STRUCTURAL CHARACTERIZATION OF ZK60 ALLOY SUBJECTED TO ECAP PROCESS - ROUTE A

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În ultimii ani, multe dintre eforturile de cercetare au fost orientate către extindea aplicațiilor aliajelor deformabile de magneziu la componentele structurale. Presarea unghiulară în canale egale este una dintre metodele deformării plastice severe care îmbunătățesc proprietăților mecanice ale materialelor prelucrate. Tehnica este capabilă de a rafina microstructurile unor materiale diferite. Presarea unghiulară în canale egale produce un grad de deformare semnificativ, fără a reduce secțiunea transversală a probei prelucrate. În această lucrare au fost analizate microstructura şi difracția de raze X a aliajului de magneziu ZK60 în stare primită, dar şi supus procesului de deformare plastică severă. Microstructura prezintă o rafinare a grăunților la creşterea gradului de deformare.

In the past years many research efforts have been made in order to extend the application of wrought magnesium to structural components. Equal channel angular pressing (ECAP) is one of the SPD´s methods that improve the mechanical properties of the processed materials. The technique is able to refine the microstructures of different materials. ECAP produces significant deformation strain without reducing the cross sectional area. In this paper the microstructure and XRD pattern of ZK60 magnesium alloy in the as-received state and with ECAP were analyzed. The microstructure shows a refinement of the grains with the increase in strain.

Kezwords: ZK60, ECAP, microstructure, XRD

1. Introduction

Due to increasing global awareness of the correlation between weight reduction, fuel economy and reducing harmful emissions the magnesium alloy research has seen a revival in the last years [1], because magnesium is one of the lightest material for manufacturing metal components [2]. The issues that limit their applications are the relatively low strength and ductility of magnesium alloys due to the hexagonal close packed structure [3].

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An important method to improve the mechanical properties of magnesium alloys is through grain refinement and lately, severe plastic deformation (SPD) techniques have been intensely investigated due to the fact that the structures formed by the SPD can provide an equitable compromise between high strength and satisfactory ductility being attractive enough for many structural applications.

The SPD methods developed and used are: accumulative roll bonding [4], severe torsion straining under high pressure [5], repetitive corrugation and straightening [6], multiple forging [7], twist extrusion [8], and equal channel angular pressing or extrusion [9].

Presently, among the methods of SPD, equal channel angular pressing (ECAP) is considered as the most promising for industrial applications.

In ECAP process, a metal billet is pressed through a die having two channels of equal cross-section intersecting at an internal angle, Φ [10], which is usually between 60º and 160º [11]. Another important feature of the die is the existence of an additional angle Ψ that defines the arc of curvature at the outer point of intersection of the two channels [12]. The ECAP die is manufactured with two channels that usually intersect at an angle with the same cross-section. The material is extruded through the die and it is mainly deformed by a shear mechanism combined with a high pressure, which exists within the die channels [13]. The exit channel of the die is usually manufactured with the same diameter as the entrance channel and hence cross-section of the processed material is not modified and so, there is no geometrical limitation to the deformations that is possible to impart to the processed materials [14].

The billet undergoes simple shear deformation when it is pressed through the intersecting corner. High plastic deformations lead to an improvement in the dislocations density in ductile crystalline materials and this improvement of the dislocation density is followed by an increase in the strength. Hence with enough accumulation of plastic strain a new structure of fine grains or even nanometer grain size replaces the former grain size [15].

Because the channels of the die are of equal cross section, the cross-sectional shape of the specimen does not change during the process. This enables the process to be repeated several times, with each run called a pass. There are several possible sequences of passes, or routes, which differ on the re-orientation of the specimen's shear plane relative to the die's shear plane [16].

During ECAP, the processing route is one parameter which can effectively change the strain path of the deformation. A nomenclature has evolved in the literature referring to the major variants as Route A - meaning no rotations between passes, Route B_A - meaning 90º back-and-forth rotations, Route B_C – meaning 90º continuous rotations and Route C meaning 180º rotations [17].

The amount of plastic strain introduced in the materials after ECAP processing depends on the die angle and the number of passes.

2. Experimental procedure

The material used in this study is a ZK60 magnesium alloy, with the chemical composition listed in Table 1.

