RESEARCH ARTICLE OPEN ACCESS

Microstructural Analysis of Commercially Pure Magnesium Processed by ECAP at Room Temperature

Gustavo B. Ferraz [1], Rodrigo E. Pereira [2]
Jefferson Fabrício C. Lins [3], Fabiano C. Ferraz [4]
Department of Metallurgical Engineering [1] & [2]
Federal Fluminense University, Volta Redonda, RJ, Brazil.
Department of Materials and Technology [3] & [4]
Guaratinguetá Engineering College, Guaratinguetá, SP
Brazil

ABSTRACT

In this paper, the evolution and distribution of the deformation structures and the hardness of pre homogenized commercially pure magnesium were analyzed after a severe plastic deformation processing called ECAP (equal channel angular pressing). The tests were performed at room temperature and the heat treatment was done to optimize the process in this condition, trying to avoid the appearance of defects such as cracks and fissures. To perform the analyzes were used: optical microscope to obtain the microstructure after processing, and a hardness map, in order to compare the evolution of the mechanical properties.

***Keywords* :-** Magnesium, ECAP, Severe Plastic Deformation

I. INTRODUCTION

Due to its low density (1.74 g/cm3) and relatively high specific strength, magnesium has attracted researcher's attention, offering the possibility of reducing structural weight by replacing steel and aluminium parts in the transportation industry [1]. Although magnesium and its alloys have a remarkable potential in this context, the application is limited because of poor deformability and limited ductility at room temperature. Therefore plastic deformation processes are generally carried out at elevated temperatures, usually between 473-673K [2].

The SPD (severe plastic deformation) techniques differ from conventional processing operations, since the plastic deformation is introduced without changes in the cross-section of the samples. Thus, the process can be repeated several times by accumulating large amounts of deformations with the refinement of the structure. The process known as Equal Channel Angular Pressing (ECAP) is the most common SPD technique, responsible for conducting intense refining due to grain fragmentation, which can reach nanometric granulometry [3]. However, ECAP processing of pure Mg at high temperatures may result in grain growth [4]. This will reduce the efficiency of SPD techniques in grain refining. Consequently, several studies have been done to enable the processing of magnesium alloys at low temperatures and some techniques are recommended, such as reducing the rate of processing deformation, decreasing the introduced strain by passage and applying back pressure [5].

Given the HC structure, the Mg usually breaks down in the ECAP process at room temperature. In this study, a magnesium sample underwent a pre-homogenization heat treatment in order to optimize the processing, reducing or eliminating the presence of these cracks and fissures.

II. EXPERIMENTAL

2.1 Material

From the magnesium in the crude state of fusion, specimens measuring 10x10x50 mm were prepared for ECAP processing, called the "as received" sample. A pre-homogenization heat treatment was performed at 415 °C for 24 hours and then cooled in water. The sample came to be called "homogenized". The chemical composition is expressed in Table 1.

TABLE I

Chemical Composition of Metallic Magnesium (Contents Expressed in% Weight).

Elements	%Zn	%Cu	%Fe	%Mn	%Mg
Sample	0,009	0,004	0,002	0,06	99,8

2.2 Methods

2.2.1 Processing by ECAP:

The specimens were processed in a system consisting of a mechanical test machine EMIC, model 23-600, with a maximum capacity of 600 KN. A two-part H13 tool steel die was used with two identical channels having dimensions of approximately 10 x 10 mm. The angle between these channels of the die is $\phi = 90°$ and arc of curvature is about 37°. The samples were pressed at a rate of 5 mm/min at room temperature, causing a equivalent true strain of $\varepsilon = 1.19$. Fig. 1 illustrates the layout of sections of the sample processed after ECAP. In this work the analysis was performed in the longitudinal sections of the samples (ND-PD).

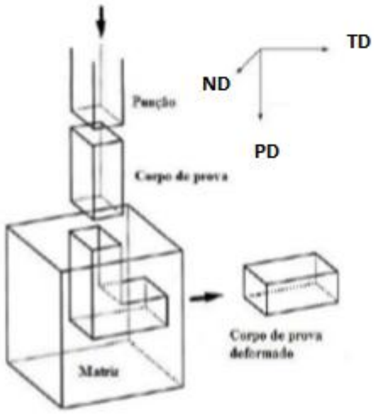

Fig 1: Schematic illustration of the representation of sample sections. PD - Press direction, TD - transverse direction and ND direction normal to the plane [6].

