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Superplastic behavior of AZ31 processed by ECAP

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Abstract

Magnesium base alloys have reached an important development as structural materials the last years due to its good strength/weight relationship. On the other hand, superplastic forming has also acquired a technological interest in the last time, due to the possibility of achieving complex geometries in only one forming step, with competitive strain rates. In this sense, grain refining promotes grain boundary sliding mechanism, responsible of superplastic behavior in metallic materials. ECAP (Equal Channel Angular Pressing) processing introduce a severe plastic deformation without modifying the sample geometry, being an efficient way to grain refining. The objective of this work was to study the influence of ECAP processing on the superplastic behavior of an AZ31 Mg alloy. Samples in "as cast" conditions were processed by ECAP with 1, 2, 3 and 4 passes. Material obtained was characterized microstructurally, obtaining grain size and recrystallized fraction, for each condition. Microhardness measurements were done as well as high temperature tension tests for different test temperatures and initial strain rates. Grain refinement, recrystallized fraction and microhardness increased with the number of ECAP passes. Deformation to fracture improved with ECAP processing, reaching 250% for 350ºC and $1x10^{-4}$ sec^{-1}. Maximum stress decreased with number of passes, temperature and diminishing strain rate.

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Keywords: AZ31, Superplasticity, ECAP, hot tensile test, grain refinement

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1. Introduction

Nomenclature

AC	as cast
AZ31	AZ31 Mg alloy
ECAP	equal channel angular pressing
FSS	fine structure superplasticity
GBS	grain boundary sliding
GS	grain size
HCP	hexagonal closed-packed
HTT	hot tensile test
HV	Vickers microhardeness
LM	light microscopy
m	strain rate sensitivity coefficient
OES	optical emission spectrometry
SEM	scanning electron microscopy
SP	superplasticity
SPD	severe plastic deformation
SPF	super plastic forming
T	temperature
$\dot{\varepsilon}$	strain rate
ε	strain
ε_f	fracture strain
σ	stress
σ_{max}	maximum stress
σ_0	flow stress

Superplastic behaviour of metallic alloys has been an area of great interest and development in the last years, due to the technological relevance acquired by the process SPF applied to complex geometries components [Ha and Chang (1999)]. The SP is one of the various micromechanisms of high temperature deformation, which is characterized by an intense deformation with a very low flow stress, previous to the fracture. Metallic alloys present the FSS mode, where GBS is the controlling mechanism for superplastic deformation [Sherby and Wadsworth (1989)].

The superplastic behavior of an alloy is determined principally by T, $\dot{\varepsilon}$, GS and σ_0 [Kawasaki et al. (2009)]. Grain size refining presents a strong influence on optimum strain rate to obtain FSS, increasing this strain rate with grain size refining [Ha and Chang (1999)]. From a technological point of view, the challenge is to reach optimum microstructural features to obtain SP at high SR and low T, to be viable the forming process.

Nowadays, methods of SPD as ECAP are widely used to produce grain refinement [Kawasaki et al. (2009), Lee (2008), Rao et al. (2008)]. This technique allows reaching very high plastic deformations without changing the section of the processed samples. Indeed, due to the high hydrostatic pressure during processing, permits lower process temperature than other methods [Furukawa et al. (1996)]. The main consequence of SPD is the formation of very fine grains with high angle boundaries, very disoriented [Sun et al. (2002), Iwahashi et al. (1998), Terhune et al. (2002), Zhilyaev et al. (2005)], being these aspects responsible for the improvement in SP deformation of processed alloys. Nevertheless, the subgrains structure formed are thermally unstable and tend to grow quickly during the deformation at high temperature. This aspect limits the maximum forming temperature, among others.

