

## Superplastic behavior of AZ31B Magnesium alloy processed by Equal Channel Angular Pressing (ECAP)

Ziad Tariq Madhloom

Production & Materials Engineering Department, University of Technology / Baghdad

Eng\_zaid188@yahoo.com

Dr. Azal R. Ismail

Production & Materials Engineering Department, University of Technology / Baghdad

Received on: 5/9/2013 & Accepted on: 6/3/2014

### ABSTRACT

AZ31B magnesium alloy with initial grain size of 11.2  $\mu\text{m}$  was grain refined using Equal Channel Angular Pressing (ECAP) the alloy was ECAP-processed up to 5-times at 473 K. As a result, the grains are refined to 2.1  $\mu\text{m}$  after three ECAP passes, the elongation was increased to 84 % at 473 K and strain rate of  $1 \times 10^{-4} \text{ s}^{-1}$  after ECAP up to 5-passes. Large fracture elongations of 261% are obtained at 623 K and strain rate  $2.45 \times 10^{-4} \text{ s}^{-1}$  for specimen made from 3 ECAR passes compared to 183% for the as received material. Also the superplastic properties of the ECAP-processed specimens were investigated at 623 K and the strain rate ranges from  $2.45 \times 10^{-4} \text{ s}^{-1}$  to  $1.2 \times 10^{-3} \text{ s}^{-1}$ . The strain rate sensitivity were determined to be 0.33

**Keyword:** Superplasticity, Magnesium alloy, AZ31B, Severe plastic deformation.

سلوك اللدونة الفائقة لسبيكة المغنيسيوم من نوع AZ31B المشكلة بواسطة قلب بثق متساوي المقطع

الخلاصة:

تم استخدام قالب بثق متساوي المقطع ECAP وذلك لتنعيم سبيكة المغنيسيوم من نوع AZ31B والتي تمتلك بنية مجهرية بحدود 11.2 مايكرو متر , تم استخدام تقنية ECAP لهذه السبيكة لخمس مرات و بدرجة حرارة 423 ° كلفن وكننتيجة لذلك تم تنعيم البنية المجهرية الى 2.1 ميكرو متر , و كذلك اصبحت الاستطالة 84% في درجة حرارة 423° كلفن و معدل انفعال  $1 \times 10^{-4} \text{ s}^{-1}$  و بعد خمسة تمريرات في قالب ECAP , ايضا تم فحص اللدونة الفائقة superplastic test حيث اعطت السبيكة استطالة بحدود 261% في درجة حرارة 623° كلفن و معدل انفعال  $2.45 \times 10^{-4} \text{ s}^{-1}$  لعينات مصنوعة من ثلاث كبسات في قالب ECAP مقارنة مع 183% استطالة للمادة بدون اجراء عملية ECAP .

### INTRODUCTION

Magnesium with specific gravity of 1.74 is the lightest structural material on earth. Magnesium has good damping capacity, high specific strength compared to steel and aluminum. Recently magnesium become more important in reduce component weight such as vehicle due to the increase in the fuel prices weight, while it is

originally used in aerospace application. However, magnesium alloys have poor formability and limited ductility at room temperature because of their H.C.P. crystal structure with limited operative slip systems at room temperature which restrict their application [1-3], as a matter of fact H.C.P. materials do not have enough independent slip system to deform without extensive occurrence of twinning. Magnesium alloys are usually manufactured through conventional casting and its narrow plastic range requires close control in forging.

Formability of magnesium alloys can be improved by superplastic forming (SPF). Severe plastic deformation techniques (SPD) is widely used to produce fine structure metallic material for SPF this type of material believed to deform principally by a grain boundary sliding mechanism, for grain-boundary sliding, and to dominate deformation it requires essentially for the microstructure less than 10 μm with equiaxed grains and high angle grain boundary. These materials exhibits superplastic behavior and deform at high temperature and low strain rate, usually the elongation to failure is above 200% which indicative of superplasticity although several materials can attain extension greater than 1000% [4], superplasticity depends on the parameters of the microstructure both matrix and secondary phase grain size, the temperature suitable for superplasticity is matrix material dependent. Presently, among the methods of SPD, equal channel angular extrusion (ECAE) is considered as the most promising for industrial applications [5]. It has shown recently that it is feasible to achieve excellent grain refinement in magnesium alloys by using ECAP [6] other techniques of SPD are equal channel angular rolling (ECAR), differential speed rolling (DSR), and many other techniques.

The aim of the present work is to investigate the effect of ECAP on the mechanical behavior and superplasticity for AZ31B Magnesium alloy.

**Experimental work**

The material used in the present study was a 11mm thickness plate of AZ31B Magnesium alloy supplied by a commercial magnesium company provider. The chemical composition is listed in table.(1) equivalent to AMS 4377 with a H24 temper, which is partially annealed after strain hardening. The 0.2% yield strength is 205 MPa and tensile strength of 265 Mpa elongation of 13% at room temperature.