Table1

Chemical composition of the ZK60 magnesium alloy (wt.%)

Mg	Zn	Zr	Al	Mn	Other elem.
bal	5.49	0.55	0.005	0.025	max 0.3

The as-received magnesium alloy was machined into square specimens with dimensions of 10 mm x 10 mm x 40 mm. The ECAP process was performed using a tool steel die with the internal angle between the two channels of 90° and an external angle of approximately 16°, having a strain of $\varepsilon = 1.07$ per pass. Back-pressure was used to ensure a uniform strain-stress distribution and to avoid failure during extrusion as reported in low ductile materials [18]. The samples were first lubricated with graphite before being inserted in the die. The ECAP was carried out following a 180° back-and-forward rotation of the billet, along the transverse axis, by rotating the die with 90° between each pass. According to Valiev et al. [19] this type of processing is however equivalent to route A. Prior to each pass, the billets were preheated to the respective temperature of ECAP in the die. The exact temperature of the ECAP was monitored through a thermocouple plugged to a multimeter placed exactly near the plane of intersection of the two channels. The extrusion speed of ECAP tests was 17.30 mm/s. The first 4 passes were conducted at 250 ± 5 °C. From the 5th pass onward, the temperature of the ECAP was reduced in steps. The 5th pass ECAP was carried at 200 ± 5 °C, the 6th pass at 150 ± 5 °C and the 7th pass at 110 ± 5 °C. For this investigation only the first four passes, where the processing temperature is the same (250 °C) will be analyzed. The ECAP specimens were cut into the middle and samples were collected from the center of each billet. The samples with and without ECAP, were cut in the direction of extrusion, were prepared according to etching and polishing standards to reveal the microstructure of the material, which was observed by Olympus BX51 optical microscope. The SEM images were captured with Philips scanning electron microscope equipped with EDAX, necessary to investigate the elemental composition of the alloy ZK60.

The X-ray diffraction was used to study the structural condition of the received ZK60 alloy, but also subjected to the route A of the ECAP process. The analysis was performed at room temperature on a diffractometer type D8 DISCOVER with an X-ray tube with copper cathode. The high voltage applied to generate X-ray was U = 40 kV.

Preparation of the samples for X-ray diffraction analysis consisted of mechanically polishing the analyzed samples with Grit 1200 sandpaper.

3. Results and discussion

The ZK60 magnesium alloy consists of primary matrix α (Mg) and the eutectic (solid solution α and MgZn and Zn_2Zr compounds), as shown in Figure 1. It can be noticed that the eutectic precipitates as a discontinuous lattice at the grain boundary.

From Figure 1, it can be seen that the microstructure of as-received ZK60 magnesium alloy is heterogeneous with large grains, reaching sizes of 80 μm, surrounded by fine grains (of approximately 1-2 μm).

To complement the results obtained from the scanning electron microscopy analysis, the X-ray diffraction was performed, as shown in Figure 2. For the as-received ZK60 alloy, the diffractogram shows a large number of peaks that belong to the 2 phases: magnesium-based solid solution (in which zinc and zirconium are dissolved) and the compounds MgZn and Zn_2Zr.

After the first pass a large grain refinement may be noticed, caused by dynamic recrystallization of the material during deformation and static recrystallization which may occur between passes (Figure 3a).

Along with a greater reduction in grain size, in the second and third pass (Figures 3b and 3c) an elongation of large grains in the extrusion direction can be seen. It should be noted that after 4 passes of ECAP (Figure 2d), some coarse grains remains still visible, which indicates that after the ECAP process the microstructure is not fully refined. A similar structure ZK60 alloy was reported by He et al. [21] after 8 passes of ECAP at 240 ° C.

X-ray diffraction analysis of ZK60 alloy subjected to ECAP process route A is shown in Figure 4. This analysis was evaluated for an 2θ angle between 30° and 80°, given that there were no identified peaks before and after these values. From the appearance of the XRD patterns of ECAP processed ZK60 alloy the same peaks as the received material can be observed, showing the composition of magnesium-based solid solution and the eutectic.