Magnesium alloys are usually employed in “as cast” condition, due to its limited ductility at room temperature (ε_f<10%) with a brittle fracture [Sherby and Wadsworth (1989)] associated to its HCP structure. Nevertheless it was demonstrated that the ductility can be improved by grain refining [Chapman and Wilson (1962)]. This has motivated the study of Mg alloys forming in the last decade. The high specific strength of Mg alloys has motivated its use in structural elements in several industries as automotive, aeronautics/aerospace, shipbuilding [Liang (2012)]. ECAP processing has been applied to Mg alloys reaching SP deformations. However, generally it is necessary to apply previous steps of deformation by means of other processes like extrusion, rolling or drawing [Kang et al. (2008), Figueiredo and Langdon (2012)].

The objective of this work was to analyze the influence of ECAP processing of an AZ31 alloy in AC condition, on its superplastic behavior. Also, a procedure for Mg alloy ECAP processing with a T-die, that minimizes the defect level was also found, as the first step in the Mg alloys ECAP processing project.

2. Experimental Procedure

The base material was as ingot of AZ31 Mg alloy of 80 mm in diameter and 150 mm in length, in AC condition. On this material chemical composition was determined by means of OES, microstructural characterization was done using LM and SEM. Vickers microhardness was also measured.

Samples of 12x12x50 were extracted from the AZ31 ingot and were ECAP processed in a T-die. For this alloy is reported the use of L-dies with different angles [Kang et al. (2008), Figueiredo and Langdon (2012)]. However, according with Lu et al. (2012), a T-die introduces a more severe deformation, reducing the number of passes required to produce a refined structure. This is a relevant aspect related with the procesability of AZ31, taking into account its limitations with this respect [Kang et al. (2008)].

Initially, different processing conditions were evaluated up to optimize the ECAP procedure, minimizing the defects formation during forming. In final procedure, the die was heated up to 350ºC with a lubricated specimen located at the entrance channel, and maintained during 15 min. During ECAP the pressing speed was 15 mm/min. In Fig. 1 (a) is shown the T-die used for ECAP.

(a) (b)

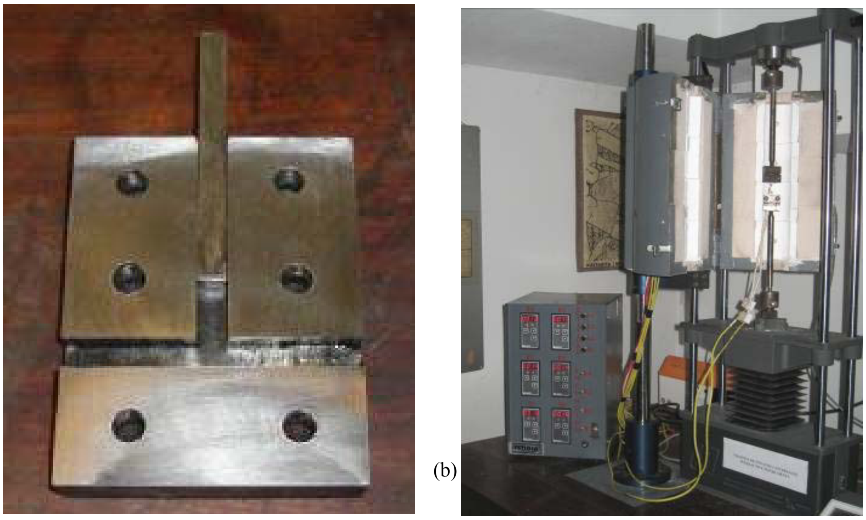

Fig. 1. (a) ECAP T-die; (b) hot tensile test machine.

With this procedure were ECAP processed samples with 1, 2, 3 and 4 passes, following a route B. From each processing condition metallograhic samples were prepared to analyze the microstructural evolution with the number of ECAP passes, using LM and SEM. Vickers microhardness was also measured. For conditions AC and 4 passes, “T-bone” tensile specimens were cut along the longitudinal direction with a gage length of 2,5 mm, which were tested at 300, 350 and 375 ºC, with different initial strain rates: $1x10^{-4}$, $5x10^{-4}$, $1x10^{-3}$, $5x10^{-3}$ y $1x10^{-2}$ sec^{-1}, in a universal testing machine II-1000 equipped with a furnace, as is shown in Fig. 1 (b).

