi/2)) \qquad (1)$$

Where Φ and ψ are the corner and the die angles, respectively while N is the number of passes. This model resolves that a higher value of channel angle will contribute to a higher strain value. In Vincentis work, the equivalent strain per pass obtained was 0.67 when 120° of die angle was used in ECAP process [54].

2.2 Temperature of operation

Applying a high operation temperature of ECAP process will increase the rate of recovery and also lead to formation of low angle grain boundaries [55, 56]. Increasing the temperature may increase the size of grain and reduce the dislocation density thus giving lower strength. However, by applying high temperature, the pressing process might be easier compared to lower temperature. On the other hand, a lower operation temperature contributes to the smaller grain size which leads to formation of high angle grain boundaries which have excellent mechanical properties. This is proved by the study done by Xirong and his colleague [57] in 2009 on multi-pass ECAP processing of commercial pure titanium at room temperature. Based on their analysis, the microstructure of pure titanium had refined from 28 µm to 250 nm after doing four ECAP passes.

2.3 Number of passes

The material properties whether mechnical properties or microstructure characterization after ECAP process are influenced by the number of strain. The amount of strain can be increased by increasing the number of passes as the original cross section is maintained. This is one of the advantages of ECAP processing.The number of passes in ECAP parameters is the number of the sample being pressed into the die either using route A, B_A, B_c or C (these are explained in the next section). The ductility and strength increase after some passes due to the increase of high angle grain boudaries fraction and the improvement on the homogeneity of microstructure [58]. Among these ECAP parameters, ECAP passes play the most important role in enhancing the mechanical properties of a material. This parameter is easy to control and the final results are more favourable compared to others.

2.4 Processing route

The microstructure characteristic of alloys can be influenced by the processing route in ECAP process. There are four fundamental routes in ECAP process namely route A, B_A, B_C and C. The differences between these four routes are in terms of how the materials are being rotated between the passes. In route A, the sample is pressed without any rotation, while in route B_A, the sample is rotated by 90° in an alternate direction between passes. On the other hand, in route B_C, the sample is rotated by 90° in the same direction between passes and route C is performed by rotating the sample by 180° between each pass [50, 51, 59]. Fig. 3 shows an illustration of four fundamental processing routes [51]. Among these four different routes, route B_C is the most successful route to reach a homogeneous microstructure for many alloys [60-62].

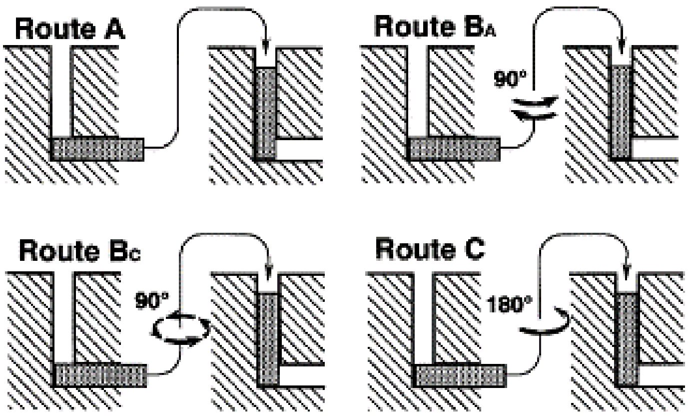


Fig. 3: Four fundamental processing routes in ECAP process [51]

Lin et al. [63] investigated on the microstructure evolution and mechanical properties of a Ti-35Nb-3Zr-2Ta biomedical alloy processed by equal channel angular pressing (ECAP). In this research, route B_C was used where the sample was rotated by 90° clockwise for each pass. It was evident that this route was able to produce a maximum grain refinement and more equiaxed grains in the alloy. In a research undertaken by Komura and his co-workers [64], route B_C was selected in their experiment. It was also notable that this route can produce equiaxed ultrafine grains in magnesium alloys.

3.0 Effect of ECAP parameters

As mentioned previously, the ECAP parameters play an important role in defining the microstructure behaviour and mechanical properties. The parameters include number of passes, temperature, angles and processing route. Table 3 shows the summary of the information gathered based on the different parameters applied during the ECAP process. It is noted that 1P, 2P, 4P refers to 1 number of pass, 2 passes and 4 passes respectively.

In year 2013, Avvari et al. [65] performed a research on the effect of ECAP on AZ31 wrought magnesium alloys. The study aimed to evaluate the grain size of wrought AZ31 alloy processed by ECAP using a die with an angle of 120° for its channel angle and 30° for curvature angle. In 2014, Jahadi et al. [66] did a research to evaluate the effect of ECAP process on the as-extruded, AM30 magnesium alloy and compared it with another magnesium alloy which was AZ31B since it exhibited similar properties that had been reported before. Yamashita et al. [67] had done a research on improving the mechanical properties of magnesium and magnesium alloy through severe plastic deformation. The research objectives were to test the potential of pure Mg and dilute Mg-Al in grain refinement and to investigate the mechanical behaviour of these materials at room temperature. A research by Minarik and his co-workers in 2013 [68] focused on the evolution of microstructure, mechanical properties and corrosion resistance of AE21 alloy after ECAP. The research highlighted corrosion behaviour besides microstructure and mechanical behaviour. In recent years, a study on the evolution of mechanical properties of LAE442 magnesium alloy processed by extrusion and ECAP was done by Minarik et al. [69] focussing biodegradable magnesium implant. LAE442 also having biodegradable and biocompatible properties that are needed for implant material. A research done by Dumitru and his co-worker [70] investigated ECAP process on material ZK60. The material was chosen because of its high strength but lower in plasticity properties. In a paper reported by Poggiali et al. [71], commercially pure magnesium (CP-Mg) was processed by ECAP from two different conditions which were as-cast condition and rolling condition. It was then followed by ECAP process at different routes and number of passes. The paper aimed to introduce a new preliminary step rolling before ECAP process to obtain better mechanical properties and smaller grain size of the material after ECAP compared to a material processed by ECAP only. In 2014, a research done by Avvari et al. [72] studied the influence of ECAP processing route on the microstructure and mechanical properties of AZ31 after 2 passes at temperature 498K. All these research findings are summarized in Table 3.

Table 3: Summarization of research findings

Authors	Material & Methods (ECAP parameters)	Findings
M. Avvari, S. Narendranath, H. S. Nayaka [65]	<table><tr><td>Material</td><td>AZ31 wrought Mg alloy</td></tr><tr><td>Route</td><td>B_c</td></tr><tr><td>No. of passes</td><td>4</td></tr><tr><td>Temperature</td><td>300°C</td></tr><tr><td>Die angle</td><td>120°</td></tr><tr><td>Curvature angle</td><td>30°</td></tr></table>	• Average grain size was reduced from 31.8μm as received to 8.0μm after ECAP process at 4P. • 1P- A few coarse grains and elongated grains were spotted • 2P- observed that the grain has been deformed and distributed heterogeneously • 3P- The average grain size was 9.4μm and the distribution of grains was more consistent. • 4P- The grain distributed more evenly as the dynamic recrystallization happened • As-received alloy had a value of microhardness about 51HV and it increased up to 2P but then it decreased after 3P and 4P.
R. Jahadi, M. Sedighi, H. Jahed [66]	<table><tr><td>Material</td><td>AM30 Mg alloy</td></tr><tr><td>Route</td><td>B_c</td></tr><tr><td>No. of passes</td><td>4</td></tr><tr><td>Temperature</td><td>275°C</td></tr><tr><td>Die angle</td><td>90°</td></tr><tr><td>Curvature angle</td><td>20°</td></tr></table>	• The as-extruded sample showed a large grain size and after ECAP process at 1P, the size decreased drastically from 20.4 μm to 7.2 μm. • After 1P, the grain size reduced gradually as the number of passes increased. • For mechanical properties, the ultimate tensile strength were about 15 MPa in 1P and around 30MPa for 2P and 3P. However it was reported that after 4P the ultimate tensile strength remained unchanged. • Meanwhile, the ductility was increased with the increase of passes. • The hardness was increased with the number of passes but it started decreasing at 2P
Akihiro Yamashita, Zenji Horita, Terence G. Langdon [67]	<table><tr><td>Material</td><td>Pure Mg and Mg-0.9wt% Al alloy</td></tr><tr><td>Route</td><td>B_c</td></tr><tr><td>No. of passes</td><td>2, 4</td></tr><tr><td>Temperature</td><td>200, 300, 400°C</td></tr><tr><td>Die angle</td><td>90°</td></tr><tr><td>Curvature angle</td><td>45°</td></tr></table>	• Grain size of Mg-0.9Al after 2P at different temperatures: i) 200°C - 17μm ii) 300°C - 48μm iii) 400°C - 78μm • The tensile strength of the material after ECAP was significantly higher than as- received. • The elongation before fracture for pure Mg was lower compared to the Mg alloy with dilute Al.
P. Minarik, R. Kral, M. Janecek, J. Pesicka [68]	<table><tr><td>Material</td><td>AE21 Mg alloy</td></tr><tr><td>Route</td><td>B_c</td></tr><tr><td>No. of passes</td><td>8</td></tr><tr><td>Temperature</td><td>180°C</td></tr><tr><td>Die angle</td><td>90°</td></tr><tr><td>Curvature angle</td><td>0°</td></tr></table>	The average grain size shows that the size was decreased to 1~2 μm from 4~5 μm in extruded sample. • For mechanical properties, the yield stress increased up to 2 passes then it started to decrease. • While the maximum stress showed a decline value of between 4P and 8P.