The plate were machined into 10× 10 × 65 mm and prepared for the ECAP process, the measured grain size in the as received condition was ~11.2 μm using Linear Intercept Method

**Table (1) the chemical composition % of AZ31B**

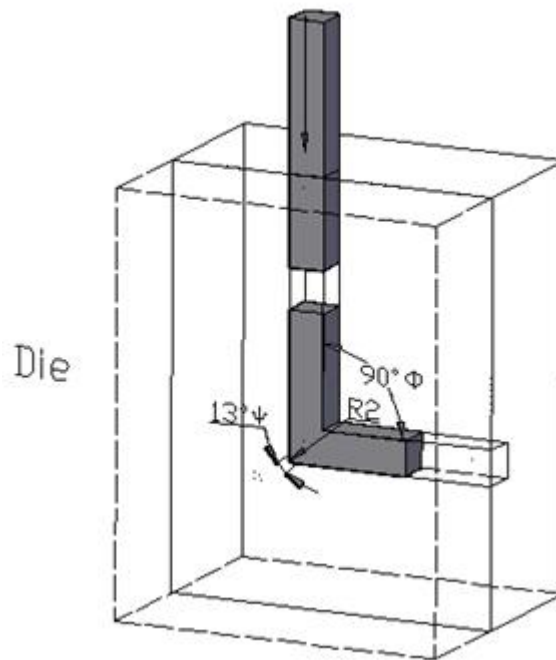
Element	Al	Zn	Mn	Fe	Cu	Si	Ni	Ca
%	3.09	0.71	0.42	0.003	0.0019	0.018	0.0005	0.0043

The ECAP processing was conducted at 473 K using a solid die made from a block of AISI 5130 alloy tool steel with chemical composition listed in table (2) which had surface hardness of 328 HV after hardening to 860 °C. The die was made in two parts that allow cleaning after the use and also to enable removing any fractured billets in case of unexpected material failure. The two halves of the die were fastened using four M12 steel

screws. A scheme of the die shown in Fig.(1). The die consist of  $10 \times 10$  mm square channel intersecting at an angle  $\Phi$  of  $90^\circ$  between the two parts of channel and an angle of curvature  $\Psi=13^\circ$  at the outer arc of curvature at the point of intersection of the two channel, these angles lead to an imposed strain 1.08 on each single pass of the specimen though the die. Pressing were conducted up to a maximum of five passes, representing an imposed strain of  $\approx 5$ , with each specimen rotated by  $90^\circ$  in the same sense between consecutive passes in the processing procedure designated route B<sub>C</sub>. In order to facilitate the pressing, each specimen was lubricated with graphite grease prior to placing in the channel within the die. To ensure thermal equilibrium, the die is pre-heated for one hour and half for the setup temperature reaches 473 K, Each specimen was held in the die at the required temperature for approximately 10 minutes till the homogeneity of the temperature the process of ECAP is performed in a speed of 10 mm per minute, After the pressing the formed billet were removed from the channel exit and then left to cool at room temperature, A billet with 1pass, 2passes, 3passes, 4 passes, 5passes were competed.

**Table (2) Chemical composition% of the die material.**

Element	C	Si	Mn	Cr	Mo	Ni	Cu
%	0.319	0.236	0.994	1.08	0.196	0.249	0.145



**Figure (1) a scheme of the ECAP die**

The die was heated by using four resistance fingers of 300 Watt heating while a thermocouple is connected to a temperature controller that controls the power supply to the heating elements as shown in Fig. (2.b).

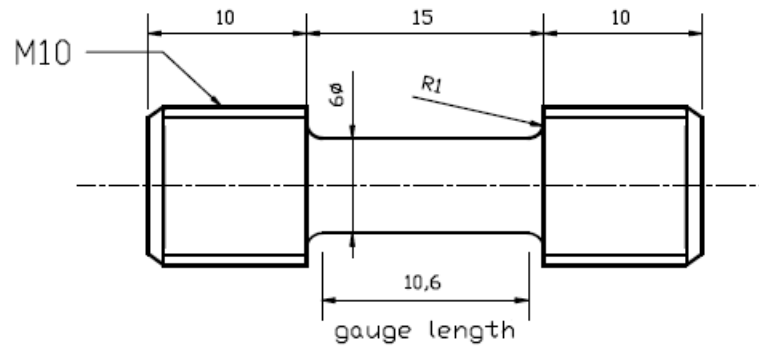


(a)

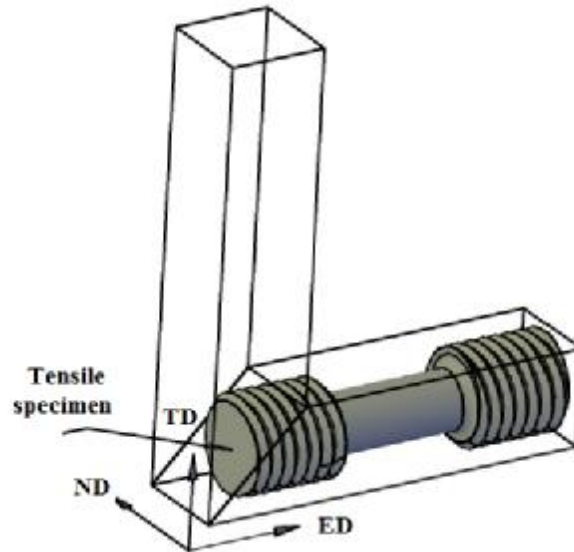
(b)

**Figure (2) ECAP die and the temperature controller (a) the die and heating elements (b) the temperature controller**

Samples from the pressed billet were cut and cold mounted using epoxy resin mixed with epoxy hardener. Six steps included five grinding steps: 120 grid SiC, 230 grid SiC, 500 grid SiC, 1000 grid SiC paper and 2000 grid SiC paper, then polishing, 3  $\mu\text{m}$  cloth with  $\text{Al}_2\text{O}_3$  powder, to obtain scratch-free surfaces with high reflectivity. Samples were etched in 4.2 g picric acid + 10 ml acetic acid + 10 ml  $\text{H}_2\text{O}$  + 70 ml ethanol (95%) solution. Microstructure examination was carried out in parallel section of X-plane (TD)-(ND).