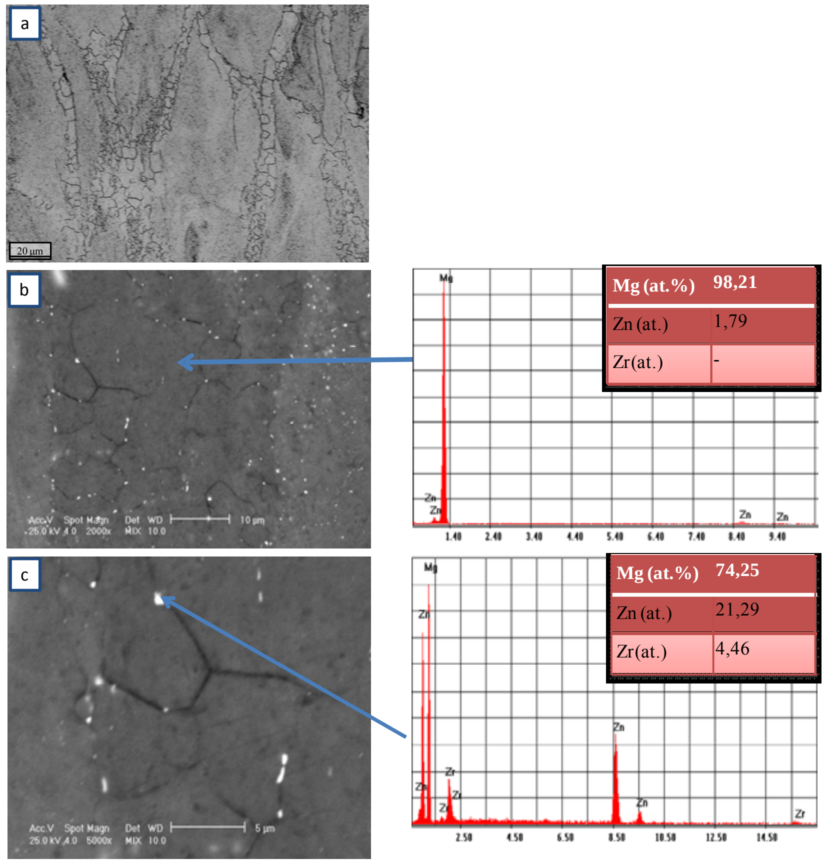

Fig. 1. (a) OM of as-received ZK60, (b) SEM image and EDS spectrum of α solid solution, (c) SEM image and EDS spectrum of β phase and local compositions of the identified phases