<table>
<tr>
<td>P. Minarik, R. Kral, J. Pesicka, F. Chmelik
[69]</td>
<td>
<table>
<tr><td>Material</td><td>LAE442 Mg alloy</td></tr>
<tr><td>Route</td><td>B_c</td></tr>
<tr><td>No. of passes</td><td>12</td></tr>
<tr><td>Temperature</td><td>180 – 230°C</td></tr>
<tr><td>Die angle</td><td>90°</td></tr>
<tr><td>Curvature angle</td><td>0°</td></tr>
</table>
</td>
<td>

For the first pass, the average grain size decreased drastically from 21µm to 11µm and after 12 passes, the grain size became 1.5µm.
The tensile strength and ultimate tensile strength increased progressively up to 4 passes and after that the strengths remained constant.
It shows that the elongation to fracture for the first pass had increase severely and increase slightly at 12 passes.

</td>
</tr>
<tr>
<td>F. D. Dumitru, O. F. Higuera- Cobos, J. M Cabrera
[70]</td>
<td>
<table>
<tr><td>Material</td><td>ZK60 Mg alloy</td></tr>
<tr><td>Route</td><td>B_C</td></tr>
<tr><td>No. of passes</td><td>4</td></tr>
<tr><td>Temperature</td><td>250°C</td></tr>
<tr><td>Die angle</td><td>90°</td></tr>
<tr><td>Curvature angle</td><td>37°</td></tr>
</table>
</td>
<td>

The grains became more equiaxed after 3P due to the existence of recrystallization either during ECAP process or the interpass time.
In 4P, the grain size slightly increased due to recrystallization and rapid grain growth.
UTS value increased at first 2P from 285MPa for as-received to 326MPa.
UTS started to decrease after 3P.
The elongation to fracture increased from 15HV to 30HV after 4P.

</td>
</tr>
<tr>
<td>F. S. J. Poggiali, C. L. P. Silva, P. H. R. Pereira, R. B. Figueiredo, P. R. Cetlin
[71]</td>
<td>
As- receive + ECAP:
<table>
<tr><td>Material</td><td>Commercially pure Mg alloy (CP-Mg)</td></tr>
<tr><td>Route</td><td>B_c</td></tr>
<tr><td>No. of passes</td><td>3</td></tr>
<tr><td>Temperature</td><td>200°C</td></tr>
<tr><td>Die angle</td><td>135°</td></tr>
<tr><td>Curvature angle</td><td>20°</td></tr>
</table>
Rolling + ECAP:
<table>
<tr><td>Material</td><td>Commercially pure Mg alloy (CP-Mg)</td></tr>
<tr><td>Route</td><td>C</td></tr>
<tr><td>No. of passes</td><td>4</td></tr>
<tr><td>Temperature</td><td>200°C</td></tr>
<tr><td>Die angle</td><td>135°</td></tr>
<tr><td>Curvature angle</td><td>20°</td></tr>
</table>
</td>
<td>

For the first experimental procedure;
Material processed by ECAP exhibited higher flow stress compared to the as-received magnesium.
It gave higher yield strength and maximum stress.
The grain size decreased from 300µm to 5µm.
In the second experimental procedure;
Material processed by rolling exhibited higher yield strength and lower elongation compared to the material processed by rolling with ECAP.
Material processed by ECAP exhibited higher hardening rate which led to higher uniform elongation than processed by rolling.
The grain structure was refined after rolling for about 10µm and completely refined to 5µm after ECAP

</td>
</tr>
<tr>
<td>M. Avvari, S. Narendranath, H. S. Nayaka
[72]</td>
<td>
<table>
<tr><td>Material</td><td>AZ31 Mg alloy</td></tr>
<tr><td>Route</td><td>A, B_c, C</td></tr>
<tr><td>No. of passes</td><td>2</td></tr>
<tr><td>Temperature</td><td>225°C</td></tr>
<tr><td>Die angle</td><td>120°</td></tr>
<tr><td>Curvature angle</td><td>30°</td></tr>
</table>
</td>
<td>
<table>
<tr><td>Route</td><td>Ave. Grain size (µm)</td><td>Micro hardness (HV)</td><td>Tensile strength (MPa)</td><td>Elongation (%)</td></tr>
<tr><td>As-R</td><td>31.8</td><td>51</td><td>225</td><td>15.2</td></tr>
<tr><td>A</td><td>14.0</td><td>60</td><td>302</td><td>31.4</td></tr>
<tr><td>B_c</td><td>13.3</td><td>64</td><td>294</td><td>33.5</td></tr>
<tr><td>C</td><td>15.6</td><td>54</td><td>274</td><td>23.63</td></tr>
</table>
</td>
</tr>
</table>

3.1 Effect of ECAP processing parameters on the microstructure

3.1.1 Channel angle and curvature angle

It has been observed that the angle could affect the grain size. It has been reported in the literatures that most of the experimental works use an angle between 90° to 120° in their research. There are limited work on comparing the properties of material using different channel angles. The larger angle can make the sample easier to be pressed compared to the smaller angle. Fig. 4 shows the ECAP setup for an experiment done by Murlidhar et al. [65]. The study shows that the smaller the angle, leads to an increase in the amount of strain and results in smaller grain size. In contrast, higher die and curvature angle may reduce the dead zone of the die as shown by Muralidhar et al.'s study [65]. Compared to an experiment that uses die angles of 90° and 120°, the grain size obtained in higher angle is not as fine as the grain size obtained by using the lower angle. The higher angle has been recorded to produce a grain size of 13.3 µm after a single pass then decreased to 8 µm after 4 passes [65]. Whereas, lower channel angle can give smaller grain as shown by a research done by Jahadi et al. [66] where the grain size was recorded at 7.2 µm after only 1 pass and can reach to the smaller size of 3.9 µm after 4 ECAP passes. Figs. 5 and 6 show the microstructure of AZ31 after using 120° die and AM30 after using 90° as its channel angle during ECAP process respectively. It clearly illustrates that the microstructure of the material using smaller angle in ECAP process exhibits smaller grain size. These Figs. are also used to compare the material behavior after 4 ECAP passes at different angles. The comparison are based on different materials of AM30 and AZ31. This is due to lack of information on material AM30 while AZ31 is reported to have similar properties with AM30 [66].

Fig. 4: Equal channel angular pressing setup for die angle and curvature angle of 120° and 30° [65].