2.2.2 Microhardness:

The mechanical Vickers hardness test was performed in order to determine the hardness before and after ECAP processing. The assay was performed on a Shimadzu microdurometer model HVM-2T. 225 measurements were performed distributed along the longitudinal section of each sample in the form of a 15 x 15 mesh. The assay load was 0.1 kgf and the penetration time was 30 seconds. Fig. 2 illustrates the layout of the printing grid for performing hardness measurements.

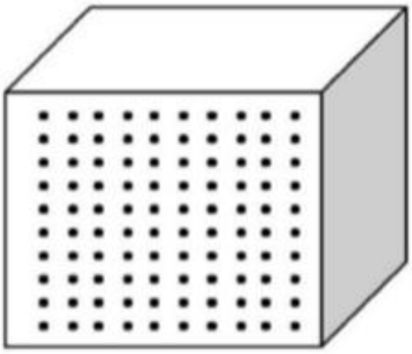

Fig 2: Printing grid scheme to perform the hardness measurements in each sample.

2.2.3 Metallographic Preparation:

The metallographic preparation consists of the following steps: sanding, polishing and chemical attack. The sanding was done with silicon carbide sanding: 180, 400, 800, 1000 and 1200. The samples were embedded with phenolic resin. Subsequently, for mechanical chemical polishing, a solution was used with 250 ml of distilled water, 15 ml of colloidal silica (MasterMet), 0.5 ml of nital (5%) and 5 ml of detergent. After polishing, the sample surface was chemically attacked with a solution of 5% nitric acid and 95% ethanol in a water-ice bath to reveal the microstructure.

2.2.4 Microscopy:

The images of this work were obtained using a NIKON LV150 optical microscope and the "Stream Basic 8.1" image capture software.

III. RESULTS AND DISCUSSION

3.1 Microstructural Characterization

Fig. 3 depicts the microstructures of commercially pure magnesium in the "as received" (a) and pre-homogenized condition (b). An average grain size of more than 300 μm was estimated for both structures. It was possible to observe the presence of twinning inside the grains for the two samples.

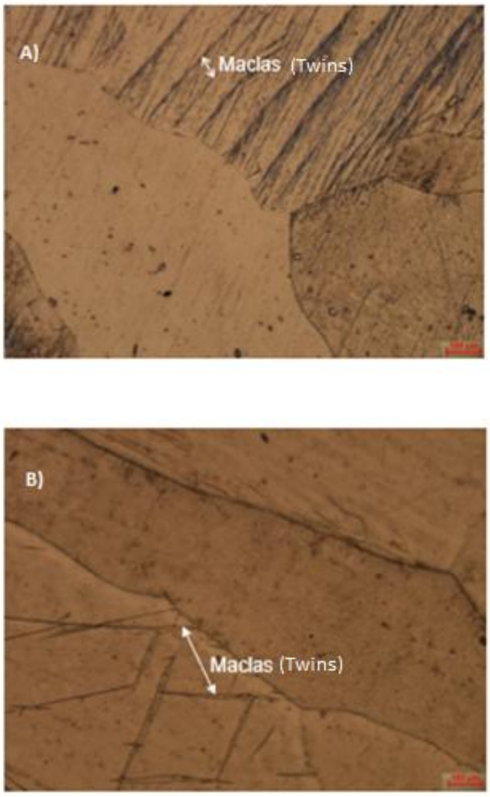

Fig 3: Microstructures of commercially pure magnesium in the "as received" (a) and pre-homogenized (b).

The magnesium sample in the "as received" state after an ECAP pass presented a severely deformed structure with elongated grains in the shear direction, Fig 4. The formation of this structure was developed in the shear region of the die between the channels. The average thickness of these elongated grains is 12 μm and the average length is 208 μm. In the work on processing of commercially pure magnesium via ECAP at room temperature performed by Su, similar structures were found [7]. In the Poggialli study, the processing was done at 250 °C and the grains also extended towards the shear plane, but there was dynamic recrystallization and new refined grains were nucleated inside the twinnings and around the grain boundaries of the structure coarser grain [8].