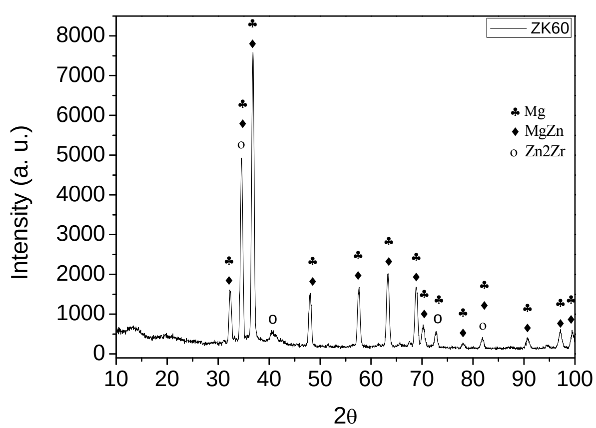

Fig. 2. X-ray diffraction of as-received ZK60 alloy

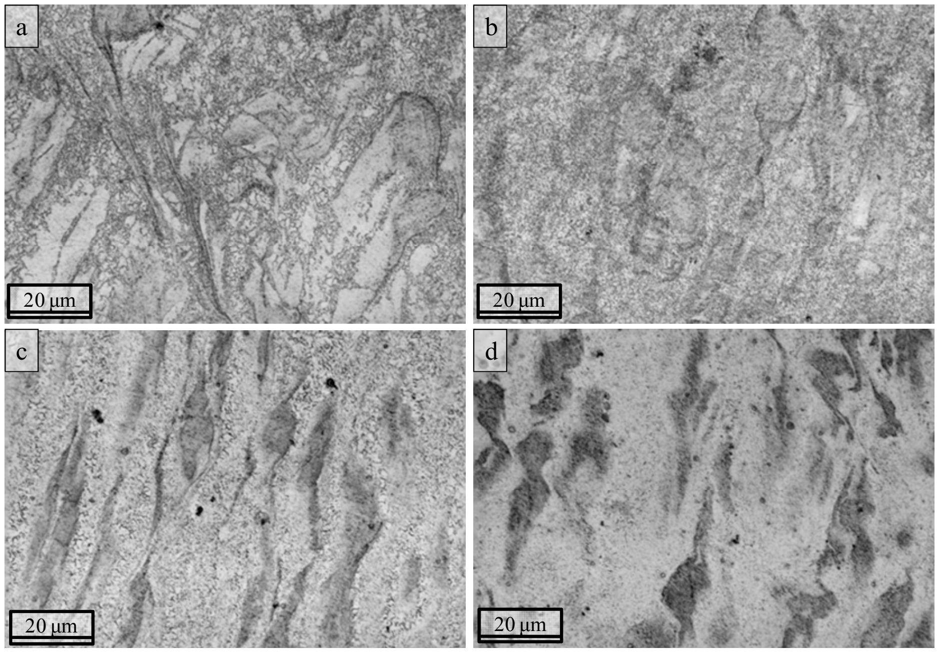

Fig. 3. Optical microstructure of ZK60 processed by ECAP after: (a) 1 pass, (b) 2 passes, (c) 3 passes and 4 passes (d) at 250°C

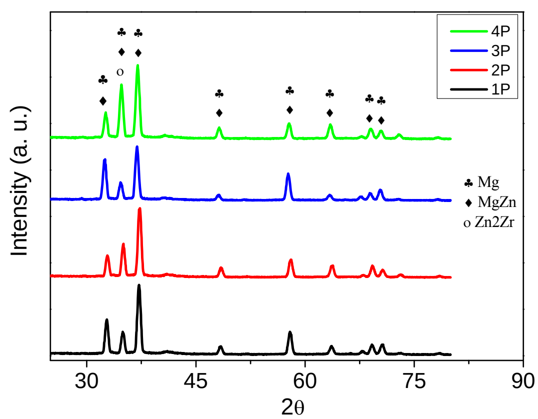

Fig. 4. X-ray diffraction of ZK60 alloy processed by ECAP route A

It was determined that the peaks show a slight variation of the angles compared to the as received analyzed material, which can be caused by the applied strain. It can be noted that with the increase in strain the peaks lose their intensity. As the second peak (belonging to the $\{0001\}$ plane) varies in intensity it can be affirmed that the basal slip system $\{0001\}\langle 11\bar{2}0\rangle$ is the one operating the deformation of ZK60 alloy, without being replaced by another system [22].

4. Conclusions

The as-received magnesium alloy presents a heterogenous microstructure with coarse grains surrounded by fine grains. Through SEM and XRD techniques the structure was determined to be composed of the primary matrix α (Mg) and the eutectic (solid solution α and MgZn and Zn_2Zr compounds).

The microstructure of the magnesium alloy ZK60 processed by ECAP shows that grains are refined with the increasing number of ECAP passes at 250°C.

For the ECAP proccesed ZK60 alloy the XRD analysis identified the same peaks as the ones encountered in the as-received material, which lose their intensity with the increase in strain. It was also identified that the main operating slip system in the magnesium alloy processed by ECAP is $\{0001\}\left\langle 11\bar{2}0\right\rangle$.

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R E F E R E N C E S

[1] *I. Ostrovsky, Y. Henn*, "Present state and future of magnesium application in aerospace industry", International Conference "New challenges in aeronautics" ASTEC'07, August 19-22, 2007, Moscow

[2] *K. Cho, T. Sano, K. Doherty, C. Yen, G. Gazonas, J. Montgomery, P. Moy, B. Davis, R. DeLorme*, " Magnesium Technology and Manufacturing for Ultra Lightweight Armored Ground Vehicles", Proceedings of the 2008 Army Science Conference, Florida, 2008

[3] *J. Lin, Q. Wang, L. Peng, H. J. Roven*, "Microstructure and high tensile ductility of ZK60 magnesium alloy processed by cyclic extrusion and compression", in Journal of Alloys and Compounds, 2009, 476(1/2), pp. 441-445

[4] *Y. Saito, H. Utsunomiya, N. Tsuji, T. Sakai*, "Novel ultra-high straining process for bulk materials-development of the accumulative roll-bonding (ARB) process", Acta Mater., Vol.47 (2) (1999), pp. 579–583