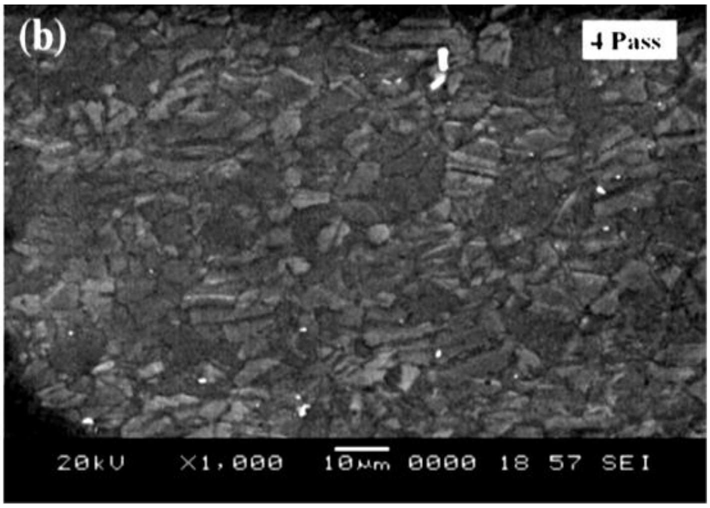


Fig. 5: SEM image of AZ31 using 120° channel angle after 4 ECAP passes [65]

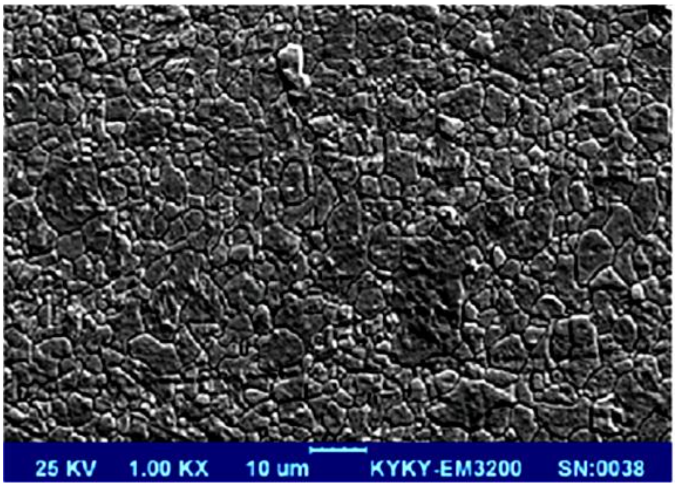


Fig. 6: SEM images of AM30 using 90° channel angle after 4 ECAP passes [66].

3.1.2 Operation Temperature

The operating temperature is also one of the influential parameters that can alter the properties of the material. However, it has been reported to give less effect on the grain refinement as compared to increasing number of passes. A research done by Yamashita et al.[67] in 2001 proved that the temperature of ECAP process can change the material characteristic. It was recorded that the grain size of the alloy incereases as the applied temperature increases. A graph of grain size versus number of passes at different temperatures is shown in Fig. 7. It shows that the presence of Al dilution helped to reduce the grain size of the material. Fig. 8 shows the microscopic image of microstructure of Mg-0.9%Al processed at different ECAP temperatures after 2 passes. This proves that smaller and finer grain size can be obtained at lower operating temperature. This finding is supported by previous works where the Mg alloy processed by ECAP at low temperature of 100°C produced ultra-fine grain structure [73, 74].

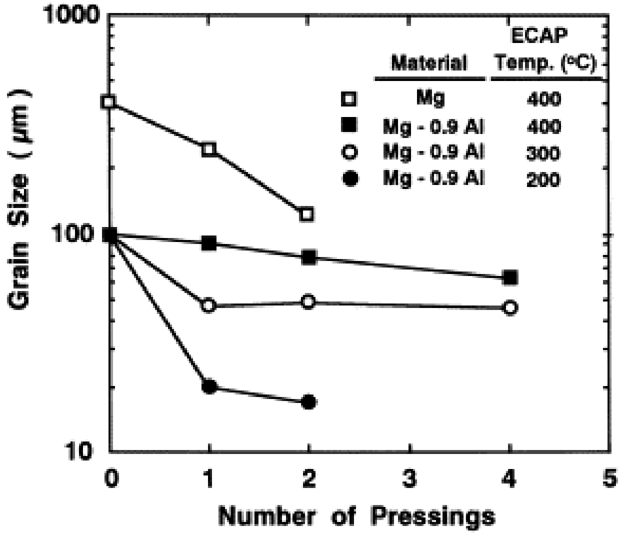


Fig. 7: Grain size versus number of passes at each different temperatures [67].

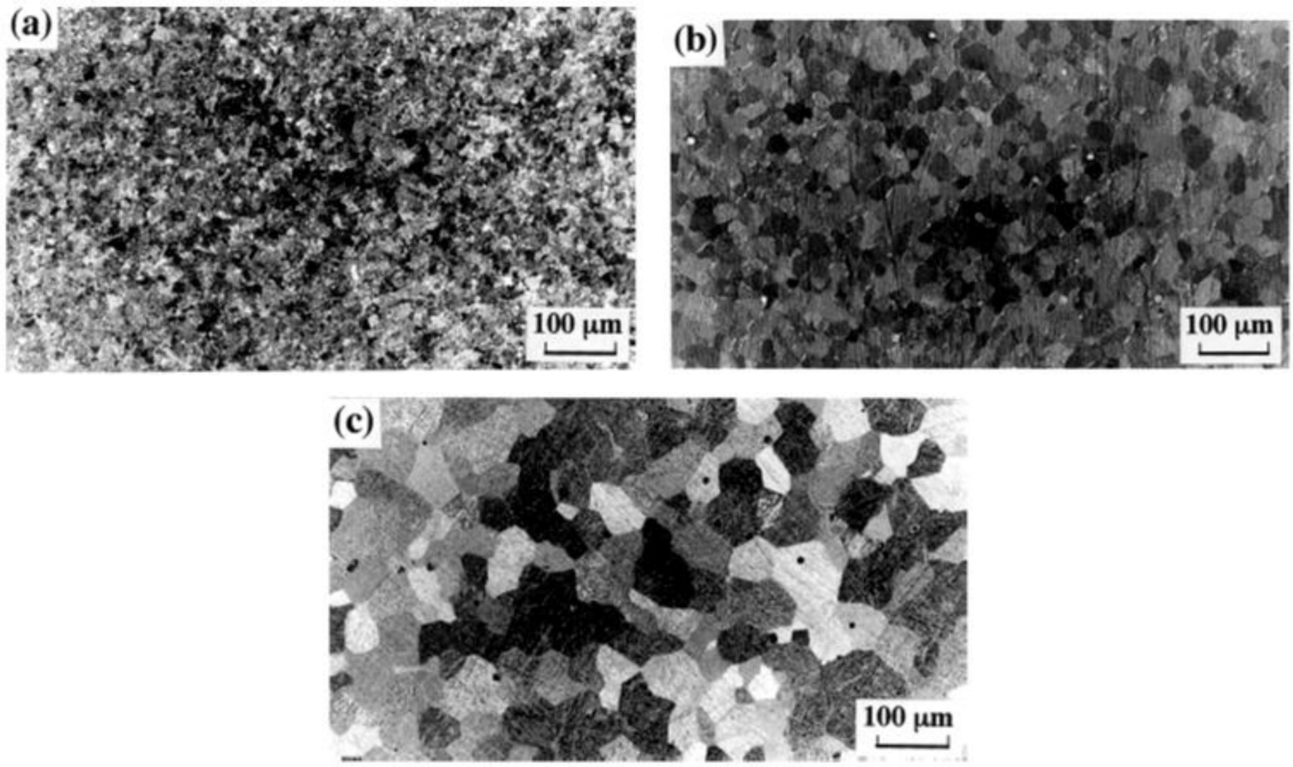


Fig. 8: Microstructure of Mg- 0.9% Al at different temperatures (a) 200°C, (b) 300°C and (c) 400°C after two passes [67]

3.1.3 Number of Passes

Refering to Table 2, it is resolved that all the parameters absolutely affect the material properties. In 2013, Minarik et al. Conducted a study on the evolution of the microstructure, mechanical properies and corrosion resistance of AE21 magnesium alloy after ECAP. Two years after that he replicated the research using different material which was LAE442 magnesium alloy [68, 69].

It was observed that as the number of passes increased, the grain size was reduced and the distribution was much better for both research. Fig. 9 shows a micrograph of TEM of AE21 alloy for each pass. It shows that in the first and second passes, the microstructure are inhomogeneous. But in the second pass, the sub grain boundary was revealed. At the end of 8 passes, high angle grain boundaries (HAGBs) were formed and the size was about 1 – 2 μm.

