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Letter

Grain refinement of Mg–Zn–Y alloy reinforced by an icosahedral quasicrystalline phase by severe hot rolling

A. Müller, G. Garcés *, P. Pérez, P. Adeva

Department of Physical Metallurgy, National Center for Metallurgical Research CENIM-CSIC, Av. Gregorio del Amo 8, 28040 Madrid, Spain

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Abstract

Severe plastic deformation allows obtaining fine grain sizes in a quasicrystalline reinforced Mg–Zn–Y alloy via hot rolling. The final grain size (~1 μm) is reached after two passes. The decreases in the grain size results in a strong increase in the yield stress.
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1. Introduction

Magnesium alloys are structural materials which are suitable for use in the aerospace and automobile industries mainly because of their low densities and high specific resistance. However, their use has been limited because of a rapid loss of strength at temperatures above room temperature and their poor creep resistance at elevated temperatures.

The Mg–Zn–Y system seems particularly promising because it is exhibits a superior mechanical performance with respect to the commercial Mg–Zn–Zr (ZK) system. Depending on the Zn/Y ratio different ternary phases have been reported [1]. Especially, with Zn/Y ratio around 5–7, a thermally stable icosahedral quasicrystalline phase (I-phase), with chemical composition of Mg_3YZn_6 [2], forms in situ as a second phase in the α-magnesium matrix during solidification. The I-phase, located in the interdendritic region, can be distributed within the magnesium matrix using thermo-mechanical treatments, obtaining an I-phase particle-reinforced metal matrix composite. A small amount of I-phase particles effectively act as dynamic recrystallization sources during hot rolling, helping to refine the grains of the magnesium matrix. Furthermore, the I-phase particles do not coarsen at high temperature due to the low diffusivity of yttrium in the alloy, inhibiting grain growth during deformation at high temperatures [3].

Several thermo-mechanical processes have been used for the microstructure refinement. Employing hot rolling at 400 °C [3,4] for alloys containing 2–4.3 at.% Zn and 0.2–0.7 at.% Y, grain distribution of 14–24 μm, yield stress of 220 MPa and ultimate tensile strength of 370 with 17% of elongation were achieved. On the other hand, via hot-extrusion [5,6], it is possible to increase the Zn and Y content, which increases the volume fraction of the I particles and therefore the mechanical strength.

Grain size reduction is an available strategy to improve the mechanical strength of the Mg–Zn–Y alloy. Severe plastic deformation (SPD) techniques have emerged in the last decade as effective methods for the production of bulk metallic materials with very fine grain sizes [7,8]. A rather large body of research has been published on the efficacy of techniques such as equal channel angular pressing (ECAP), high-pressure torsion (HPT) and severe rolling (SR) for grain refinement of a number of metallic materials. The processing-microstructure relationships corresponding to these techniques are still only scarcely known. SR, in particular, has potential to be adopted by industry to produce fine-grained materials in the form of large sheets, due to its feasibility as a continuous process. This SPD method has been successfully used to fabricate ultra fine-grained microstructures in magnesium alloys [9–12].

The present study aims to explore the possibility of applying successfully the severe hot rolling (SHR) technique for grain refinement of a quasicrystalline reinforced magnesium alloy, as well as to investigate the evolution of microstructure, texture and mechanical strength of this material during processing.

* Corresponding author. Tel.: +34 915538900.
E-mail address: ggarces@cenim.csic.es (G. Garcés).

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2. Experimental procedure

The alloys, with nominal composition of $Mg_{97.26}Zn_{2.34}Y_{0.29}Zr_{0.11}$ (at.%), was prepared by melting high purity elements in an electric resistance furnace and cast in a cylindrical steel mould (42 mm of diameter).

Slabs of 20 mm height were cut out from the initial cylinders. Before hot rolling, slabs were forged to 8 mm (92% reduction) at 400 °C. After forging, the samples were hot rolled in 2 passes. The reduction after the first and second passes was 2.6 mm (pass 1: 114% reduction) and 1 mm (pass 2: 100% reduction; 208% total reduction), respectively.

Microstructural characterisation of the alloy was carried out by X-ray diffraction, optical, scanning (SEM) and transmission (TEM) electron microscopy.

Texture analysis was performed by the Schulz reflection method, using a SIEMENS TM Kristalloflex D5000 diffractometer equipped with a close Eulerian cradle. The X-radiation used was β-filtered Cu Kα. The orientation distribution functions (ODFs) were computed from the measure of (0 0 0 2), (1 0 $\bar{1}$ 1), (1 0 $\bar{1}$ 2), (1 0 $\bar{1}$ 3) and (1 1 $\bar{2}$ 0) pole figures by the series expansion method.

Metallographical preparation for optical and SEM observation consisted of mechanical polishing and etching in a solution of 5 g of picric acid, 0.5 ml of acetic acid, 5 ml of water and 25 ml of ethanol. Specimens for TEM observation were prepared by electrolytic polishing using the reactive mixture of 5% perchloric, 35% ethylene glycol and 60% methanol at −40 °C. Then, ion milling at liquid nitrogen temperature was used to remove a fine oxide film formed on the surface for examination in a JEOL JEM-2010 microscope operating at 200 kV.

Grain size and volume fraction of quasicrystalline phase was estimated by quantitative metallographic techniques using random selected areas.