**(a)Figure (3) To be continued**



(b)

**Figure(3). The tensile specimen (a) the specimen alignment with the extrusion direction (b) Dimensions of tensile test specimens for investigation of tensile properties (mm).**

In order to evaluate the potential of superplastic flow a tensile specimen of round cross-section of 6 mm in diameter and 10.6 mm gauge length was machined from the ECAP billet, Fig. 3. a. The samples had tensile axes lying parallel to the ED direction as shown in the Fig. 3.b the tensile testing was conducted at elevated temperature as follow:

1. Tensile test at 473 K and strain rate of a  $1 \times 10^{-4} \text{ s}^{-1}$  for specimens made from 1, 2, 3, 4, and 5, passes ECAP.
2. Tensile test at 623 K at initial strain rates ranges from  $2.4 \times 10^{-4} \text{ s}^{-1}$  to  $1.2 \times 10^{-3} \text{ s}^{-1}$  for strain rate sensitivity evaluation.

## RESULTS AND DISCUSSION

### Effect of passes on microstructures

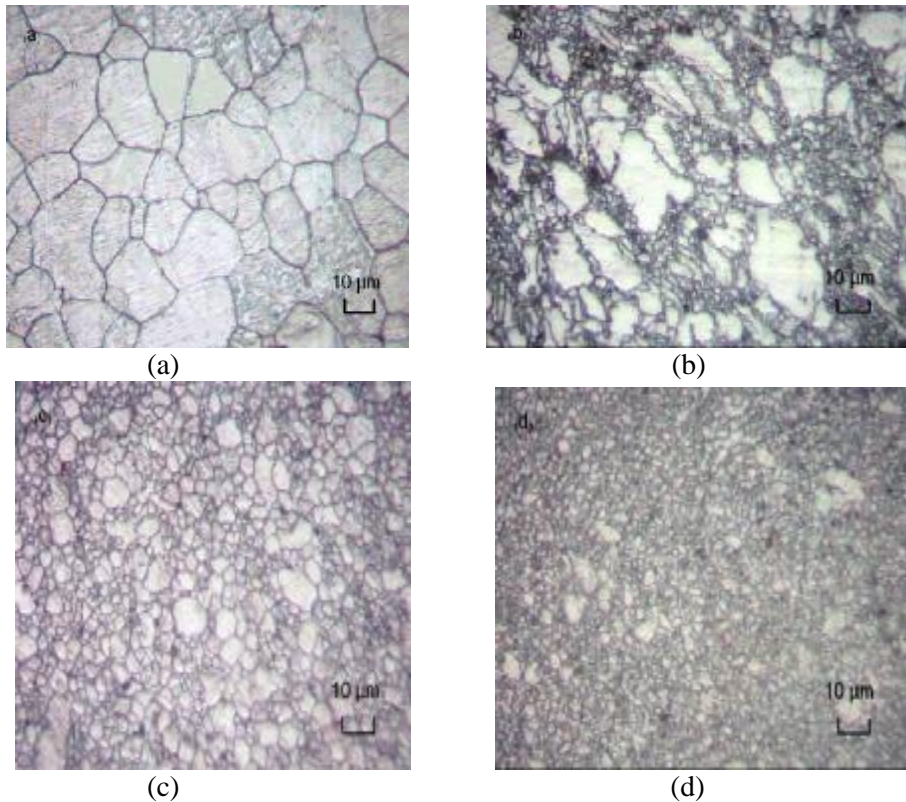
The microstructure of the as-received material prior to ECAP is shown in Fig. (4. a.) The mean grain size,  $d$  was determined using linear intercept method [7]. Fig.(4) shows the optical microstructure of the material before and after ECAP (route B<sub>C</sub> and temp. 473 k) for (a) initial condition (b) 1-pass, (c) 2-passes, (d) 3-passes, (d) 4-passes and (f) 5-passes. The microstructure of AZ31B alloy in the initial condition shows variety distribution of grain sizes from 5-30  $\mu\text{m}$ , measurements gave an average grain size in this condition of 11.2  $\mu\text{m}$  as shown in Fig. (4). a also some twins were observed in the microstructure. The first ECAP pass characterize as “bimodal” distribution, for such

model the microstructure consist of very fine grains ( $0.5\text{-}1\ \mu\text{m}$ ) as well as coarse grains ( $9\text{-}20\ \mu\text{m}$ ) the bimodal distribution of grains is probably due to limited slip system in magnesium alloys, where new fine grains are formed along the initial boundaries and along twin boundaries [8] as shown in Fig. (4. B), the measured average grain size was  $3.6\ \mu\text{m}$ . The second ECAP processing at the same temperature increases the volume fracture of fine grains while large decreases and more homogeneous microstructure obtained, as shown in Fig. (4. C), the measured average grain size was  $3.2\ \mu\text{m}$ . After three passes the coarse grains have been removed completely and the grains become more equiaxed along the billet cross section as shown in Fig. (4. D). For this condition, the measured average grain size was  $2.1\ \mu\text{m}$ . further ECAP passes shows less effect to the grain refinement. The average grain size for the specimen with Fourth Passes was  $2.2\ \mu\text{m}$  and for the fifth passes  $2.4\ \mu\text{m}$  as shown in (Fig.4) .e and f respectively.