International Journal of Engineering Research and Technology. ISSN 0974-3154, Volume 13, Number 11 (2020), pp. 3855-3871
© International Research Publication House. https://dx.doi.org/10.37624/IJERT/13.11.2020.3855-3871

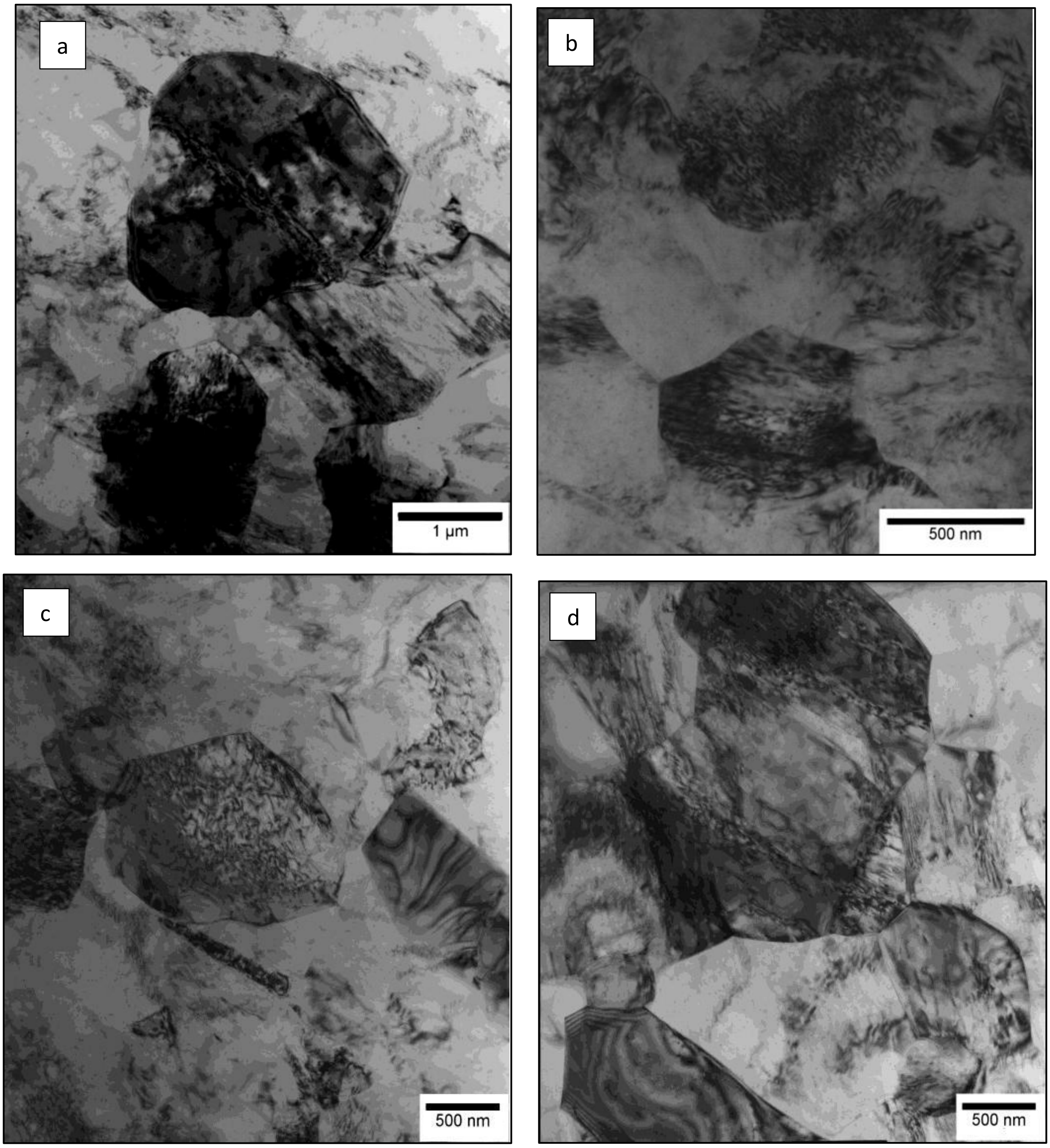


Fig. 9: TEM micrograph of AE21 at different passes where (a) 1 pass, (b) 2 passes, (c) 4 passes and (d) 8 passes [68].

Dumitru et al. [70] stated that there are two steps of refinement process. The first one is formation of low angle grain boundaries (LAGBs) through first and second passes and the second one is recrystallization process which appears at the boundaries. Another researcher wrote on comparison of as-received material combined with ECAP and rolling process combined with ECAP process [71]. The output shows that the second experimental procedure which is rolling combined with ECAP gave better material properties in term of their grain structure and mechanical properties.

3.1.4 Processing Route

Avvari and his co- worker [72] investigated on the effect of processing routes on AZ31 alloy by severe plastic deformation. In this research, 3 different routes have been selected to access its efficiency. It was found that the average grain size of the material after processing was decreased to 15.9 µm for route C, 14.0 µm for route A and the smallest grain obtained by route BC with a size of 13.3 µm. As observed in route B_C, the alloy exhibits a maximum deformation due to the grain refinement compared to route A and C. Fig. 10 shows the microstructure of route A, C and B_C after 2 passes at a temperature of 498 K.

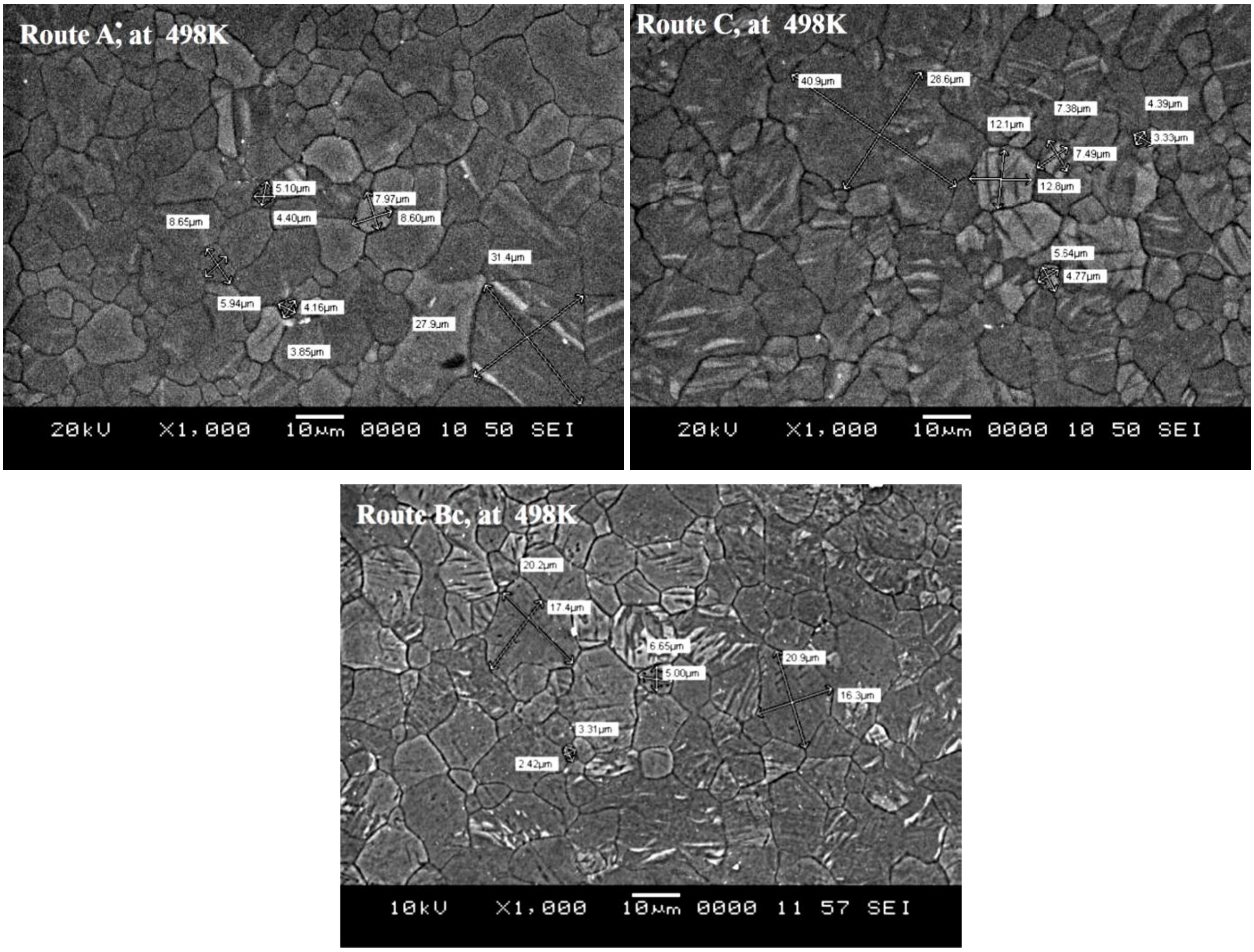


Fig. 10: Microstructure of AZ31 using (a) route A, (b) route C and (c) route Bc after 2 passes at 498K [72].