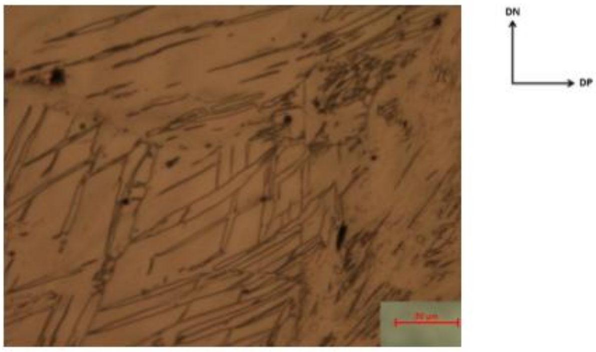

Fig 4: Microstructure of the magnesium in the state "as received" after an ECAP pass.

In Fig. 5, a significant amount of deformation twins are seen, which are bounded by grain boundary as highlighted in the image. These twins have an average length of 97 µm and average thickness of 3 µm. In general, this type of defect occurs mainly in metals with HC structure. Kwak, when characterizing the magnesium processed by ECAP, observed the same type of deformation found in this study [9].

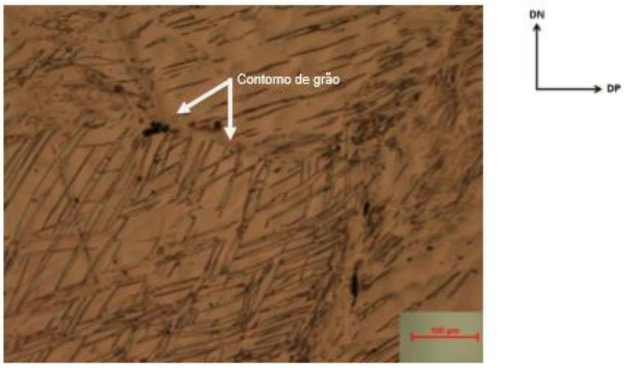

Fig 5: Microstructure of the magnesium in the state "as received" characterizing the deformation twins around a grain boundary.

It can be seen in Fig. 6 a structure with characteristics of a shear band, which crosses a section of at least 560 µm in the longitudinal plane ND-PD. The width of the shear bands found in this work for the "as received" samples were 20 µm, and are inclined at ~ 45 ° with respect to the extrusion plane. S. Suwas, explain this behavior by the preference of the grains to elongate in this direction or the consequence of the restricted crystallographic slip [10].

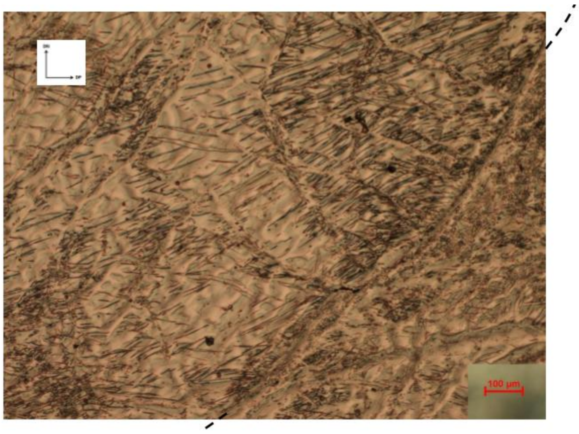

Fig 6: Microstructure of the magnesium in the state "as received" characterizing the deformation twins around a grain boundary.

The homogenized magnesium sample showed similar structures. However, it was possible to observe the presence of recrystallized equiaxed fine grains in some regions of the longitudinal section, as observed in Fig. 7. The average size of these grains is estimated to be 5 µm. Because the material has been hot-embossed, small recrystallization may have occurred within the deformation twins formed during extrusion. Another factor that supports this possibility is the formation of refined grains inside a shear band with a thickness varying from 10 to 17 µm, formed at ~ 45 ° from the extrusion axis, as can be observed in Fig. 8.

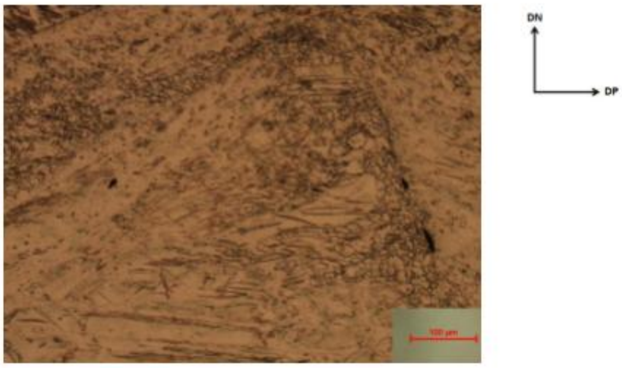

Fig 7: Microstructure of the homogenized magnesium sample after an ECAP pass.