On the head of a tensile specimen with 4 passes, tested at the lower strain rate (the highest time at temperature) it was analyzed the microstrucutre and it was compared with the initial one, with the aim of determine the evolution of it with the thermal cycle of the test.

3. Results and Discussion

Table 1 shows the chemical composition measured on the analyzed material.

Table 1. AZ31 Chemical Composition.

Al (%)	Zn (%)	Mn (%)	Fe (%)	Si (%)	Cu (%)	Mg (%)
2.57	0.84	0.19	0.02	0.01	0.02	Bal.

It can be seen, it is a Mg base alloy with near 3% Al and 1% Zn, according with the specification. In Fig. 2 (a) and (b) it is shown the microstructure in AC condition with LM and SEM, respectively. It has equaxial grains with a size of 150 microns approximately. Twins are also observed in some grains.

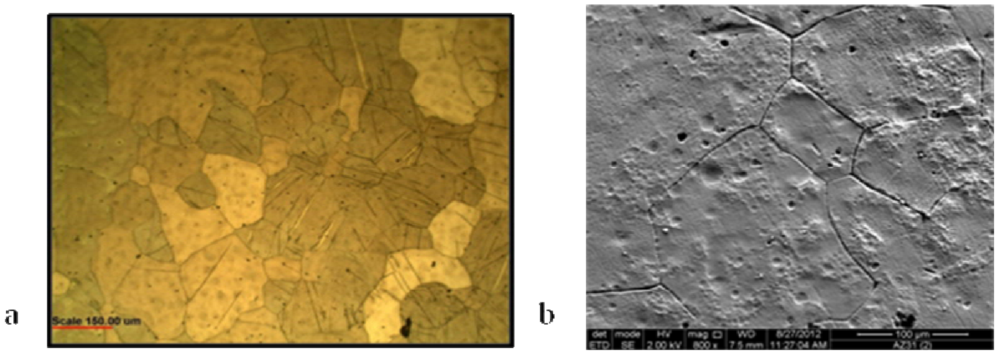

Fig. 2. Microstructure in AC condition: (a) LM, (b) SEM.

Figs. 3 (a)-(d) show the microstructures obtained for samples processed with 1, 2, 3 and 4 passes of ECAP, respectively.

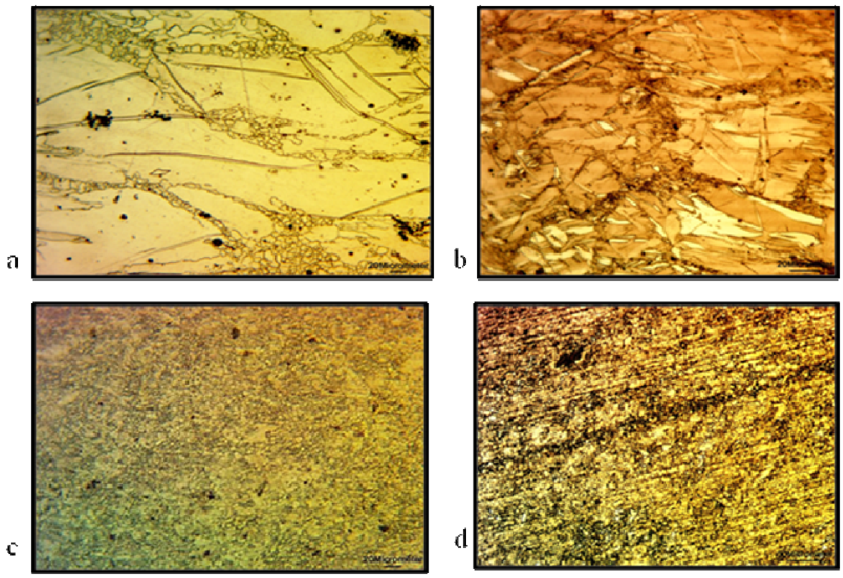

Fig. 3. Microstructures of ECAP processed samples: (a) 1 pass, (b) 2 passes, (c) 3 passes and (d) 4 passes.

