Processing magnesium alloys by severe plastic deformation

Roberto B Figueiredo¹, Maria Teresa P Aguilar¹, Paulo Roberto Cetlin², Terence G Langdon³,⁴

¹Department of Materials Engineering and Civil Construction, School of Engineering, Universidade Federal de Minas Gerais, Belo Horizonte 31270-901, Brazil
²Department of Mechanical Engineering, School of Engineering, Universidade Federal de Minas Gerais, Belo Horizonte 31270-901, Brazil
³Materials Research Group, Faculty of Engineering and the Environment, University of Southampton, Southampton SO17 1BJ, U.K.
⁴Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, CA 90089-1453, U.S.A.

E-Mail: figueiredo@demc.ufmg.br

Abstract. The use of severe plastic deformation techniques for processing magnesium alloys has moved from the early difficulties of processing to a stage of tailoring the best properties of these materials. The present paper reviews processing, structure and mechanical properties characterization. It is shown that ultrafine-grained structures are obtained in magnesium alloys processed by multiple passes of Equal-Channel Angular Pressing at moderate temperatures. Ultrafine-grained structures are also obtained by room temperature processing by High-Pressure Torsion. The ultrafine-grained structures increase strength and introduce excellent superplastic capabilities in many magnesium alloys. Moreover, processing magnesium alloys by severe plastic deformation leads to the development of anisotropy in mechanical behavior.

1. Introduction
There is an increasing interest in improving forming capability and mechanical properties of magnesium alloys for use in structural applications. Grain refinement is a promising way to improve both. Severe plastic deformation [1] techniques have proven to be effective processing for achieving significant grain refinement in metallic materials down to the sub-micrometer range. The grain structures obtained by severe plastic deformation are finer than those obtained in conventional thermo-mechanical processing. Among the severe plastic deformation techniques Equal-Channel Angular Pressing (ECAP) [2] and High-Pressure Torsion (HPT) [3] are the most known. Ultrafine-grained structures are readily obtained in metallic materials processed by severe plastic deformation (SPD) at low temperatures. However, processing magnesium alloys by SPD is not trivial due to the limited ductility of these alloys.

The early attempts to process pure magnesium and magnesium alloys by ECAP were carried out at high temperatures and the grain refinement was not satisfactory [4]. Later it was found that the use of back-pressure [5,6], increasing the die angle [7,8], introducing an intermediate processing step by extrusion [9-16] or gradually decreasing the ECAP temperature [17,18] allow processing these alloys at low and moderate temperatures and produce significant grain refinement. A model for grain refinement of magnesium processed by ECAP was proposed [19,20]. Recently, magnesium alloys...
have been processed by HPT [21-26] as well. The hydrostatic pressure allows processing of this material at low temperatures without cracking and ultrafine grains are readily attained.

The grain refinement produced by SPD leads to significant changes in mechanical properties of magnesium alloys. Provided the ultrafine grains are stable at high temperatures, superplasticity is observed. Also, the ultrafine grains increase hardness of the alloys and the texture developed leads to anisotropic behavior in both tension and compression.

The present paper reviews the processing, structure and mechanical properties characterization of pure magnesium and magnesium alloys subjected to severe plastic deformation by ECAP and HPT.

2. ECAP processing and grain refinement

Equal-Channel Angular Pressing is a severe plastic deformation technique in which a billet is pressed through two intersecting channels of equal cross-section. Shear strain takes place in the intersection of the channels. Processing pure magnesium and magnesium alloys by ECAP at room temperature leads to cracking of the billets. Therefore, in order to avoid cracking the processing temperature is increased. Figure 1 shows the appearance of a billet of as-cast commercially pure magnesium partially pressed by ECAP using a die with 90° between channels at 523 K [27]. The billet exhibits some inhomogeneities at the top surface but no cracks were developed. The initial coarse grain structure with average grain size of ~1 mm was significantly refined as shown in Figure 1. Many small grains are formed between some coarse grains in a necklace pattern. Attempts to process this material at lower temperatures failed.