Among these three different routes applied, the most conventional reported to achieve a homogeneous microstructure for most alloys is route B_C [75]. According to Komura et al. [76] the optimum super plastic ductility is achieved by route B_C due to the most rapid generation of equiaxed grains with the high angle of grain boundaries. Stolyarov et al. [77] indicated that route B_C is the most effective while route B_A is the least efficient in terms of grain refinement. Route B_A and C tend to form elongated grains. Tong et al.[78] conducted a review based on the microstructure and mechanical properties of Mg alloy with the ECAP route process where the result for route B_C is the most capable in grain refinement while route A is the least capable. Kim and Namkung et al. [79] stated that routes A and B_A have the lowest strain distribution homogeneity while routes C and B_C face the highest strain spread uniformly.

3.2 Effect of ECAP processing parameters on the mechanical properties

The mechanical behaviour of material after ECAP process are obtained by performing tensile test and microharness test. Tensile strength is conducted according to certain standard depending on the condition of the samples. For example, in a research done by Avvari et al. [72], the samples were rod-shaped, hence the tensile test were done by following standard ASTM E-8 which the gauged length and diameter of the specimen to be 15 mm and 5 mm respectively.

3.2.1 Channel angle and curvature angle

In the research done by Avvari et al. [65] and Jahadi et al. [66], the microhardness properties showed the same trend where it was increased up to 2 passes before decreasing for the next passes. The highest value of vicker's microhardness that can be reached by AZ31 using 120° of ECAP die was 69 HV after 2 passes. As for ECAP die with chnnel angle of 90°, the highest value was 64.4 HV also after 2 passes. The reduction of material hardness was related to the ductility and the tensile strength of the material. Tensile strength of the material after being processed by using 90° ECAP die was basically slightly higher than by using the larger angle [82-84]. But it showed similar decreasing trend after several passes. The tensile strength was decreased due to the changing of the material texture [85-87]. Increasing the number of passes lead to an increase in the strain hardening thus the elongation percentage observed was increased [65, 66].

3.2.2 Operation Temperature

Lower operation temperature could produce finer grain size hence increasing the hardness properties. On the other hand, lowering the processing temperature while increasing the number of passes increase the alloy hardness. The ultimate tensile strength of the Mg- 0.9%Al was increased by decreasing the operation temperature. The results obtained in a research done by Yamashita et al. [67], at 200°C, showed that the tensile strength went up to 225MPa while at a higher temperature, the strength obtained was 200MPa. The elongation before fraction also increased at low temperature. Compared to pure Mg, the Mg with dilute Al exhibits better properties in term of their mechanical properties and microstructure. It is also mentioned that the existance of dilute Al leads to higher ductility

It was noticed that Mg – 0.9% Al tested at temperature 200°C can withstand up to 2 passes only. More than 2 passes may led to occurence of cracking. The fact that the processing temperature must be above 200°C to avoid cracking can be found as reported by Agnew et al. [80]. A research done by Kang et al [81] on AZ31 also testified that magnesium alloy will exhibit fracture characteristic when the temperature is below 200°C.

3.2.3 Number of Passes

The tensile strength and ultimate tensile strength observed from both studies [80-81] show the same patern where it is increased up to 4 passes before remaining constant for the next passes. In a research done by Minarik and his co-worker [69] on evolution of mechanical properties LAE442 magnesium alloy processed by extrusion and ECAP, the increase of LAGBs fraction at first pass led to lower segregation of calcium in LAE442 along the grain boundaries thus giving higher elongation. The small amount of calcium in LAE442 caused intergranular fracture in magnesium alloys thus decreasing the elongation to fracture due to the separation of calcium [69]. For the following passes, the LAGBs fraction decreased and resulted in an increase in the grain boundary fraction. Then the calcium may be dispersed evenly in the material so that the ductility was almost unchanged [69].

The ultimate tensile strength of AE21 and LAE442 are displayed in a graph in Fig. 11. The graph shows the same trend where the strength increased as the number of passes increased. For AE21 magnesium alloy, the tensile strength were increased up to 4 passes then decreased while for material LAE442, it increased up to 4 passes before it saturated [68,69]. The changing of tensile strength depended on the texture evolution and density dislocation [88].

3.2.4 Processing Route

According to Avvari et al. [72], route B_C exhibited the highest microhardness value and also in percentage of elongation compared to others. Fig. 12 shows the graph of microhardness and elongation versus type of route used. The microhardness increased from 51HV up to 60HV, 64HV and 54HV for as-received material, route A, Bc and C respectively. Route Bc improved by about 25.5% on its material hardness followed by route A with 17.65% while route C sample contributed the least increment of hardness percentage relatively to the as-received condition. The hardness of material increased when the grain size decreased. The tensile strength of the materials also increased as it undergo ECAP process. On the contrary, the tensile strength of the material after the process using ECAP route A exhibit highest value of 302MPa followed by route B_C with 294MPa compared to the as-received sample value of 225MPa. On the other hand, the elongation to fraction of route B_C sample shows highest value of 33.5% followed by route A with percentage of 31.4%. It can be concluded that route B_C is more suited to deform the material in all plane since the main advantage of ECAP is to increase the elongation percentage with increased strength [72].

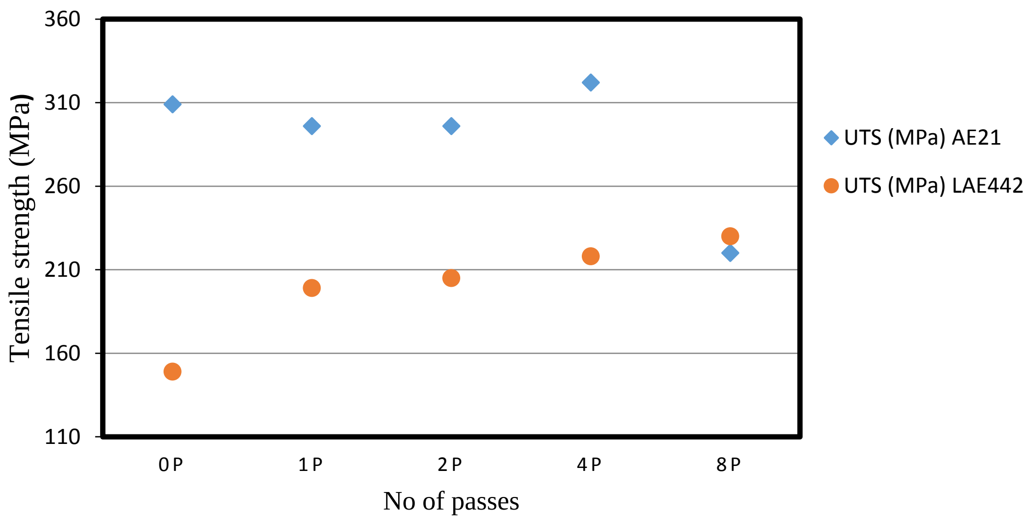


Fig. 11: Tensile strength of LAE442 and AE21 alloys at different number of passes [68, 69].