The microstructure observations demonstrate that three passes of ECAP are sufficient to produce a homogeneous grain structure in the AZ31B alloy and the grin size remains stable after this condition.

The microstructure shows significant grain refinement due to continuous dynamic recrystallization by ECAP passes.

The mean grain sizes of the as-received condition and ECAP processed alloys, d, measured based on the microstructure in Fig. 4 are plotted in Fig. 5 per each ECAP passes.



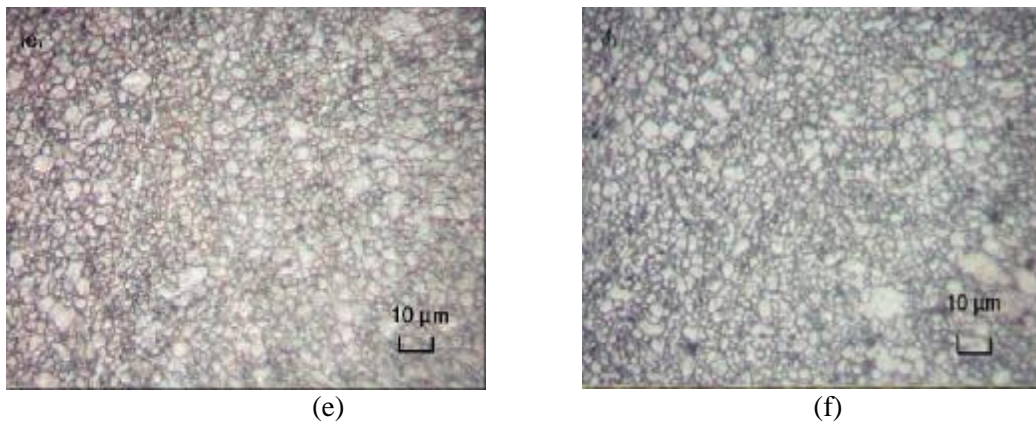


Figure (4) Microstructure of AZ31B magnesium alloy (a) as received condition 11.μm. (b) 1 pass ECAP 3.6μm. (c) 2 passes ECAP 3.2μm. (d) 3 passes ECAP 2.1μm. (e) 4 passes ECAP 2.2μm. (f) 5 passes ECAP 2.4μm.

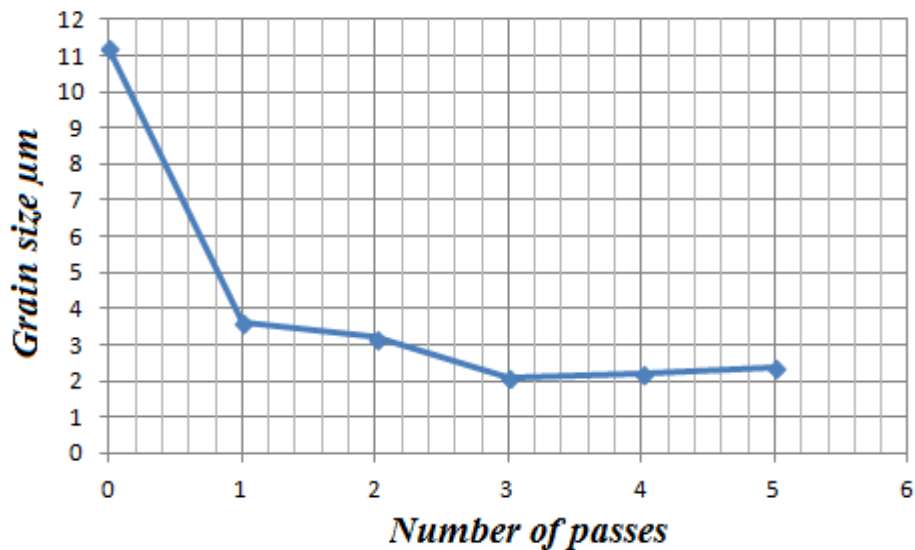


Figure (5) The grin size as a function of ECAP passes.

**Mechanical properties at elevated temperature**

**Tensile test at 473 K**

True Stress true strain relations for the ECAP processed AZ31B at 473 K and strain rate of  $1 \times 10^{-4} \text{ s}^{-1}$  is presented in Fig.(6). The test was carried out in order to investigate any low temperature superplasticity. Inspection shows that the flow stress decreases after reaching a peak value flowed by continuous decrease in the flow stress down to failure where this is attributed to necking during the tensile deformation. Furthermore, the material exhibits hardening in the initial stages of deformation and this effect are more

pronounced at the lower temperatures (like 473 K). The yield stress increases with increasing ECAP passes , yield stress increases from 97 Mpa for the specimen made from 1 pass ECAP to 150 MPa for the specimen with 2 Passes and reaches the maximum of 159 MPa for the specimen 3 passes, further passes shows decreasing to the yield stress as in the specimen 4 passes, the yield stress decreased to 104 MPa and for the specimen 5 passes the yield stress is increased slightly to 114 MPa, the decrease of the yield stress is due to the strain imposed to the material with further ECAP passes.