Commercial magnesium alloys may be pressed at lower temperatures provided they are processed after extrusion. Extruded rods of AZ31 [20] and ZK60 [16] alloys have been processed at 200 °C without cracking. Figure 2 shows the evolution of the grain structure in the extruded AZ31 alloy (a) before and after (b) 2 and (c) 4 passes of ECAP at 200 °C using a die with 110° between channels [20]. It is observed that the extruded alloy already exhibits a fine grain structure with average grain size of ~15 µm despite the presence of some coarser grains with tens of microns. The structure is significantly refined after 2 passes of ECAP but some coarse grains are still observed surrounded by small grains. The grain structure is completely refined after 6 passes.

Figure 1. Appearance of a billet of commercially pure magnesium partially processed by ECAP at 523 K using a die with 90° between channels (left). Grain structure of the billet after processing by ECAP (right) [27].
3. Model for grain refinement

The conventional model for grain refinement of f.c.c. materials processed by ECAP [28] does not apply for magnesium alloys because dynamic recrystallization takes place in the temperature range that magnesium is usually processed by ECAP (~420 – 600 K) [29-32]. Dynamic recrystallization in magnesium alloys occurs in such a way that new grains nucleate at grain boundaries and twin boundaries of old grains. This leads to a necklace distribution of fine grains around coarse grains. Thus, a multi-modal distribution of grain size is expected at the early stage of deformation of magnesium alloys. This is observed in Figure 1 and in Figure 2b where fine grains surround coarse grains. This multi-modal distribution of grain sizes will be replaced by a homogeneous distribution of fine grains when the cores of the initial coarse grains are consumed by the new fine grains. This is observed after 6 passes of ECAP in the AZ31 alloy in Figure 2c.

In order to address the unique grain refinement observed in magnesium alloys processed by ECAP, a model is shown in Figure 3 [20]. The model considers different initial grain structures from coarse (top) to intermediate (middle) to fine (bottom). The model also considers the possible difference in size of the newly formed grains and the amount of strain imposed. The second column corresponds to the initial passes of ECAP (low strain) while the third column corresponds to multiple passes (high strain). It is shown that a multi-modal distribution of grain sizes is observed when the initial grain size is significantly larger than the size of the new grains. This situation is shown in rows 1, 2 and 3. A homogeneous distribution of fine grains is readily obtained in the early passes of ECAP when the initial grain size is sufficiently small to be totally consumed by the new grains. This condition is shown in the bottom row.

In practice, processing magnesium alloys from the as-cast or annealed conditions, in which the grain size is large, leads to the grain refinement described in rows 1 and 2. The top row corresponds to the situation in which processing is carried out at low temperatures and high strain rates and the new grains are very fine. This situation is associated with a high tendency for deformation localization along grain boundaries and cracking of the billet. The second row corresponds to the situation where processing is carried out at high temperatures and the new grains are not very small. Intermediate processing of magnesium alloys by extrusion, rolling or ECAP might produce minor grain refinement leading to initial grain sizes described in rows 3 and 4. The magnesium alloys with these initial grain sizes may be processed at moderate temperature leading to very small final grain sizes.

Figure 2. Grain structure of the AZ31 alloy (a) in the extruded condition and after (b) 2 and (c) 6 passes of ECAP at 200 ºC [20].
4. Superplasticity in magnesium alloys

Provided the refined grain structure produced by ECAP is stable at high temperatures, magnesium alloys exhibit excellent superplastic properties. Superplastic elongations in the range of 1000% were observed in the AZ31 alloy processed by ECAP and tested at 623 – 673 K at ~10^{-4} \text{s}^{-1} \[6,33-35\]. Elongations even higher are observed in the ZK60 alloy. Lapovok et al. \[36,37\] reported an elongation of 2040% in this alloy after processing by rolling and ECAP. A record elongation of 3050% for magnesium was observed after extrusion and 2 passes of ECAP in the ZK60 alloy \[38\]. Figure 4 shows the appearance of the specimens tested after different numbers of passes of ECAP. Superplastic elongations are observed after any number of passes of ECAP in this alloy. However, the maximum is observed after 2 passes only. It was shown \[38\] that the material processed by 2 passes exhibits a lower rate of hardening during superplastic deformation which leads to a sustainable deformation up to larger amounts of strain. Later, it was shown that ECAP processing also leads to high strain rate superplasticity in the ZK60 alloy provided some processing conditions are controled \[39\].