After the first pass of ECAP it can be observed a mixed microstructure with deformed big grains and zones with very fine grains, which locate in the original grain or twin boundaries. After the second pass of ECAP it can be seen a very similar microstructure with more severely deformed grains and analogous grain refined fraction. For three ECAP passes, the obtained structure is uniform and almost completely refined, as well as for four ECAP passes. In Table 2 is shown the grain sizes of each zone and the fraction of refined zone measured after each pass. For deformed grains, the grain sizes show in the Table 2 represents the length of the shorter side of the measured grains.

Table 2. Grain size for different conditions.

Nº passes	GS Def. (μm)	GS Rec. (μm)	Refined fraction (%)
AC	-	150	0
1	46.7	8.3	34
2	45.8	5.3	35
3	-	3.3	95
4	-	3.1	96

It can be seen that in the first pass, the refined fraction is low, but with a strong refinement from the initial grain size. With subsequent passes la refined fraction is increased and an additional refinement is achieved. For three passes 95% of the area is refined and the grain size reaches almost 3 microns. With the fourth pass no longer significant benefits are obtained. This kind of evolution with the number of ECAP passes has been observed for this alloy by Feng and Ai (2009) using L-die ECAP.

Fig. 4 shows the evolution of microhardness with the number of ECAP passes.

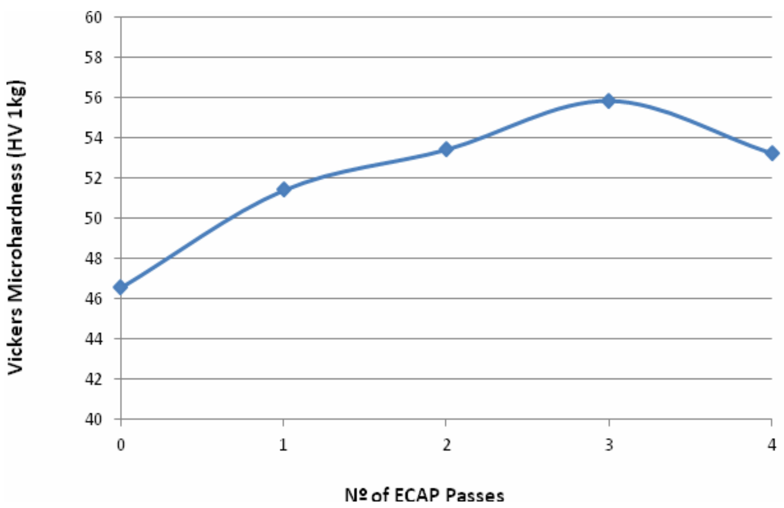

Fig. 4. Evolution of Vickers microhardness (HV) with ECAP passes.

Microhardness increases slightly with the number of passes from 46 HV for AC condition up to 55 HV for three passes. These results are consistent with previous works [Kang et al. (2008)]. In passes 1 to 3 it could be a strain hardening of non-refined grains and in the 3-4 passes it could be a grain refinement hardening, according with the metallographic observations.

In Table 3 are presented the HTT results (ε_f, σ_{max}) for AC and 4 conditions, tested at different temperatures and strain rates.

Table 3. Hot tensile test results for conditions AC and 4.

Nº Passes	T *[ºC]*	$\dot{\varepsilon}$ *[1/s]*	ε_f *[%]*	σ_{max} *[MPa]*
AC	300	0.0001	69	21
AC	300	0.0005	77	21
AC	300	0.001	83	34
4	300	0.0005	134	22
4	300	0.001	160	32
4	300	0.005	124	40
4	300	0.01	116	47
AC	350	0.0001	100	19
AC	350	0.0005	100	20
AC	350	0.001	103	27
4	**350**	**0.0001**	**244**	**10**
4	350	0.0003	233	14
4	350	0.0005	182	23
4	350	0.001	158	18
4	350	0.005	113	24
4	350	0.01	128	24
4	375	0.0001	140	7
4	375	0.0005	103	15
4	375	0.001	169	15

In general, condition 4 presents a higher ε_f as well as a lower σ_{max} than AC, for the same temperature and initial strain rate. The best results were obtained for 350ºC and $1x10^{-4}$ seg^{-1}, reaching a deformation at fracture of 244% and a maximum stress of 10 MPa. In Fig. 5 is shown the influence of $\dot{\varepsilon}$ on the ε_f at 350ºC for AC and 4 conditions.