The ultimate tensile stress shows similar behavior to the yield stress, for the first and the second passes the ultimate tensile stress is a bit higher than the yield stress up to three passes where the difference became much higher, the reason for this behavior caused by the higher strain accumulation to the billet during the ECAP process as shown in Fig. 7.

The test displays improved ductilities at 473 K with increasing the number of ECAP passes, the elongation increases from 18.4% for the specimen with one pass to 49.7% for the two passes and to 59.8% for the third pass , 79.9% for the forth pass and to 84.7% for the fifth pass as shown in Fig. (7).

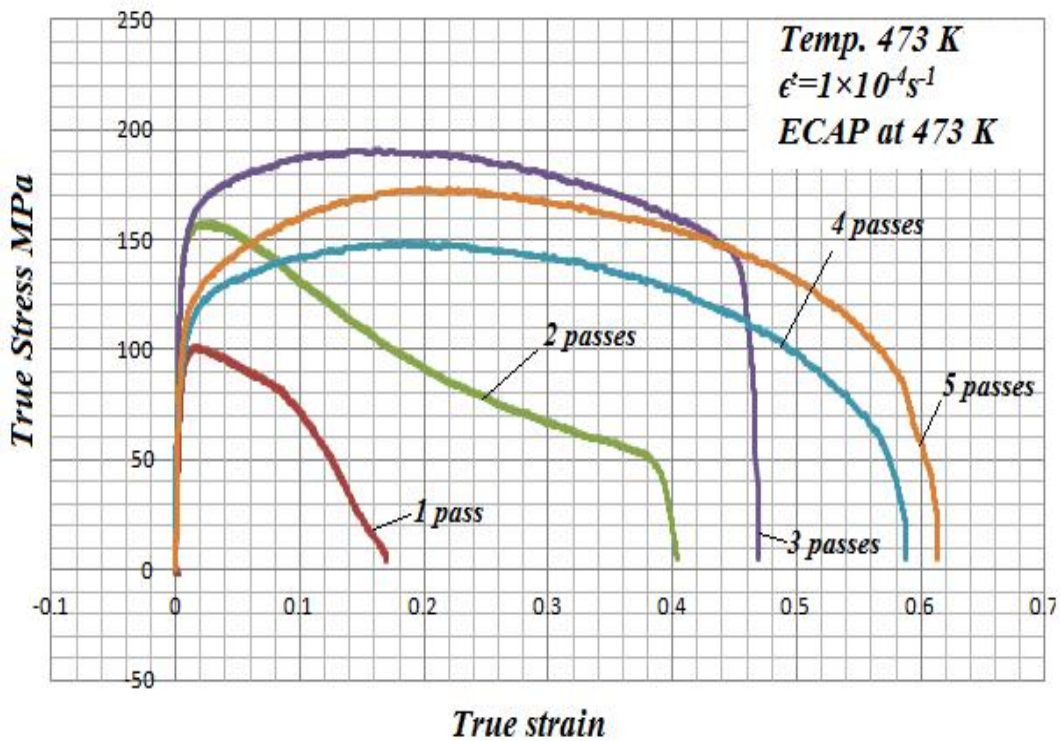


Figure (6) stress strain curves for specimens made from the ECAP billet



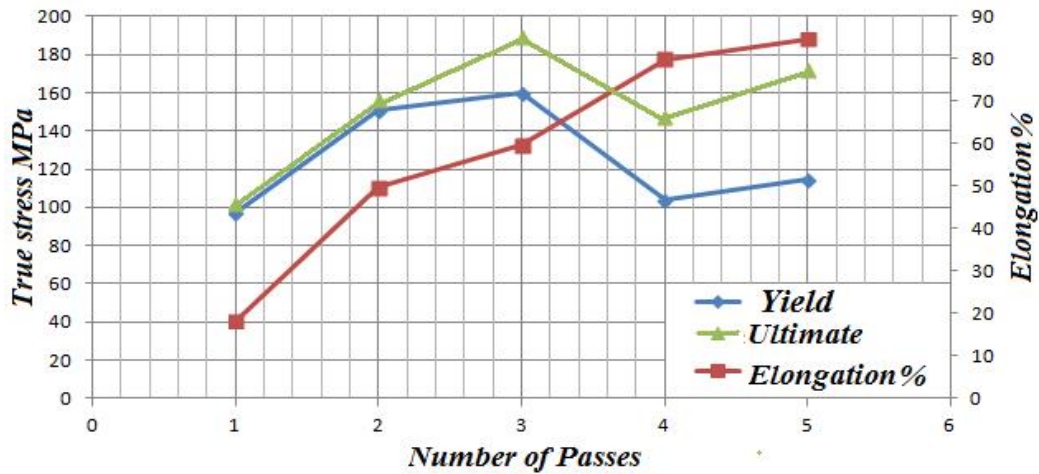


Figure (7) Yield, ultimate stress and elongation as a function of ECAR passes.

