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Microstructure and mechanical properties of Mg-4Zn-0.5Ca alloy fabricated by the combination of forging, homogenization and extrusion process

Jin-wen Kang, Cui-ju Wang, Kun-kun Deng*, Kai-bo Nie, Yan Bai, Wei-jian Li

Shanxi Key Laboratory of Advanced Magnesium-based Materials, College of Materials Science and Engineering, Taiyuan University of Technology, Taiyuan 030024, PR China

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The Mg-4Zn-0.5Ca alloy was processed by the combination of forging, homogenization and extrusion in present study. The results showed that homogenization treatment between forging and extrusion process can release stored energy and diminish precipitated $MgZn_2$ phase, both of which result in the finer size and low volume fraction of dynamic recrystallized (DRXed) grains, as well as strong basal plane texture in the as-extruded alloy (FD-H-ED). As compared with the as-extruded alloy without homogenization treatment (FD-ED), the FD-H-ED alloy exhibited high yield strength which is thought to originate from the dislocation strengthening and texture strengthening. Only stage III and stage IV appeared in the θ vs. $(\sigma - \sigma_{0.2})$ curves of FD-ED and FD-H-ED alloys. Large amount of fine DRXed grains in FD-ED alloy led to the high dynamic recovery rate at stage III. However, the unDRXed regions in FD-H-ED alloy resulted in its unobvious starting point and promote the decrease of θ value at Stage IV.

1. Introduction

Magnesium alloys had received considerable research for potential applications in the electronics, automobile and aerospace industries because of their low density and high specific strength [1,2]. However, the low strength and poor ductility of conventional Mg alloys strongly restricted their commercial application. Microalloying and microstructure refinement had been thought as effective ways to improve the mechanical properties of Mg alloys [3,4].

Among the commonly used Mg alloys, Mg-Zn system alloy showed significant potential for high strength wrought Mg alloys [5] due to their low cost, good precipitation hardenability [6] as well as creep resistance [7]. Minor Ca addition in Mg–Zn alloy can reduce flammability, improve the oxidation resistance and refine dynamic recrystallized (DRXed) grains [8,9]. Therefore, the Mg-Zn-Ca series alloy has the potential to possess better mechanical properties and attract more researchers' attention over next few years.

Conventional thermomechanical processing is thought as the most effective ways for strength and ductility improvement of Mg alloys through refining grains. Especially, the several plastic deformation (SPD), such as equal channel angular pressing (ECAP) [10], extrusion [11] and rolling [12] had been applied on Mg-Zn-Ca series alloy. The ultra-fine grained Mg-5.25Zn-0.6Ca (wt. %) alloy had been achieved through ECAP method by Tong et al. [10]. Their further research on this kind alloy indicated that the yield strength (YS) of 220 MPa and elongation (EL) of 21.4% can be obtained after being processed by extrusion [11]. Besides, recent study on Mg-4.5Zn-1.13Ca (wt. %) alloy prepared by double extrusion, showed that the YS of single-extruded alloy was 173 MPa, while the value could be increased to 320 MPa after subsequent second extrusion [8]. Similarly, Tong et al. [12] also reported that the YS of extruded Mg-5.3Zn-0.6Ca-0.5Ce/La (wt. %) alloy was remarkably increased from 163 MPa to 316 MPa via the warm rolling process. Hence, multi-step deformation is a more effective way to improve the mechanical properties of Mg alloys.

In addition, the initial microstructure also has important influence on the as-deformed alloy. Wang et al.'s investigation [13] indicated that different initial grain size and texture gave rise to different mechanical properties after rolling. To improve the formability, the secondary phase was usually dissolved into Mg alloys through homogenization treatment before hot deformation.

* Corresponding author.
E-mail address: jamsdk@163.com (K.-k. Deng).

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However, the secondary phase had been proved to have great influence on the microstructure and mechanical properties of Mg alloys after deformation. The as-cast AZ91 alloy containing and without $Mg_{17}Al_{12}$ phase were both subjected to hot extrusion by Wang et al. [14], the result showed that the YS is almost same, whereas the ultimate tensile strength (UTS) and EL were higher than that without $Mg_{17}Al_{12}$ phase. Nevertheless, Shang et al.'s investigation [15] had proved that the SiCp/Mg–Al–Zn composite containing $Mg_{17}Al_{12}$ phase would exhibit higher mechanical properties after hot extrusion. Even though some research had been carried out in Mg-Al series alloys, the Mg-Zn series alloys are less studied.

As a consequence, the homogenization treatment is used to control grain size and precipitate of Mg-4Zn-0.5Ca alloy between the combination of forging and extrusion process. And its influence on the microstructure and mechanical properties of Mg-Zn-Ca alloy after subsequent extrusion is to be given and analyzed in the present work.

2. Experimental procedures

2.1. Materials

The Mg-4Zn-0.5Ca (wt. %) alloy was initially prepared by conventional gravity casting, using an electric resistance furnace melting of high purity Mg (wt. 99.95%), Zn (wt. 99.95%) and Ca (wt. 99.95%) under a cover gas mixture of CO_2 and SF_6. The melts were held at 730 °C for 20 min and casting them into a preheated steel mold (300 °C). Billets 30 mm × 30 mm × 60 mm in size were machined from the ingot.