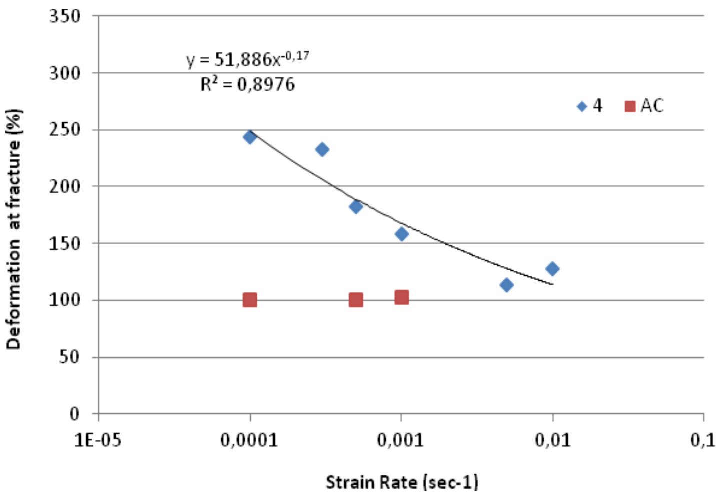

Fig. 5. Fracture strain - Strain rate at 350 ºC, for AC and 4 conditions.

As was mentioned before, for the lower $\acute{\varepsilon}$, ε_f for condition 4 was 2.5 times higher than AC one. This condition was not affected for the strain rate, while condition 4 showed a strong decreasing of ε_f with strain rate. An experimental expression that estimates the mentioned relationship with a good correlation level was obtained. For AZ31 processed with ECAP, with an extruded initial condition, it has been reach higher deformations to fracture [Figueiredo and Langdon (2012)], for AC initial condition this results are satisfactory.

In Fig. 6 are shown the samples processed with 4 passes of ECAP and tested at 350ºC with different initial strain rates.

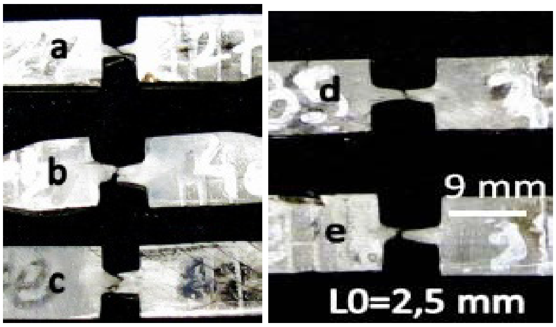

Fig. 6. Samples 4 tested at 350ºC: (a) 0.01 sec^{-1}; (b) 0.001 sec^{1}; (c) 0.005 sec^{-1}; (d) 0.0005 sec^{-1}; (e) 0.0001 sec^{-1}.

It could be noted higher fracture deformation and homogeneity of deformations as the strain rate decrease. This fact could be associated to a high strain rate sensitivity coefficient (*m*) [Sherby and Wadsworth (1989)].

In Fig. 7 can be observed the evolution of σ_{max} with $\acute{\varepsilon}$, for 4 conditions, at the different temperatures analyzed.