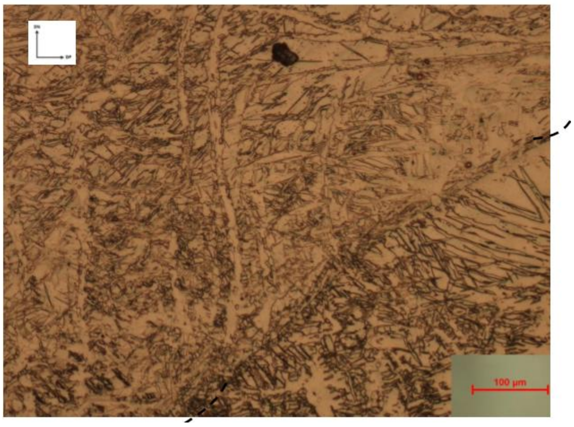

Fig 8: Microstructure of the homogenized magnesium after an ECAP pass characterized by shear bands.

3.2 Aspect of the Sample

Fig. 9 shows the magnesium sample “as received” shortly after ECAP processing. Fissures and fractures were observed for both samples. The cracks were at 45 ° from the longitudinal axis and were not sufficient to damage the transverse plane of the sample.

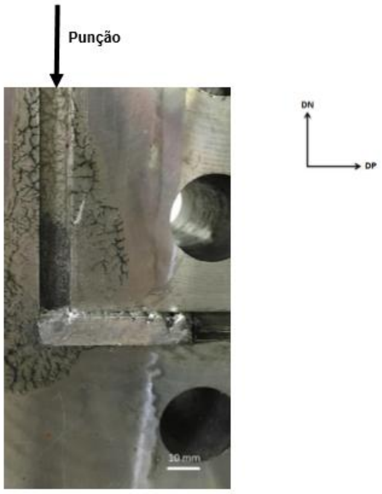

Fig 9: Sample of thermally homogenized magnesium after ECAP processing with cracks.

3.3 Microhardness Vickers

The hardness values found in the longitudinal section of the sample "as received" before being processed by ECAP ranged from 31 to 49 vickers, with an average value of 40 vickers and a standard deviation of 4. In the sample of homogenized magnesium in this same stage, the hardness ranged from 25 to 41 and an average value of 33 vickers with a standard deviation of 3. A more homogeneous behavior of the thermally treated sample was observed in relation to the magnesium sample "as received ", which can be seen on the map of hardness represented by Fig. 10.

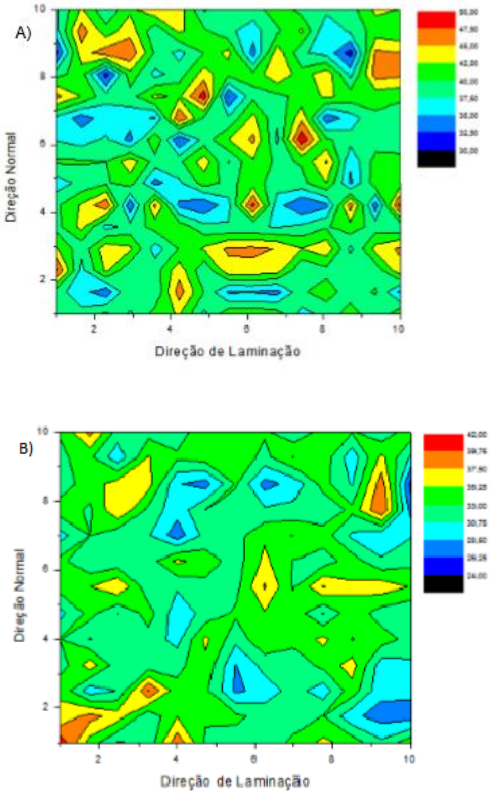

Fig 10: Hardness map for samples “as received” (a) and pre-homogenized (b) before ECAP.