**Superplastic behavior at 623 K**

Tensile test were carried out for a variety of strain ranges from  $2.4 \times 10^{-4} \text{ s}^{-1}$  to  $1.2 \times 10^{-3} \text{ s}^{-1}$  and temperature of 623 K (equivalent to  $\sim 0.67 T_m$  where  $T_m$  is the melting point expressed in Kelvin) for specimens made from ECAP billet with 3 passes. The typical true stress true strain curves for this test is shown in Fig. (8). It can be seen clearly that the flow stress increases concurrently with increasing strain rate. The highest elongation achieved under these conditions was 261% at a strain rate of  $2.4 \times 10^{-4} \text{ s}^{-1}$ , the tensile specimens shows subsequent softening after strain hardening due to the large amount of elongation encountered by the tensile load during the test, these phenomena decreases with increasing strain rate.

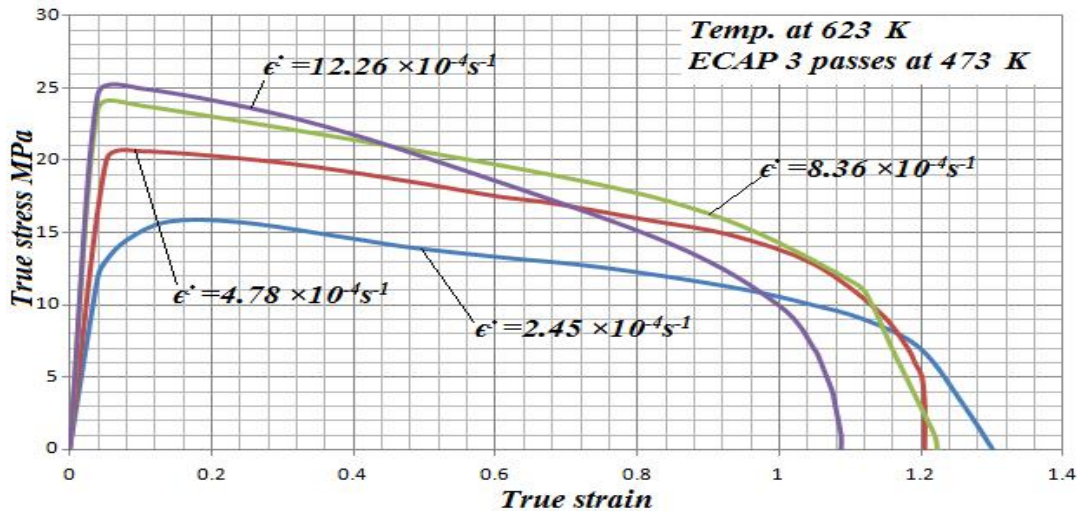


Figure (8) Typical stress and strain curves for AZ31B tensile loaded at 623 K

A full summary of tensile elongations is given in Fig. (9) where the results are plotted against the testing strain rate at 623 K for the experiments in Fig. (8).

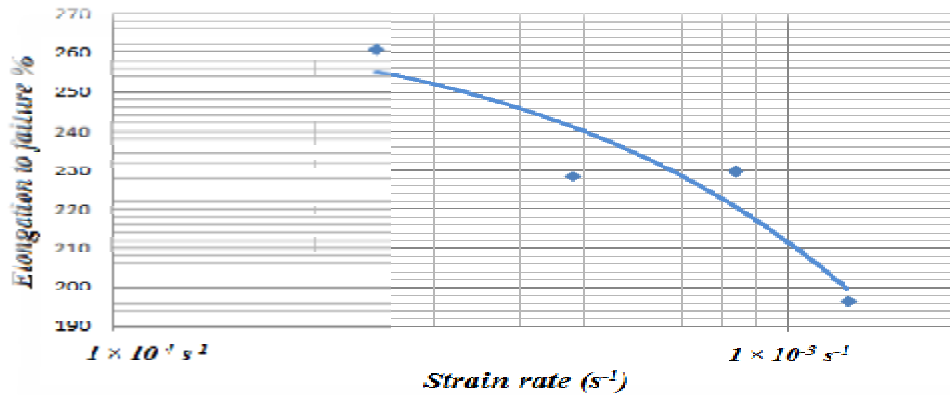
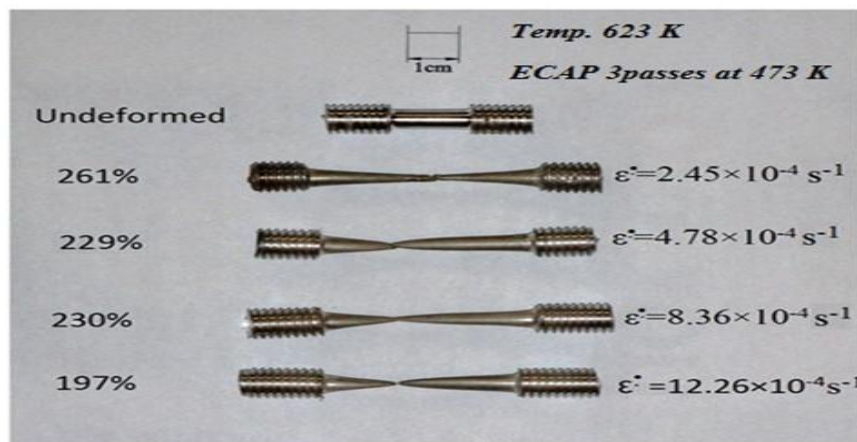


Figure (9) The trend of tensile elongation as a function of test strain rate for AZ31B magnesium alloy.

