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Effect of pretreatment and annealing on microstructure and mechanical properties of Mg–1.5Zn–0.25Gd (at%) alloys reinforced with quasicrystal

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ABSTRACT

Mg–1.5Zn–0.25Gd-based alloys containing the icosahedral quasicrystalline phase (I-phase) were fabricated to investigate the effect of heat treatment on the microstructure, mechanical properties and anisotropy of the as-extruded alloys. The results show that compared with the samples extruded without homogenized annealing, the samples extruded after homogenized annealing show larger grains, lower strength and elongation, which can be attributed to the secondary-phase particles precipitated in the matrix after homogenized annealing and to the fact that only a few volume fraction of nano-scale I-phase is precipitated in the matrix during extrusion. Moreover, the anisotropy is mitigated in the alloys extruded at as-cast condition because of the grain refinement and the I-phase precipitation. We also find that after annealing at 473 K or 673 K, yield strength decreases while the elongation increases slightly, indicating that annealing has a small effect on the improvement of room-temperature strength, especially the one at relatively higher temperature. The choice of appropriate annealing temperature only imposes a little impact on the ductility enhancement of the extruded samples.

1. Introduction

Since the discovery of quasicrystal in 1984 [1], which was a periodic solid that exhibits rotational symmetries incompatible with conventional periodic lattice order [2], many studies have been carried out, and show that quasicrystal has many intrinsic properties such as high strength and hardness at elevated temperature, low friction coefficients and low surface energy [3]. In addition, magnesium and its alloys have attracted increasing attention in recent years for engineering applications in the automotive, aerospace and other industries due to their low densities [4]. Therefore, great efforts have been made to introduce quasicrystal to magnesium as strengthening phases so as to take full advantage of their excellent intrinsic properties. The stable quasicrystalline phase was firstly reported in Mg–Zn–RE (RE=Y and rare earth element) system by Luo et al. [5], classified as the Frank–Kasper type according to their constituent elements, in which the atoms are tetrahedrally packed [6]. Mg–Zn–RE alloys reinforced with quasicrystal have been studied widely, especially those after thermal mechanical processing, such as extrusion [7–11], rolling å[12–14], equal channel angular extrusion [15,16] and so on. In as-casted condition, quasicrystal formed at the grain boundary as dendritic secondary phase, which is helpful to improve elevated temperature mechanical properties but not has key effect to room temperature mechanical properties because the size of the quasicrystal is large [17,18]. After thermal mechanical deformation, dendritic quasicrystal phase broken to micro-scale and grains are refined as well as nano-scale quasicrystals are precipitated in the matrix, which contribute to the improvement of mechanical properties. Previous studies also found that nano-scale quasicrystals had an orientation relationship and coherent interface with the matrix [19–21], so these nano-scale quasicrystals are helpful for Mg alloys to achieving high strength and excellent plasticity simultaneously [19]. Therefore, how to achieve large volume fraction of nano-quasicrystal becomes of the key problem to improve the mechanical properties of Mg alloys reinforced with quasicrystal.

Heat treatment is the most commonly used method to optimize the microstructure and then improve the mechanical properties of the samples. However, previous study results showed that homogenization of the cast ingots and the quenching-plus-aging treatment of the extruded bars decreased the strength of Mg–Zn–Zr–RE

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alloys [22]. Hence, in our previous studies we usually extruded the billets at as-cast conditions [23–25]. But Osawa et al. [26] and Somekawa et al. [27] previous study results showed that a homogeneous structure before the wrought process was effective for producing more homogeneously fine-grained structures. Therefore, we also studied Mg–Zn–Gd alloys reinforced with quasicrystal extruded at solution treated condition [19,28]. However, few studies systematically investigated the effect of heat treatment on the microstructure and mechanical properties of Mg–Zn–Gd alloys reinforced with quasicrystal, especially the effect of solution treatment on the precipitation of nano-scale quasicrystal during thermal mechanical processing. Therefore, in this study, we aim at investigating systematically how the heat treatment can have an impact on microstructure and mechanical properties of Mg–1.5Zn–0.25Gd alloys with a special focus on their effect on nano-scale quasicrystal precipitates. Then, microstructures of the as-cast, solution treated, extruded and annealed are comparatively studied, mechanical properties of the extruded and annealed alloys are measured and the effects of heat treatment are discussed comprehensively.

2. Experimental procedure

An alloy with the composition of Mg–1.5Zn–0.25Gd (at%) was fabricated, and the details were reported in our previous study [18]. The as-cast billets were machined into rods with 60 mm in diameter for extrusion. Before extrusion, rods materials were divided randomly into two groups: one group was subjected to solution heat treatment at 673 K for 8 h according to Ref. [19,29] and the other directly went through hot extrusion deformation. Extrusion was carried out on a 3150 KN press machine and the oil-based graphite was used as the lubricant. The extrusion temperature was 523 K and the extrusion ratio was 25:1. The as-extruded samples with and without solution heat treatment were named as A1E25 and A1E25Z, respectively.