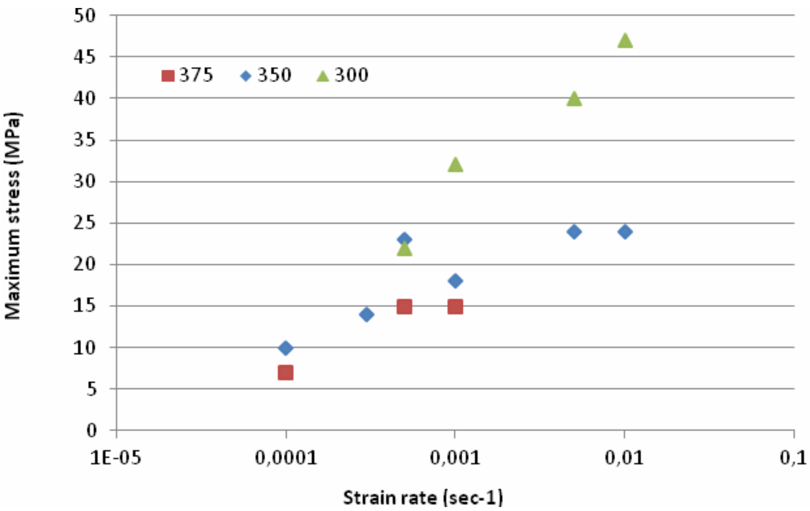

Fig. 7. Maximum Stress –Strain rate for different temperatures, for condition 4.

The slope of these curves is associated with the *m* value. Generally, this slope decrease with the increase of the strain rate, becoming temperature independent for lower strain rates, reaching values closed to 0.5, consistent with a mechanism of grain boundary sliding [Figueiredo and Langdon (2012)]. Additionally, as temperature increase, the strain rate at which the slope starts to change, decreases. This observation could explain the better performance at low strain rates of the processed material, consistently with a higher value of *m*. Nevertheless, for 300ºC the diffusion would be insufficient to produce the material flow.

Fig. 8 shows the microstructure in the undeformed zone (head) of a HTT sample of 4 condition, before and after testing at 350ºC and $1x10^{-4}$ sec^{-1}.

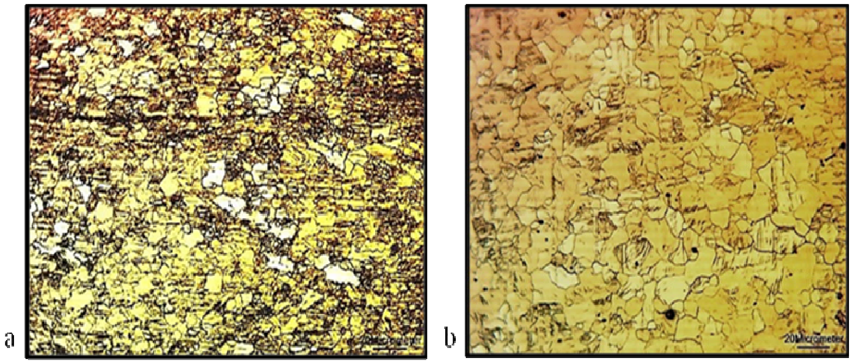

Fig. 8. Microstructure of sample 4: (a) before test; (b) after HTT at 350ºC and $1x10^{-4}$ seg^{-1}.

It can be seen that, as a consequence of permanence during almost 7 hs at 350 ºC, a normal grain growth was produced, related with a limited thermal stability of the refined structure obtained after ECAP processing [Figueiredo and Langdon (2012)]. This fact can affect the extent of superplastic deformation of the processed material.

4. Conclusions

In the present work, an AZ31 Mg alloy was processed by ECAP from an AC condition, with 1 to 4 passes. The obtained microstructure and its superplastic behavior were analyzed by HTT at different temperatures and initial strain rates.

It was developed a procedure to ECAP processing this alloy with a T-die, defining 350 ºC and 15 mm/min as the operative parameters.

The refined fraction increased and the grain size decrease with number of ECAP passes up to reach 95% and almost 3 microns, respectively, with 3 passes. The Vickers microhardness increased slightly with the number of ECAP passes up to 3.

Deformation at fracture increased when strain rate decreased, while maximum stress decreased. Related to temperature, the best performance was observed at 350ºC. Superplastic behavior for 4 ECAP passes reach the maximum deformation at fracture (250%) at 350ºC and $1x10^{-4}$ sec^{-1}, being 2.5 times higher than that obtained for the AC condition. For low strain rates, *m* values are close to 0.5, consistently with a grain boundary sliding mechanism.

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