2.2. Hot deformation procedure

The process flow diagram of Mg-Zn-Ca alloy is given in Fig. 1. According to Ref. [16], the forging was carried out at 300 °C with a pressing speed of 0.1 mm s^{-1}, using a press with a 450 kN load limit. Once the forged die attained the desired temperature, a period of 30 min was allowed to elapse before the forging was carried out. Schematic representation of the forging could be found in Ref. [17].

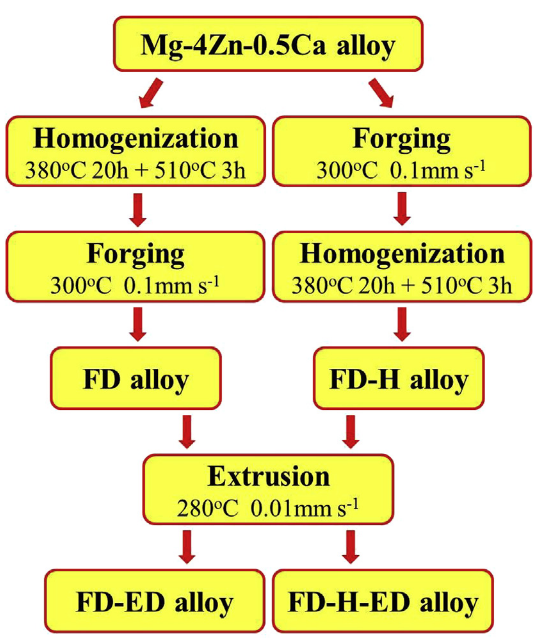

Fig. 1. The process flow diagram of Mg-4Zn-0.5Ca alloy.

Two-step homogenization treatment was applied according to Ref. [18], the Mg-Zn-Ca alloy was homogenized at 380 °C for 20 h firstly, and then be held at 510 °C for 3 h. Then, the Mg-Zn-Ca alloy was forged at height reduction of 50%, which was denoted as FD alloy. For comparison, the Mg-Zn-Ca alloy was forged firstly and then followed by homogenization, which was denoted as FD-H alloy. Subsequently, the FD-H alloy was extruded at 280 °C with an extrusion ratio of 16 and a ram speed of 0.01 mm s^{-1}, which was denoted as FD-H-ED alloy. To emphasize the influence of homogenization treatment between forging and extrusion process, the FD alloy was also extruded at the same conditions for comparision (denoted as FD-ED alloy).

2.3. Microstructure observation

Microstructure characterization was carried out using an 4XC optical microscope (OM), MIRA3 LMH scanning electron microscope (SEM) and transmission electron microscopy (TEM). Samples for OM, SEM and X-ray diffraction (XRD) analysis were all cut along extrusion direction (ED) and/or carried out in the central part of specimens parallel to the forging axis. The specimens for OM were ground, polished and etch in oxalic [4 g oxalic + 96 ml H_2O]. The average size and volume fraction of the DRXed grains and precipitates were measured by Image-Pro Plus 6.0 software. To ensure the statistical reliability, the measurements were based on four images. The volume fraction of the DRX grains (V_{DRX}) could be defined as follows [19]:

$$V_{\mathrm{DRX}} = \frac{\sum A_f}{\sum A_i} \tag{1}$$

where A_f denotes the area of DRXed grains in every metallographs, A_i denotes the area of every metallographs. TEM specimen was ion-milled to perforation at an ion accelerating voltage of 3 KV. The distribution of secondary phase was analyzed by SEM equipped with Energy dispersive spectrometer (EDS). The phase identification and analysis were performed on X-ray diffraction using Cu-Kα radiation, a scanning rate of 3°/min and a sample tilted angle ranging from 20° to 80°. The texture of as-extruded alloy was tested along ED using the Schultz reflection method with Rigaku D/max-2400 X-ray diffraction pattern machine.

2.4. Tensile test

The tensile specimens of as-extruded alloy with a gauge length of 50 mm and cross-sectional areas of 6 mm × 2 mm were cut parallel to extrusion direction. A total of three tensile specimens for each sample were performed at room temperature using an Instron Series 5569 test machine at a tensile rate of 0.5 mm min^{-1}.

3. Results and discussion

3.1. Microstructures of as-cast Mg-4Zn-0.5Ca alloy

Fig. 2 shows the XRD pattern of the as-cast Mg-4Zn-0.5Ca alloy. The result reveals that the as-cast alloy mainly consists of α-Mg and $Ca_2Mg_6Zn_3$ phase. Fig. 3 shows the SEM images and EDS surface scan of as-cast Mg-4Zn-0.5Ca alloy. It reveals that most of the secondary phase exhibits eutectic morphology of network, which is either distributed around grain boundaries or in grain interiors. Fig. 3(b) is the high magnification of Fig. 3(a), showing two shape of secondary phase. The magnified Fig. 3(b) is given in Fig. 3(d), which exhibits the ellipsoidal divorced eutectic morphology as well as the lamellar eutectic morphology at the triple junctions of grain boundaries. The EDS surface scan of Fig. 3(b) is given in Fig. 3(c), it