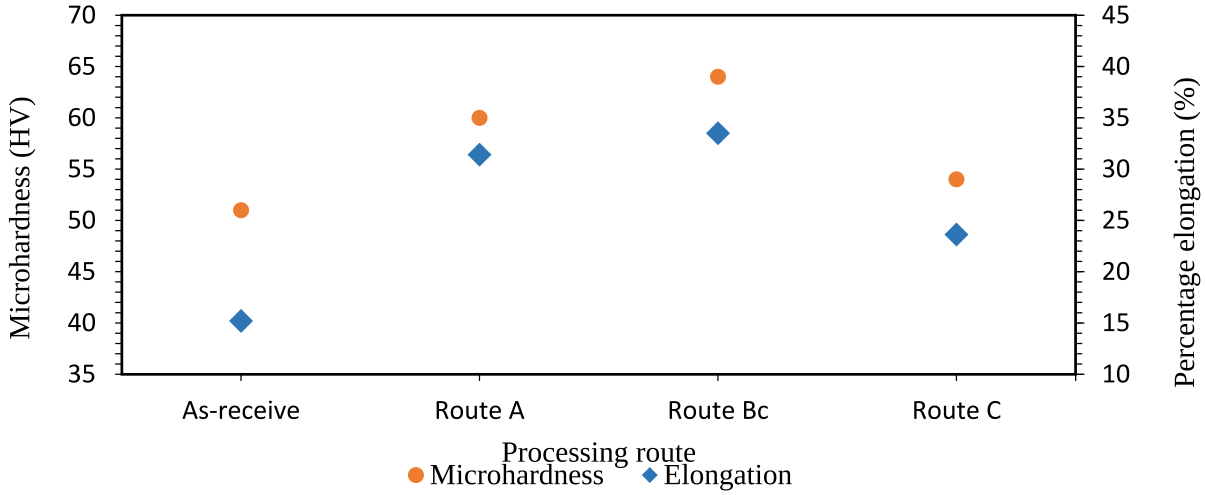


Fig. 12: Graph of microhardness and elongation to fracture of AZ31 alloy at different processing route [72].

4.0 CONCLUSION

ECAP is one of the important techniques to produce an ultra-fine grain microstructure. This process could be used to improve the mechanical properties of magnesium alloys. The parameters of ECAP have an influence on the material behaviour after the process. It can be concluded that:

i. The grain size of magnesium alloys becomes smaller as the number of ECAP passes increased up to 4 passes.

ii. The increase in temperature increases the grain size due to increasing time of recovery. The production of low angle grain boundaries is greater than high angle grain boundaries.

iii. Route B_C gives the best final material properties as it can produce the finest grain size compared to other routes.

iv. Channel angle of ECAP die has a significant effect on the microstructure of the Mg alloys. Larger angle die with lower processing temperature and higher number of ECAP passes (i.e 4 passes), produced a smaller grain size and better mechanical properties for the Mg alloys.

v. The number of ECAP passes plays an important role in improving the strength of material. ECAP processing route has been ranked as second place on its efficiency to improve the material strength and while operation temperature plays the least role in improving the material properties.

ACKNOWLEDGEMENT

Authors would like to thanks the Universiti Teknikal Malaysia Melaka (UTeM) and Ministry of Education (MoE), Malaysia for financial support of this research under grant FRGS/2018/FKP-AMC/F00379.

REFERENCES

[1] F. Djavanroodi, B. Omranpour, M. Ebrahimi, and M. Sedighi. 2012. Designing of ECAP parameters based on strain distribution uniformity. *Progress in Natural Science: Materials International*. 22(5): 452-460.

[2] A. Azushima, R. Kopp, A. Korhonen, D.Y. Yang, F. Micari, G.D. Lahoti, P.Groche, J. Yanagimoto, N. Tsuji, A. Rosochowski, and A. Yanagida. 2008. Severe Plastic Deformation (SPD) Processes for Metals. *CIRP Annals-Manufacturing Technology*. 57: 716-736.

[3] Z.J. Zheng, Y.Gao, J.W.Liu, M.Zhu. 2015. A hybrid refining mechanism of microstructure of 304 stainless steel subjected to ECAP at 500°C. *Materials Science and Engineering A*. 639: 615-625.

[4] I. Dimic, I. Chijovic-Alagic, B. Volker, A. Hohenwarter, R. Pippan, D. Veljovic, M. Rakin, B. Bugarski. 2016. Microstructure and metallic ion release of pure titanium and Ti–13Nb–13Zr alloy processed by high pressure torsion. *Materials & Design*. 91: 340-347.

[5] Y. J. Chen, Y. C. Chai, H. J. Roven, S. S. Gireesh, Y. D. Yu, and J. Hjelen. 2012. Microstructure and mechanical properties of Al- xMg alloys processed by room temperature ECAP. *Materials Science and Engineering A*. 545: 139-147.

[6] M. S. Shadabroo, A. R. Eivani, H. R. Jafarian,S. F. Razavi, J. Zhou. 2016. Optomization of interpass annealing for a minimum recrystallized grain size and further grain refinement towards nanostructured AA6063 during equal channel angular pressing. *Materials Characterization*. 112: 160-168.

[7] V. L. Sordi, A. A. Mendes FilhoSordi, G.T. Valio, P. Springer, J. B. Rubert, M. Ferrante. Equal-channel angular pressing: Influence of die design on pressure force, strain homogeneity, and corner gap formation. *Journal of Materials Science*. 2016. 51(5): 2380-2393.

[8] S. A. Nikulin, A. B. Rozhnopurv, S. O. Rogachev, V. M. Khatkevich, V. A. Turchenko, E. S. Khotulev. 2016. Investigation of structure, phase composition, and mechanical properties of Zr-2.5% Nb alloy after ECAP. *Materials Letter*. 169: 223-226.

[9] M. Isik, M. Niinomi, K. Cho, M. Nakai, H. Liu, H. Yilmazer. Z. Horita, S. Sato, T. Narushima. 2016. Microstructural evolution and mechanical properties of biomedical Co–Cr–Mo alloy subjected to high-pressure torsion. *Journal of the Mechanical Behavior of Biomedical Materials*.59:226-235.

[10] S. V. Dobatkina, O. V. Rybalchenko, N. A. Enikeev, A. A. Tokar, M. M. Abramova. 2016. Formation of fully austenitic ultrafine-grained high strength state in metastable Cr–Ni–Ti stainless steel by severe plastic deformation. *Materials Letters*. 166: 276-279.

[11] J. S. Carpenter, T. Nizolek, R. J. McCabe, M. Knezevic, S. J. Zheng, B. P. Eftink, J. E. Scott, S. C. Vogel, T. M. Pollock, N. A. Mara, I. J. Beyerlein. 2015. Bulk texture evolution of nanolamellar Zr- Nb composites process via accumulative roll bonding. *Acta Materialia*. 92: 97-108.

[12] A. Fattah-alhosseini, S. O. Gashti. 2015. Corrosion Behavior of Ultra-fine Grained 1050 Aluminum Alloy Fabricated by ARB Process in a Buffer Borate Solution. *Journal of Materials Engineering and Performance*. 24(9): 3386-3393.

[13] S. S. Satheesh Kumar, T. Raghu. 2015. Strain path effects on microstructural evolution and mechanical behaviour of constrained groove pressed aluminium sheets. *Materials and Design*. 88:799-809.

[14] F. Khodabakhshi, M. Haghshenas, H. Eskandari, and B. Koohbor. 2015. Hardness- strength relationships in fine and ultra-fine grained metals processed through constrained groove pressing. *Materials Science and Engineering: A*. 636: 331-339.

[15] N. Haghdadi, A. Zarei-Hanzaki, H. R. Abedi, D. Abou-Ras, M. Kawasaki, A. P. Zhilyaev. 2016. Evolution of microstructure and mechanical properties in a hypoeutectic Al-Si-Mg alloy processed by accumulative back extrusion. *Materials Science and Engineering: A*. 651: 269-279.

[16] N. Haghdadi, A. Zarei-Hanzaki, D. Abou-Ras, M. H. Maghsoudi, A. Ghorbani, M. Kawasaki. 2014. An investigation into the homogeneity of microstructure, strain pattern and hardness of pure aluminium processed by accumulative back extrusion. *Materials Science and Engineering: A*. 595: 179-187.

[17] G. Faraji, P. Yavari, S. Aghdamifar, M. Mosavi Mashhadi. 2014. Mechanical and microstructural properties of ultra-fine grained AZ91 magnesium alloy tube processed via multi pass tubular channel angular pressing (TCAP). *Journal of Materials Science and Technology*. 30(2): 134-138.

[18] F. Reshadi, G. Faraji, S. Aghdamifar, P. Yavari, M. M. Mashhadi. 2015. Deformation speed and temperature effect on magnesium AZ91 during tubular channel angular pressing. *Materials Science and Technology (United Kindom)*. 31(5): 1879-1885.