[5] *O. V. Mishin, V. Y. Gertsman, R. Z. Valiev, G. Gottstein*, "Grain boundary distribution and texture in ultrafine-grained copper produced by severe plastic deformation", Scripta Mater. 35 (1996), pp. 873–878

[6] *T. C. Lowe, R. Z. Valiev*, "The use of severe plastic deformation techniques in grain refinement", Journal of The Minerals, Metals & Materials Society 56 (10) (2004), pp. 64–68

[7] *G. A. Salishchev, O. R. Valiakhmetov, R. M. Galeyev, "*Formation of submicrocrystalline structure in the titanium alloy VT8 and its influence on mechanical properties", J. Mater. Sci. 28 (1993), pp. 2898–2902

[8] *Y. Beygelzimer, D. Orlov, V. Varyukhin*, "A new severe plastic deformation methd: twist extrusion", TMS Annual Meeting, 2002, pp 297–302

[9] *V. M. Segal*, "Equal channel angular extrusion: from macromechanics to structure formation", Mater. Sci. Eng. A271 (1999), pp 322–333

[10] *Z. Zhao, Q. Chen, Y. Wang, D. Shu*, "Microstructural evolution of an ECAE-formed ZK60-RE magnesium alloy in the semi-solid state", Materials Science and Engineering A 506 (2009), pp. 8–15

[11] *K. Furuno, H. Akamatsu, K. Oh-ishi, M. Furukawa, Z. Horita, T. G. Langdon*, "Microstructural Development in Equal-Channel Angular Pressing Using a 60 Degrees Die", ActaMater. 52 (2004), pp 2497–2507

[12] *V. Spuskanyuk, A. Spuskanyuk, V. Varyukhin*, "Development of the equal-channel angular hydroextrusion", Journal of materials processing technology 203 (2008), pp 305–309

[13] *B. Ghiban, F. - D. Dumitru, N. Ghiban, R. Saban, A. Semenescu, M. Marin*, "Consideration regarding die design for equal channel angular extrusion", 21st International DAAAM Symposium "Intelligent Manufacturing & Automation: Focus on Interdisciplinary Solutions", Vol. 21, 2010, pp. 0189-0190, ISSN 1726-9679

[14] *C. J. Luis, R. Luri, J. Léon*, "Strain and temperature analysis of AA-1370 processed by ECAE at different temperature*s*", Journal of Materials Processing Technology 164–165 (2005), pp. 1530–1536

[15] *Gil, J. Sevillano, P. Van Houtte, E. Aernoudt*, "Large strain work hardening and textures", Progress in Materials Science, No. 25, 1980, pp. 69-134

[16] *S. M. Sivakumar, M. Ortiz*, "Microstructure evolution in the equal channel angular extrusion process", Comput. Methods Appl. Mech. Engrg. 193 (2004), pp 5177–5194

[17] *B. Aour, F. Zaïri, R. Boulahia, M. Naït-Abdelaziz, J. M. Gloaguen, J. M. Lefebvre*, "Experimental and numerical study of ECAE deformation of polyolefins", Computational Materials Science 45 (2009), pp. 646–652

[18] *R. Ye. Lapovok*, "The role of back-pressure in equal channel angular extrusion", Journal Of Materials Science 40 (2005), pp. 341– 346

[19] *R. Z. Valiev, T. G. Langdon*, "Principles of equal-channel angular pressing as a processing tool for grain refinement", Progress in Materials Science 51 (2006), pp. 881–981

[20] *A. Bussiba, A.B. Artzy, A. Shtechman, S. Ifergan, M. Kupiec*, "Grain refinement of AZ31 and ZK60 Mg alloys — towards superplasticity studies", Materials Science and Engineering A302 (2001), pp. 56–62

[21] *Y. He, Q. Pan, Y. Qin, X. Liu, W. Li, Y. Chiu, J. J. J. Chen*, "Microstructure and mechanical properties of ZK60 alloy processed by two-step equal channel angular pressing", Journal of Alloys and Compounds 492 (2010), pp. 605-610

[22] *X.-M. Feng, T.-T. Ai*, "Microstructure evolution and mechanical behavior of AZ31 Mg alloy processed by equal-channel angular pressing", Trans. Nonferrous Met. Soc. China 19 (2009), pp. 293-298