**Figure 3.** A model for the grain refinement of magnesium alloys processed by ECAP: the left column shows the initial condition, the second column shows the structure after one pass and the third column shows the structure after multiple passes; the upper two rows show the same starting structure with different processing parameters and the third and fourth rows show different starting structures \[20\].
An analysis of the data for flow stress, strain rate, temperature and grain size during superplastic deformation of the magnesium alloy AZ31 [33] showed good agreement with the model for grain boundary sliding deformation [40]. Figure 5 shows a plot of strain-rate compensated for temperature and stress as a function of the inverse of the grain size. The data agree well with the predicted line for grain boundary sliding superplasticity and shows the exponent for the inverse of the grain size in the creep equation is equal to 2. This confirms the superplastic deformation observed in magnesium alloys processed by ECAP can be predicted by the general equation for grain boundary sliding superplasticity. Thus, it is expected that decreasing the grain size leads to an increase in the strain rate for superplasticity or a decrease in the temperature at which superplasticity is observed.

4. Mechanical properties

The grain refinement introduced by severe plastic deformation usually leads to an increase in strength of metallic materials. For example, the hardness of the ZK60 magnesium alloy increases from ~72 Hv in the extruded condition to 87 Hv, 90 Hv, 85 Hv and 88 Hv after 1, 2, 4, 6 and 8 passes of ECAP, respectively [41]. It was shown by X-ray profile analysis that the evolution of hardness agrees well with the evolution of dislocation density and an average size factor that incorporates the effects of twin and subgrain boundaries [42]. However, it was also shown that the increase in hardness is not accompanied by an increase in tensile strength. The tensile behavior actually shows decreased strength and improved ductility [41]. This is attributed to a texture effect which was also observed in an AZ31 alloy [43]. It was shown that the tensile behavior depends strongly on the testing direction after the material is processed by ECAP.

The anisotropy in mechanical behavior of magnesium alloys processed by ECAP is also observed during compression testing. Figure 6 shows stress vs. strain curves determined along orthogonal directions of the AZ31 alloy in (a) the extruded condition and after ECAP processing by (b,c) 2 and (d,e) 4 passes. The processing route was (b,d,e) B and (c) C and the ECAP die angle was (b,c,d) 90º and (e) 135º [44]. The testing was carried out parallel to the pressing direction (X-direction), from top to bottom (Z-direction) and along the through thickness direction which is perpendicular to the ECAP channels (Y-direction). It is shown that the magnesium alloy exhibits significant anisotropy before and after ECAP processing and this anisotropy depends on processing route and die angle. The twinning signature is clearly observed when the sample is compressed along the Y-direction after ECAP.

Figure 4. Appearance of specimens of ZK60 alloy pulled to failure at 473 K at 10^{-4} s^{-1} after processing by ECAP for different numbers of passes: the upper specimen is untested [38].
Figure 5. Strain-rate compensated for temperature and stress plotted as a function of the inverse of the normalized grain size for the AZ31 alloy [35].

Figure 6. Stress vs. strain curves determined by compression tests along the billet axial direction, X, along the through-thickness direction, Y, and along the top-bottom direction, Z, in the magnesium alloy in the (a) as-received condition and after processing by 2 passes of ECAP through route (b) B_C and (c) C and after 4 passes of ECAP using dies with (d) 90º and (e) 135º between channels [44].
5. High-Pressure Torsion of magnesium

Samples in the shape of thin discs are pressed and subjected to torsion in the process of High-Pressure Torsion (HPT). The compression load leads to high compressive hydrostatic stresses in the sample which prevent cracking. This allows processing magnesium and its alloys at low temperatures without cracking. The low temperature deformation prevents dynamic recrystallization and leads to pronounced grain refinement and strain-hardening. However, recent reports have shown that deformation localization might take place in magnesium alloys processed by HPT [23,25,26]. The grain structure and hardness distribution varies along the thickness of the sample.