[19] Y. Estrin, A. Vinogradov. 2013. Extreme grain refinement by severe plastic deformation: A wealth of challenging science. *Acta Materialia*. 61: 782-817

[20] D. H. Shin, J. J. Park, Y. S. Kim, K. T. Park. 2002. Constrained groove pressing and its application to grain refinement of aluminium. *Materials Science and Engineering A*. 328: 98-103.

[21] F. Witte. 2010. The history of biodegradable magnesium implants: Areview. *Acta Biomaterialia*. 6: 1680- 1692.

[22] K. Inal, R. K. Mirshra. 2010. Crystal plasticity based numerical modelling of large strain deformation in hexagonal closed packed metals. *Procedia IUTAM*. 3: 239-273.

[23] J. Levesque, K. Inal, K. W. Neale, R. K. Mishra. 2010. Numerical modelling of formability of extruded magnesium alloy tubes. *International Journal of Plasticity*. 26: 65- 83.

[24] S. T. Adedokun. 2011. A review on equal channel angular extrusion as a deformation and grain refinement process. *Journal of Emerging Trends in Engineering and Applied Science (JETEA*S). 2(2): 360-363.

[25] M. Kawasaki, T. G. Langdon. 2015. Review: Achieving superplastic properties in ultrafine-grained materials at high temperatures. *Journal of Materials Science*. 51(1): 19-32.

[26] P. S. Roodposhti, N. Farahbakhsh, A. Sarkar, K. L. Murty. 2015. Microstructural approach to equal channel angular pressing of commercially pure titanium- A review. *Transaction of Nonferrous Metals Society of China*. 25: 1353-1366.

[27] I. Sabirov, M. Y. Murashkin, and R.Z. Valiev. 2013. Nanostructured aluminium alloys produces by severe plastic deformation: New horizons in development. *Materials Science and Engineering A*. 560: 1-24.

[28] P. W. J. Mckenzie and R. Lapovok. 2010. ECAP with back pressure for optimum strength and ductility in aluminium alloy 6016. Part 1: Microstructure. *Acta Materialia*. 58: 3198-3211.

[29] C. R. Ruiz, I. A. Figueroa, C. Braham, J. M. Cabrera, I. Alfonso and G. Gonzalez. 2015. Texture and lattice distortion sturdy of an Al-6061-T6 alloy produced by ECAP. *Materials Transactions*. 56(11): 1781-1786.

[30] V. M. Segal. 1999. Equal channel angular extrusion: From macromechanics to structure formation. *Material Science and Engineering A*. 271: 322-333.

[31] Y. Huang, and T. G. Langdon. 2013. Advance in ultrafine-grained material. *Materials Today*. 16(3): 85-93.

[32] A. Mishra, V. Richard, F. Gregori, R. J. Asaro, M. A. Meyers. 2005. Microstructural evolution in copper

processed by severe plastic deformation. *Materials Science and Engineering A*. 410-411: 290-298.

[33] Y. G. Kim, Y. G. Ko, D. H. Shin, and S. Lee. 2010. Effect of equal-channel angular pressing routes on high-strain-rate deformation behaviour of ultra-fine-grained aluminium alloy. *Acta Materialia*. 58: 2545-2554.

[34] Y. Iwahashi, Z. Horita, M. Nemoto and T. G. Langdon. 1997. The process of grain refinement in equal-channel angular pressing. *Acta Metallurgica*. 46(9): 3317-3331.

[35] A. Bommareddy, M. Z. Quadir, and M. Ferry. 2012. Time and temperature regime of continuous grain coarsening in an ECAP-processed Al (0.1 wt% Sc) alloy. *Journal of Alloys and Compounds*. 527: 145-151.

[36] M. Avvari. S. Narendranath. 2014. Influence of route-R on wrought magnesium AZ61 alloy mechanical properties through equal channel angular pressing. *Journal of Magnesium and Alloy*. 2: 159- 164.

[37] J. Suh, J. Victoria-Hernandez, D. Letzig, R. Golle, S. Yi, J. Bohlen, W. Volk. 2015. Improvement in cold formability of AZ31 magnesium alloy sheets processed by equal channel angular pressing. *Journal of Materials Processing Technology*. 217: 286- 294.

[38] F. D. Torre, R. Lapovok, J. Sandlin, P. F. Thomson, C. H. J. Davies, and E. V. Pereloma. 2004. Microstructures and properties of copper processed by equal channel angular extrusion for 1-16 passes. *Acta Materialia*. 52. 4819-4832.

[39] W. Blum, J. Dvorak, P. Krai, P. Eisenlohr, and V. Sklenicka. 2014. Effect of grain refinement by ECAP on creep of pure Cu. *Materials Science and Engineering A*. 590: 423-432.

[40] H. S. Kim, W. Y. Kim, and K. H. Song. 2012. Effect of post-heat-treatment in ECAP processed Cu-40%Zn brass. *Journal of Alloys and Compounds*. 536: 200-203.

[41] Y. Fukuda, K. Oh- ishi, Z. Horita, T. G.Langdon. 2002. Processing of a low-carbon steel by equal-channel angular pressing. *Acta Materialia*. 50: 1359- 1368.

[42] M. Eddahbi, M. A. Monge, T. Leguey, P. Fernandez, and R. Pareja. 2011. Texture and mechanical properties of EUROFER 97 steel processed by ECAP. *Materials Science and Engineering A*. 528: 5927-5934.

[43] K. R. Cardospo, D. N. Travessa, W. J. Botta, A. M. Jorge Jr. 2011. High strength AA7050 Al alloy processed by ECAP: Microstructure and mechanical properties. *Materials Science and Engineering A*. 528: 5804-5811.

[44] Y. Han, J. Li, G. Huang, Y. Lv, X. Shao, W. Lu, and D. Zhang. 2015. Effect of ECAP numbers on microstructure and properties of titanium matrix composite. *Materials and Design*. 75: 113-119.

[45] Z. J. Zheng, Y. Gau, J. W. Liu, and M. Zhu. 2015. A hybrid refining mechanism of microstructure of 304 stainless steel subjected to ECAP at 500°C. *Materials Science and Engineering A*. 639: 615-625.

[46] G. Reglitz, B. Oberdorfer, N. Fleischmann, J. A. Kotzurek, S. V. Divinski, W. Sprengel, G. Wilde, and R. Wurschum. 2016. Combined volumetric, energetic and microstructural defect analysis of ECAP-processed nickel. *Acta Materialia*. 103: 396-406.

[47] W. Blum, Y. J. Li, Y. Zhang, and J. T. Wang. 2011. Deformation resistance in the transition from coarse-grained to ultrafine-grained Cu by severe plastic deformation up to 24 passes of ECAP. *Materials Science and Engineering A*. 528 (29-30): 8621-8627.

[48] N. D. Stepanov, A. V. Kuznetsov, G. A. Salishchev, G. I. Raab, and R. Z. Valiev. 2012. Effect of cold rolling on microstructure and mechanical properties of copper subjected to ECAP with various number of passes. *Materials Science and Engineering A.* 554: 105-115.

[49] F.Djavanroodi, and M. Ebrahimi. 2010. Effect of die parameters and material properties in ECAP with parallel channels. *Materials Science and Engineering A*. 527: 7593-7599.

[50] X. H. Zhang, S. J. Luo. Z. M. Du. 2008. Uniformity and continuity of effective strain in AZ91D processed by multi- pass equal channel angular extrusion. *Transaction of Nonferrous Metals Society of China*. 18: 92- 98.

[51] R. Z. Valiev and T. G. Langdon. 2006. Principles of equal channel angular pressing as a processing tool for grain refinement. *Progress in Materials Science*. 51: 881-981.

[52] J. Li, F. Li, C. Zhao, H. Chen, X. Ma, J. Li. 2016. Experimental study in pure copper subjected to different severe plastic deformation modes. *Materials Science and Engineering A.* 656: 142-150.

[53] Y. Iwahashi, J. Wang, Z. Horita, M. Nemoto and T. G. Langdon. 1996. Principle of equal channel angular pressing for the processing of ultra-fine grained materials. *Acta Metallurgica*. 35(2): 143-146.

[54] N. S. D. Vincentis, A. Kliauga, M. Ferrante, M Avalos, H. G. Brokmeier, and R. E. Bolmaro. 2015. Evaluation of microstructure anisotropy on room and medium temperature ECAP deformed F138 steel. *Materials Characterization*. 107: 98-111.