After processing by ECAP, the magnesium sample “as received” presented a hardness variation of 38 to 58 and an average value of 49 vickers with a standard deviation of 3. For the homogenized sample, the hardness ranged from 32 to 53 and a value average of 46 vickers with a standard deviation of 3. This hardening after ECAP is attributed to the work hardening caused by the formation of subgrain bands and the increase in the density of dislocations arising with the shear strain within the initial grain [9]. Therefore, due to the greater measure of hardness obtained, it can be said that the magnesium in the "as received" state obtained a greater degree of work hardening in relation to the homogenized magnesium. Although not completely refined, it was possible to observe a better microstructural homogeneity of the samples after processing by ECAP, which can be verified by the hardness maps obtained, Fig. 11.

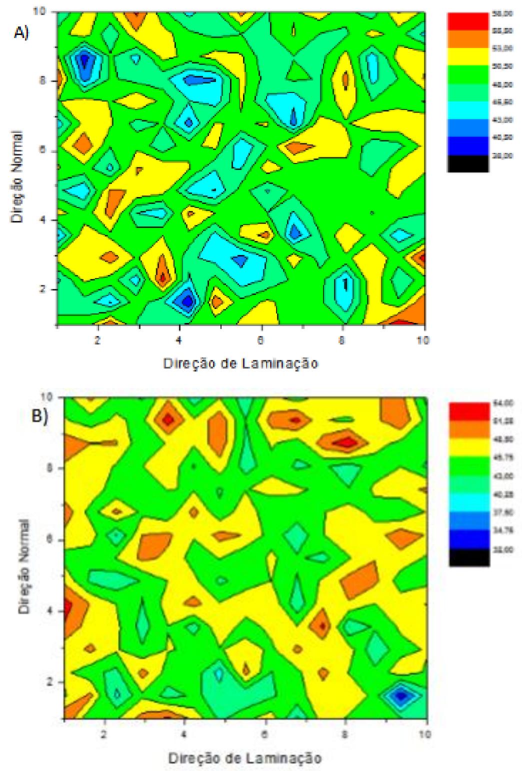

Fig 11: Hardness maps for the samples "as received" (a) and homogenized (b) after an ECAP pass.

IV. CONCLUSIONS

The In the obtained results, it was possible to observe the microstructural evolution and the mechanical properties of pure magnesium processed at room temperature. Therefore, the study found that the processing of pure magnesium under these conditions is not sufficient to render the structure completely refined, and that the heat treatment carried out did not cause any significant improvement in relation to the microstructure and mechanical properties, when comparing the homogenized sample with the sample "as received". However, it was possible to observe an increase in hardness and a better homogeneity after the process for both samples.

REFERENCES

[1] H. Friedrich, S. Schumann. (2001). *Journal of Materials Processing Technology*. pp. 276-281.

[2] YAMASHITA, A.; HORITA, Z.; LANGDON, T. G. (2001). *Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation*. Science and Engineering A, v.300, pp. 142-147.

[3] Valiev RZ, I. R. (2000). *Bulk nanostructured materials from severe plastic deformation. Progress in Materials Science*, pp. 103-189

[4] Weiwei Lei; Wei Liang; Hongxia Wang; Yanhao Sun. (2017). *Effect of annealing on the texture and mechanical properties of pure Mg by ECAP at room temperature.* Vacuum, V. 144, pp. 281-285

[5] N. Bayat Tork; N.Pardis; R.Ebrahimi. (2013). *Investigation on the feasibility of room temperature plastic deformation of pure magnesium by simple shear extrusion process*. Material Science and Engineering: A, v560, pp.34-39.

[6] Luri, R. P. (2006). *New configuration for equal channel angular extrusion dies.* Journal of Manufacturing Science and Engineering, v. 128, pp. 860-865.

[7] C.W.Su, L.Lu, & M.O.Lai. (2006). *A model for the grain refinement mechanism in equal channel angular pressing of Mg alloy from microstructural studies*. Mater. Sci. Eng., V. 434, 227-236.

[8] Poggiali, F. S., Figueiredo, R. B., Aguilar, M. T., & Cetlin, P. R. (2012). *Grain refinement of commercial purity magnesium processed by ECAP (Equal Channel Angular Pressing).* Materials Researsh. V.15, n.2, 312-316.

[9] Kwak, E. J., Bok, C. H., Seo, M. H., Kim, T.-S., & Kim, H. S. (2008). *Processing and Mechanical Properties of Fine Grained Magnesium by Equal Channel Angular Pressing.* Materials Transactions, Vol. 49, No. 5, 1006-1010.

[10] S. Suwas, G. Gottstein, R. Kumar, Mater. *Sci. Eng.*, A 471 (2007) 1 – 14.