Pure magnesium has been processed by HPT leading to an average grain size of 1 µm [24]. Commercially pure magnesium and the AZ31 alloy were processed by 10 turns of HPT at room temperature and the resulting structure is shown in Figure 7 [45]. The average grain size of the CP-Mg is ~1.46 µm and for the AZ31 it is ~0.2 µm. This shows that HPT is able to produce ultrafine grained structure in magnesium alloys with average grain sizes smaller than in ECAP.

![Figure 7. Grain structure of the (a) CP-Mg and (b) the AZ31 alloy processed by 10 turns of HPT at room temperature [45].](image)

The pronounced grain refinement and the strain-hardening introduced by HPT leads to a significant increase in hardness of the magnesium alloys. The average hardness observed in CP-Mg is 45 Hv and the hardness observed in the AZ31, AZ91 and ZK60 alloys are 120 Hv, 131 Hv and 123 Hv, respectively [45]. These values of hardness are much higher than the values observed after conventional thermomechanical processing and even after ECAP. These values are in the range of hardness observed in special laboratory alloys. Table 1 summarizes values of hardness observed in magnesium and its alloys after HPT and other processing techniques.

5. Summary and conclusions

1. Magnesium and its alloys can be processed by ECAP at high temperatures and can be processed at moderate temperatures provided the initial grain structure is partially refined. The grain structure can be fully refined by ECAP.

2. The mechanism of grain refinement during ECAP processing of magnesium alloys differs from the mechanism in f.c.c. metals. A model is proposed and it incorporates the effect of initial grain structure, size of the newly formed recrystalized grains and the amount of strain imposed.
3. Excellent superplastic elongations are observed in magnesium alloys processed by ECAP including a record elongation for a magnesium alloy of >3000%. The rate-controlling mechanism agrees with the theoretical prediction for grain boundary sliding.

4. The grain refinement introduced by ECAP leads to improved hardness but the texture developed produces anisotropic behavior in tension and compression.

5. The hydrostatic stresses developed in HPT prevent cracking and allow processing magnesium alloys at low temperatures leading to finer grain sizes and enhanced hardness.

**Table 1.** Summary of peak hardness developed in pure magnesium and Mg alloys processed by different techniques [45].

| Alloy | Hv [kgf/mm²] | Processing History | Reference |
|-------|-------------|--------------------|-----------|
| Mg    | 45          | HPT                | [45]      |
| AZ31  | 120         | HPT                | [45]      |
| AZ91  | 131         | HPT                | [45]      |
| ZK60  | 123         | HPT                | [45]      |
| Mg-9 Al | 120     | HPT                | [21]      |
| ZK60  | 90          | ECAP               | [42]      |
| AZ31  | 87          | ECAP               | [5]       |
| AZ31  | 91.3        | cold drawn         | [46]      |
| ZK60  | 85          | Friction stir welding | [47]   |
| AZ31+SiC particles | 75 | Friction stir processing | [48] |
| ZK60  | 70          | Age-hardening      | [49]      |
| ZK60+Y | 82       | Age-hardening      | [50]      |
| Mg-2 Gd-1.2 Y-0.2 Zr | 130 | Age-hardening    | [51]      |
| Mg-12 Gd-3 Y | 147  | rolling + Age hardening | [52] |
| Mg-5 Y-2.5 Zn | 103.3 | LPSO phase        | [53]      |
| Mg-6.5 Gd-2.5 Dy-1.8 Zn | 142 | LPSO + Age-hardening | [54] |
| Mg-2 Zn-4 Y-0.004 Sr | 123 | LPSO phase + ECAP | [55] |

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