[55] A. Yamashita, D. Yamagichi, Z. Horita, T. G. Langdon. 2000. Influence of pressing temperature on microstructural development in equal channel angular pressing. *Materials Science and Engineering A*. 287: 100-106.

[56] Y. Y. Wang, P. L. Sun, P. W. Kao, C. P. Chang. 2004. Effect of deformation temperature on the microstructure developed in commercial purity aluminium processed by equal channel angular extrusion. *Script Materialia*. 50: 613-617.

[57] Y. Xirong, Z. Xicheng, F. Wenjie. 2009. Deformed microstructures and mechanical properties of CP-Ti processed by multi-pass ECAP at room temperature. Ra*re Metal Materials and Engineering*. 38(6): 955-957.

[58] K. Furuno, H. Akamatsu, K. Oh-0shi, M. Furukawa, Z. Horita, T. G. Langdon. 2004. Microstructure development in equal channel angular pressing using a 60° die. *Acta Materialia*. 52: 2497-2505.

[59] J. Jiang, Y. Wang, J. Qu. 2013. Microstructure and mechanical properties of AZ61 alloys with large cross-sectional size fabricated by multi – pass ECAP. *Materials Science and Engineering A*. 560: 473- 480.

[60] V. V. Stolyarov, R. Lapovok, I. G. Brodova, P. F. Thomson. 2003. Ultrafine- grained Al-5wt. % Fe alloy processed by ECAP with backpressure. *Materials Science and Engineering A*. 357(1-2): 159-167.

[61] C. Xu, K. Xia, T. G. Langdon. 2009. Processing of magnesium alloy by equal channel angular pressing using back pressure. *Materials Science and Engineering A*. 527(1-2): 205-211.

[62] V. V. Stolyarov, Y. T. Zhu, I. V. Alexandrov, T. C. Lowe. R. Z. Valiev. 2001. Influence of ECAP routes on the microstructure and properties of pure Ti. *Materials Science and Engineering A.* 299 (1-2): 59-67.

[63] Z. Lin, L. Wang, X. Xue, W. Lu, J. Qin, D. Zhang.2013. Microstructure evolution and mechanical properties of a Ti-35Nb-3Zr-2Ta biomedical alloy processed by equal channel angular pressing (ECAP). *Materials Science and Engineering C*. 33:4551-4561.

[64] S. Komura, M. Fukurawa, Z. Horita, M. Nemoto. T. G. Langdon. 2001. Optimizing the procedure of equal channel angular pressing for maximum superplasticity. *Materials Science and Engineering A*. 297 (1-2): 111-118.

[65] M. Avvari, S. Narendranath, H. S. Nayaka. 2013. Effect of equal channel angular pressing on AZ31 wrought magnesium alloys. *Journal of Magnesium and Alloys*. 1: 336- 340.

[66] R. Jahadi, M. Sedighi, H. Jahed. 2014. ECAP effect on the microstructure and mechanical propertied of AM30 magnesium alloy. Materials Science and Engineering *A*. 593: 178- 184.

[67] Akihiro Yamashita, Zenji Horita, Terence G. Langdon. 2001. Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation. *Journal of Materials Processing Technology*. 217: 286- 293.

[68] P. Minarik, R. Kral, M. Janecek, J. Pesicka. 2013. Evolution of the microstructure, mechanical properties and corrosion resistance of AE21 magnesium alloy after ECAP. *Metal 2013: 22nd International Conference on Metallurgy and Materials*. 1-6.

[69] P. Minarik, R. Kral, J. Pesicka, F. Chmelik. 2015. Evolution of mechanical properties of LAE 442 magnesium alloy processed by extrusion and ECAP. *Journal of Materials Research and Technology*. 4 (1): 75-78.

[70] F. D. Dumitru, O. F. Higuera- Cobos, J. M Cabrera. 2014. ZK60 alloy processed by ECAP: microstructural, physical and mechanical characterization. *Materials Science and Engineering A*. 594: 32- 39.

[71] F. S. J. Poggiali, C. L. P. Silva, P. H. R. Pereira, R. B. Figueiredo, P. R. Cetlin. 2014. Determination of mechanical anisotropy of magnesium processed by ECAP. *Journal of Materials Research and Technology*. 3 (4): 331- 337.

[72] M. Avvari, S. Narendranath, H. S. Nayaka. 2014. Effect of processing routes on AZ31 alloy processed by severe plastic deformation. *Procedia Materials Science*. 5: 1560-1566.

[73] K. Xia, J. T. Wang, X. Wu, G. Chen, M. Gurvan. 2005. Equal chnnel angular pressing of magnesium alloy AZ31. *Materials Science and Engineering A*. 410- 411: 324- 327.

[74] L. Jin, D. Lin, D. Mao, X. Zeng, W. Ding. 2005. Mechanical properties and microstructure of AZ31 Mg alloy processed by two- step equal channel angular extrusion. *Materials Letters*. 59 (18): 2267-2270.

[75] M. Avvari, S. Narendranath, H. S. Nayaka. 2014. Effect of processing routes on AZ31 alloy processed by severe plastic deformation. *Procedia Materils Science*. 5: 1560-1566.

[76] S. Komora, M. Furukawa. Z. Horita, M. Nemoto, T. G. Langdon. 2001. Optimizing the procedure of equal-channel angular pressing for maximum superplasticity. *Materials Science and Engineering A*. 297: 111-118.

[77] V. V. Stolyarov, Y. T. Zhu, I. V. Alexandrov, T. C. Lowe, R. Z. Valliev. 2001. Influence of ECAP routes on the microstructure and properties of pure Ti. *Materials Science and Engineering A*. 299: 59-67.

[78] L. B. Tong, M. Y. Zheng, X. S. Hu, K. Wu, S. W. Xu, K. Y. Kojima. 2010. Influence of ECAP routes on microstructure and mechanical properties of Mg-Zn-Ca alloy. *Materials Science and Engineering A*. 527: 4250-4256.

[79] W. J. Kim, J. C. Namkung. 2005. Computational analysis of effect of route on strain uniformity in equal channel angular extrusion. *Materials Science and Engineering A*. 412: 287-297.

[80] S. R. Agnew, J. A. Horton, T. M. Lillo, D. W. Brown. 2004. Enhanced ductility in strongly textured magnesium produced by equal channel angular processing. *Scripta Materialia*. 50 (3): 377- 381.

[81] F. Kang, J. T. Wang, Y. Peng. 2008. Deformation and fracture during equal channel angular pressing of AZ31 magnesium alloy. *Materials Science and Engineering A*. 487: 68- 73.

[82] I.P. Semenova, G.I. Raab, L.R. Saitova, R.Z. Valiev. 2004. The effect of equal-channel angular pressing on the structure and mechanical behavior of Ti–6Al–4V alloy. *Materials Science and Engineering A,* 387-389: 805-808.

[83] Z. Horita, M. Furukawa, M. Nemoto, A. J. Barnes and T. G. Langdon. 2000. Superplastic forming at high strain rates after severe plastic deformation, *Acta Materialia*. 48 (14): 3633-3640.

[84] S. Ferrasse, V. M. Segal, F. Alford. 2004. Texture evolution during equal channel angular extrusion (ECAE): Part II. An effect of post- deformation annealing. *Materials Science and Engineering A*. 372 (1-2): 235-244.

[85] L. S. Toth, R. A. Massion, L. Germain, S. C. Baik, S. Suwas. 2004. Analysis of texture evolution in equal channel angular extrusion of copper using a new flow field. *Acta Materialia.* 52 (7): 1885- 1898.

[86] S. Li, I. J. Beyerlein, C. T. Necker, D. J. Alexander, M. Bourke. 2004. Heterogeneity of deformation texture in equal channel angular extrusion of copper. *Acta Materialia*. 52 (16): 4859- 4875.

[87] C. Y. Yu, P. L. Sun, P. W. Kao, C. P. Chang. 2005. Mechanical properties of submicron-grained aluminium. *Scripta Materialia*. 52 (5): 359. 363.

[88] M. Moradi, M. Nili-Ahmadabadi, B. Poorganji, B. Heidarian, M. H. Parsa, T. Furuhara. 2010. Recrystallization behavior of ECAPed A356 alloy at semi-solid reheating temperature. *Materials Science and Engineering: A*. 527:4113-4121.