Some of the extruded samples were annealed at 473 K and the others were annealed at 673 K for 2 h, 4 h, 8 h and 16 h, respectively, according to our previous study [24]. Phase constituents were analyzed using X-ray diffraction (XRD, D/MAX 2550) with the Cu-$K\alpha$ radiation at 40 kV and 100 mA. The step was fixed to å4°/min and the measurement angle ranged from 20° to 80°. X-ray diffraction curves of the as-cast and homogenized treated samples were reported in Ref. [18]. Microstructure was characterized by optical microscopy (OM, Leica MEF4M), scanning electron microscopy (SEM, FEI NOVA NanoSEM and FEISirion 2000) and transmission electron microscopy (TEM, JEOLJEM-2010). The as-cast and solution treated samples for the OM and SEM observations were polished and etched in a mixed solution of 4% nitric acid and 96% alcohol, while a solution of 1 g oxalic, 1 mL nitric acid, 1 mL acetic acid and 150 mL distilled water was used for the as-extruded and annealing samples. The linear intercept method was applied to measure the average grain size for the samples [30]. Macro-texture analysis of the as-extruded bars was performed on their section perpendicular to the extrusion direction using X-ray diffractometer (XRD, PW3040/60). Analysis of the micro-texture was carried out at the center of the compressed samples by EBSD, using data acquisition software (TSL-OIM 5.0). Micro-textures of the samples were scanned by the EBSD equipment over a fixed area (90 μm × 140 μm) with a step size of 0.25 μm. The samples for TEM observations were made by mechanical thinning and then ion milling. The hardness of as-cast and solution-treated specimens were checked using a Vickers hardness tester under a load of 49 N and holding time of 20 s. Samples for the tensile and compressive tests were prepared by cutting along the extrusion direction (ED) with electric spark machining. To eliminate uncertainties, more than three samples were tested for each condition. The tests were carried out at an initial strain rate of 1×10^{-3} s^{-1} using the Zwick/RoellZ020 testing machine.

3. Results

3.1. As-casted and solid solution treated microstructures

Fig. 1 shows the as-cast and solid solution treated microstructures of the study alloys. The as-cast microstructure of Mg–1.5Zn–0.25Gd alloy shows typical dendritic structure with the second dendrite arm spacing of about 27 μm, as shown in Fig. 1(a). After solid solution heat treatment, the microstructure only has a little change. Only small amount of secondary phase solution into the matrix, and the microstructure of the alloy become more homogeneous than that of the as-casted sample, as shown in Fig. 1(b). Fig. 1(c) is the magnified SEM image of the as-cast microstructure. The matrix is very clear and there are no any other secondary phases in the matrix except the secondary phases distributed along the grain boundaries. After solid solution heat treatment, SEM observation found that plenty of newly formed tiny secondary phases precipitated dispersedly in the matrix except for the changes in the sizes of the grain boundary secondary phase, as shown in Fig. 1(d).

To further study the as-cast and solid solution heat treated microstructures of the alloy, TEM observation and diffraction analysis were carried out, as shown in Fig. 2. Fig. 2(a) is the TEM bright field image for the as-casted sample. The largest dendritic secondary phase has an interface (the dotted line as shown in Fig. 2(a)), which divided the secondary phase into two parts. The selected area diffraction (SAD) patterns are shown in Fig. 2(b)–(e), respectively. The diffraction patterns in Fig. 2(b)–(d) reveal that the left part is Mg_2Zn_3 phase. The SAD pattern in Fig. 2(e) reveals a å5-fold symmetry, which is the typical characteristic for the icosahedral quasicrystalline structure (I-phase) [19,24,29]. Fig. 2(f) is the magnified images of the matrix zone for the as-casted sample. Fig. 2(g) is the SAD pattern of the matrix along the zone axis [−1 1 0 0] and Fig. 2(h) is the composite diffraction pattern of the matrix and the largest secondary phase in Fig. 2(f). The growth direction of the needle-like Mg_4Zn_7 phase is parallel to the (0 0 0 1) of the Mg matrix and $[0\,0\,1]_{Mg4Zn7}$ zone axis is parallel to $[-1\,1\,0\,0]_{Mg}$ zone axis. Fig. 2(i) shows the TEM bright field image of the solution heat-treated sample, and the SAD patterns along different zone axis of the eutectic secondary phase with the largest size are shown in Fig. 2(j)–(m). These diffraction patterns show 2-fold, 3-fold, minor 2-fold and 5-fold symmetry, respectively, which reveals that the I-phase remained after solution heat treatment because it has high thermal stability [29,31]. In addition, many newly formed secondary phase is precipitated in the matrix, as shown in Fig. 2(n), with needle-like shape. The SAD patterns of the needle-like secondary phase, as shown in Fig. 2(o), reveals that it is $MgZn_3Gd$ crystal phase. Because the crystal secondary phase is not the main issue of this paper, so we do not analyze this phase systematically.`

3.2. The microstructure and constituent phase of the extruded samples

Fig. 3 is the microstructures of the extruded Mg–1.5Zn–0.25Gd alloy. As reported in the previous articles [19,24], after extrusion, the I-phases in the interdendritic region for as-cast alloy and solid solution heat treated alloy were sharply destroyed and formed a band structure along the extrusion direction, as shown in åFig. 3 (a) and (b) as the black string. Multimodal microstructure, similar to the previous study [9,22,32,33], was formed and the mean grain