Fig.(10) shows the fractured specimens obtained at temperature of 623 K and various strain rates. Clearly, the elongation increases gradually with decreasing strain rate. At the lowest strain rate of  $2.45 \times 10^{-4} \text{ s}^{-1}$ , a maximum elongation of 261% is obtained implying uniform deformation of AZ31B Mg alloy at low strain rate ( $< 2.4 \times 10^{-4} \text{ s}^{-1}$ ). While at the relatively high strain rate range of  $4.78 \times 10^{-4} \text{ s}^{-1} \sim 1.2 \times 10^{-3} \text{ s}^{-1}$ , the change of elongation with strain rate is rather small and the necking becomes more obvious at high strain rates. This shows that the resistance of AZ31B Mg alloy to flow localization is significantly reduced with decreasing strain rate; therefore superplasticity cannot be obtained at high strain rates ( $> 2.4 \times 10^{-4} \text{ s}^{-1}$ ).



Figure(10) Fracture Specimens tested at 623 K with various strain rates.

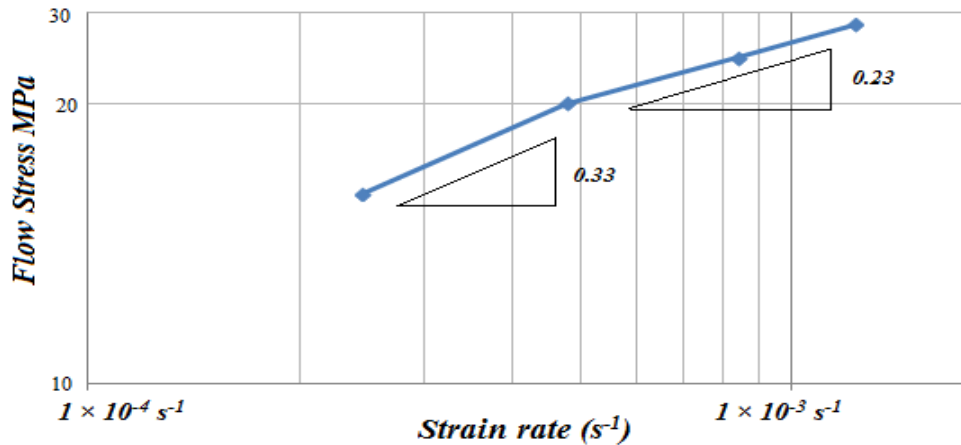


Figure (11) Double logarithmic scale of Flow stress-strain rate relationship at 0.1 strain rate and 623 K.

Fig.(11) shows the variation of flow stress with strain rate in a double logarithmic scale at 623 K. It is noticed that, the flow stress increases significantly with increasing strain rate and a sigmoidal relationship is found between flow stress and strain rate.

It can be seen from the figure that the flow stress decreases as the strain rate increases. At strain rate range of  $2.4 \times 10^{-4} \text{ s}^{-1} \sim 4.7 \times 10^{-4} \text{ s}^{-1}$ , the maximum strain-rate-sensitivity exponent  $m$  reaches 0.33, suggests that deformation probably occurs through viscous glide control creep process where  $m=0.3$  [9].

Fig.(12) shows the fractured specimens obtained at tensile temperature of 623 K, strain rate of  $2.4 \times 10^{-4} \text{ s}^{-1}$  and two kinds of specimens one made from the as received material and the other made from 3passes ECAP. Clearly, the elongation increases remarkably with decreasing the alloy grain size.

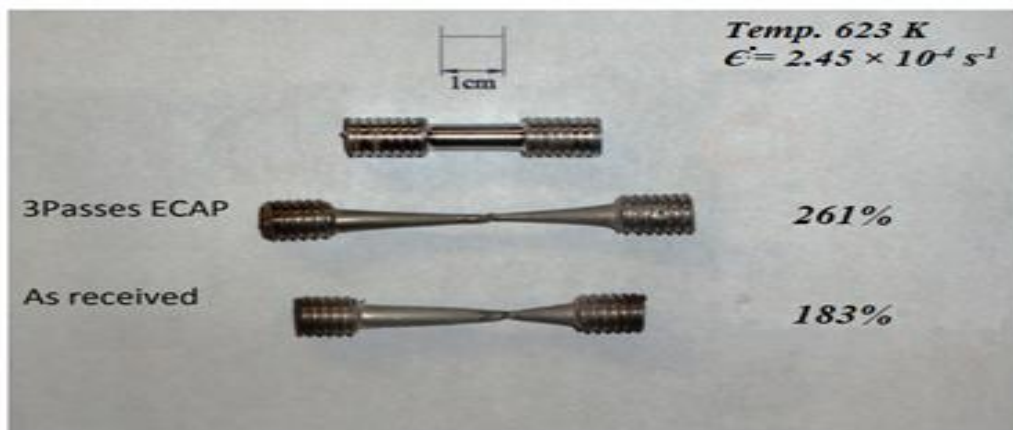
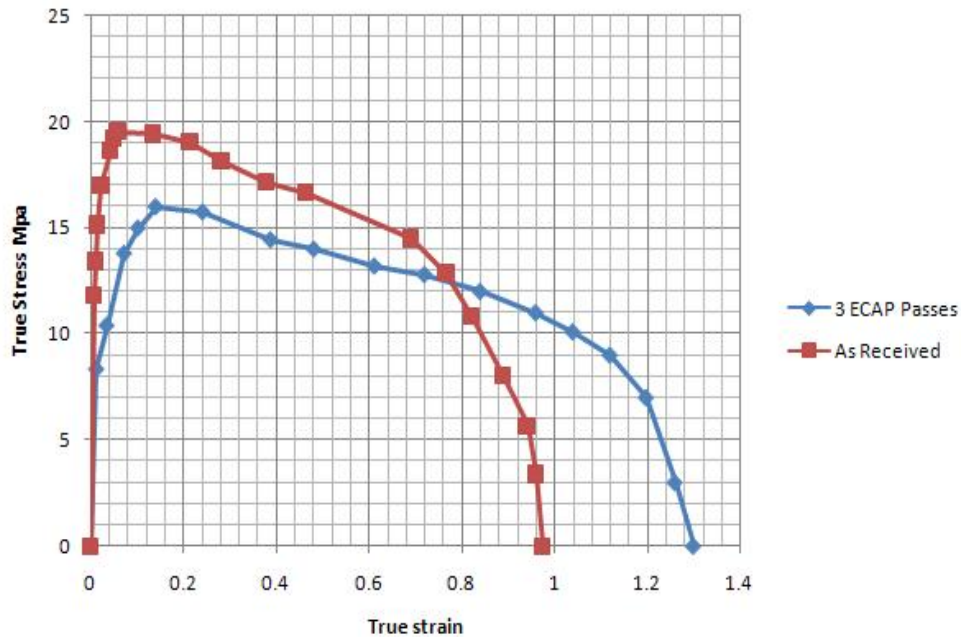