Mechanical behaviour of the Mg–Zn–Y–(Zr) alloy after severe hot rolling was evaluated by tensile tests at room temperature at an initial strain rate of 10^{-4} s^{-1}. Flat specimens were electrospark machined from the cast alloy. Tensile samples had a cross-section of 4 mm × 2 mm, a gauge length of 6.5 mm and a curvature radius r_c of 3 mm.

3. Results and discussion

The microstructure of the as-cast alloy (Fig. 1a) consisted of two phases of magnesium dendrites and eutectic I-phase in the interdendrite areas. Phases were identified by X-ray diffraction (Fig. 1b). The volume fraction of I-phase, measured by an image analysis method, was around 5%.

Fig. 2 shows the microstructure of the $Mg_{97.26}Zn_{2.34}Y_{0.29}Zr_{0.11}$ alloy after each thermo-mechanical step: forging (a and b), hot rolling (pass 1) (c) and hot rolling (pass 2) (d). During thermo-mechanical treatments, the eutectic I-phase is broken and progressively distributed within the magnesium matrix. It is important to point out that in the forging step, the brakeage of the quasicrystalline phase facilitates the consecutive severe hot rolling steps.

Fig. 3 shows the grain structure for the severe hot-rolled alloy in both passes. The grain size is 4 and 1.3 μm for pass 1 and pass 2, respectively. This difference in grain size is related to the distribution of the quasicrystalline particles. The I-phase particles act as dynamic recrystallization sources during hot rolling, helping to refine the grains of the α-Mg matrix. In the alloy rolled in the pass 1, the I-phase is not completely destroy or homogeneously distributed in the magnesium matrix (Fig. 3b). As the thickness reduction increases, the presence of more homogeneous and finer distribution of I-phase particles, located at grain boundaries, inhibits grain growth during hot rolling and results in a decrease in the grain size.

It is also interesting to point out the presence of small magnesium-zinc-rich precipitates within the magnesium matrix

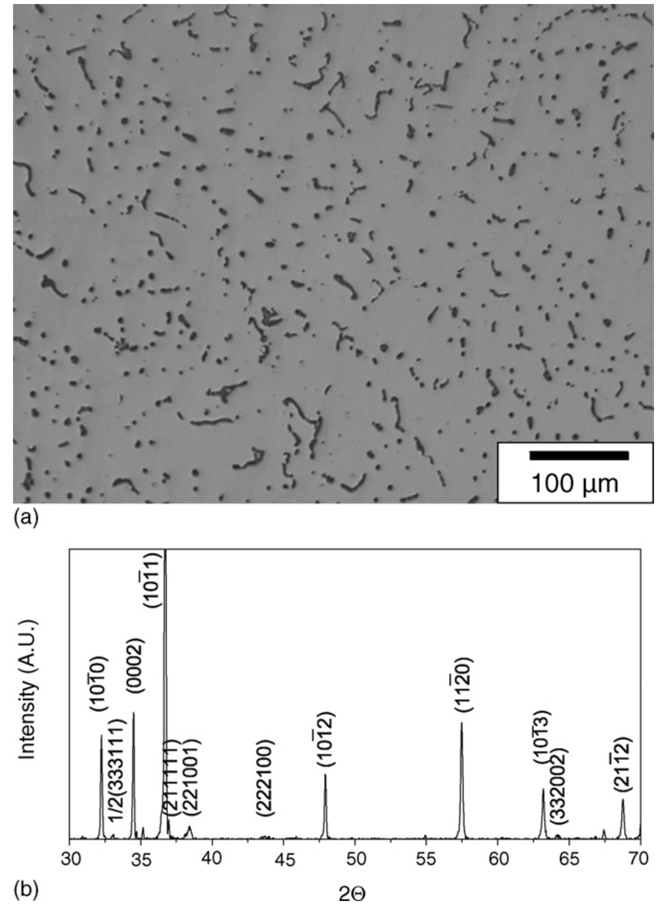

Fig. 1. Microstructure (a) and X-ray diffraction (b) of the cast $Mg_{97.26}Zn_{2.34}Y_{0.29}Zr_{0.11}$ alloy.

(Fig. 3). Different morphologies and structures have been reported [6,13–15]: rod-shape β_1', disc-shape β_2', τ_1, Mg_2Zn_{11}, $MgZn_2$ and MgZn. Further precipitation studies are been in progress.

The mechanical properties of magnesium alloys depend strongly on the grain orientation. Texture analysis has been performed to determine the effect of thermo-mechanical treatments on grain orientation. Fig. 4 shows the (0 0 0 2) pole figures in each TM step. It can be seen that, after forging (Fig. 4a), a weak basal-type texture is already developed. This fiber texture is strengthened during hot rolling as seen in Fig. 4b and c. Finally, after the last reduction (pass 2) a double peak becomes apparent, in which each intensity maximum is shifted from the ideal basal orientation by a rotation of 15° around the rolling direction. After TM processes such as rolling, extrusion, etc., magnesium alloys develop a strong texture with the basal plane perpendicular to the deformation direction [16–18]. The double peak basal texture is typical from rolled and extruded magnesium alloys [19]. Preferential deformation by $\{1 0 \bar{1} 2\} \langle 1 0 \bar{1} 1 \rangle$ twinning [20], double twinning [21] or non-basal $c + a$ slip [16] are proposed as plausible mechanisms. The present observations suggest that twinning could be ruled out as possible mechanism for the formation of the observed rolling texture since no twinning activity has been observed. Thus, the explanation put forward by Agnew et al.

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