Figure (12) Fractured specimens after pulled to failure for the as received and 3 Passes ECAP at 623 K.

Fig. (13) shows the true stress- true strain curve for the specimens made from the as-received condition and 3-passes ECAP loaded at 623 K and initial strain rate of  $2.4 \times 10^{-4} \text{ s}^{-1}$ .



**Figure (13) True stress true strain curve for the as received and 3passes ECAP at strain rate of  $2.45 \times 10^{-4} \text{ s}^{-1}$  and temperature of 623 K.**

## CONCLUSIONS

1. Experiments were conducted using a commercial wrought AZ31B magnesium alloy the as received magnesium alloy were ECAP processed up to 5 passes at 473 K the microstructure shows remarkable grain refinement from  $14.2\mu\text{m}$  to  $2.1\mu\text{m}$ .
2. The mechanical properties of ECAP processed specimen were examined using hot tensile test at 473 K the experiments shows that with increasing the number of passes the elongation increased to 84.7% for 5-passes.
3. The yield stress decreases after the third pass due to the large strain imposed into the material.
4. Superplastic elongation were achieved in specimens machined from billet processed through 3-passes ECAP with maximum elongation of 261% at strain rate of  $2.45 \times 10^{-4} \text{ s}^{-1}$  and test temperature of 623 K
5. Only 183% fracture elongation were achieved using AZ31B without ECAP compared to an elongation of 261% for the specimens with 3-passes ECAP at strain rate of  $2.45 \times 10^{-4} \text{ s}^{-1}$  and test temperature of 623 K
6. The measured strain-rate sensitivity of  $m \approx 0.33$  suggests that deformation occurs through viscous glide control process.

**REFERENCES**

- [1] Abu-Farha, Fadi K. Khraisheh, Marwan K." Mechanical Characteristics of Superplastic Deformation of AZ31 Magnesium Alloy". Journal of Materials Engineering & Performance, Apr2007, Vol. 16 Issue 2, p192.
- [2] Z. j. Wang, W. P. Jia, J. Z. Cui," Study on the Deformation Behavior of Mg-3.6% Er Magnesium Alloy "Journal of Rare Earths 2007, 25, 744–748.
- [3] Z. J. Wang, Q. X. Yang, J. Qiao " Phase structure of ZK60-1Er magnesium alloy compressed at 450 °C "Trans. Nonferrous Metals Society of China 2010, 20, s567.
- [4] Franchitti S., Giuliano G.,Palumbo G., Sorgente D.,Tricarico L."on the optimization of superplastic Free Test of an AZ31 Magnesium Alloy Sheet"Int J Mater Form (2008) Suppl 1:1067-1070
- [5] Ghiban B., Dumitru F.-D., Ghiban N., Saban R., Semenescu A., Marin M., Consideration regarding die design for equal channel angular extrusion. Proceedings "Intelligent Manufacturing& Automation: Focus on Interdisciplinary Solutions", Zadar, 20 – 23 octombrie 2010, Croatia, 0189-0190
- [6] FENG Xiao-ming, AI Tao-tao "Microstructure evolution and mechanical behavior of AZ31 Mg alloy processed by equal-channel angular pressing" Met. Soc. China 19(2009) 293–298
- [7] Annual book of ASTM Standard, section 3:03.01,E112 96. P. 229. American society of testing and material Philadelphia, PA, 2004.
- [8] R. B. Figueiredo, T. G. Langdon" Grain refinement and mechanical behavior of a magnesium alloy processed by ECAP" J Mater Sci, 45, pp.4827–4836, (2010).
- [9] F.A. Mohamed and T.G. Langdon" The transition from dislocation climb to viscous glide in creep of solid solution alloys" Acta Metall., 1974, vol. 22,pp. 779-88.