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Fabrication of a high strength Mg–11Gd–4.5Y–1Nd–1.5Zn–0.5Zr (wt%) alloy by thermomechanical treatments

Zijian Yu [a,b,c], Yuanding Huang [b], Xin Qiu [a], Guanfu Wang [d], Fanzhi Meng [a], Norbert Hort [b], Jian Meng [a,*]

[a] State Key Laboratory of Rare Earth Resources Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, PR China
[b] MagIC-Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, D-21502 Geesthacht, Germany
[c] University of Chinese Academy of Sciences, Beijing 100049, PR China
[d] Wenzhou Medical College, Affiliated Taizhou Hospital, Luqiao Division, Taizhou, Zhejiang 318050, China

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ABSTRACT

A high strength Mg–11Gd–4.5Y–1Nd–1.5Zn–0.5Zr (wt%) alloy has been successfully fabricated via hot extrusion, cold rolling and ageing treatment. The alloy exhibits an average 0.2% proof stress (YS) of 481.6 ± 19.9 MPa, an average ultimate tensile strength (UTS) of 517.4 ± 27.5 MPa and an average elongation to failure of 2.0 ± 0.4% at room temperature. The best mechanical property obtained in the present study has a YS of 502.0 MPa, an UTS of 546.8 MPa and an elongation to failure of 2.6%. The high strength of this alloy is attributed to the fine grains, stacking faults (SFs), long period stacking ordered (LPSO) phase, and precipitates of Mg_5RE phase at grain boundaries and of β' phase inside the grains. Cold rolling improves the mechanical properties and enhances the ageing hardening response, but decreases the ductility. Two texture components are found simultaneously in the deformed alloy. One is the typical Mg–RE texture and another one is the unusual prismatic texture.

1. Introduction

The rare-earth (RE) containing Mg alloys have been investigated for decades because of their excellent mechanical properties. Normally, the heavy RE elements such as Gd, Dy and Y have a high solubility in Mg at elevated temperatures. In contrast, the light RE such as Nd and Ce has a low solubility in Mg. For example, the solubility of Y and Nd is 12.5 wt% and 3.6 wt% at eutectic temperature in pure Mg, respectively [1]. In comparison with Y, Gd has a much higher solubility of 23.5 wt% at Mg–Gd eutectic temperature of 548 °C, which declines sharply to 3.8 wt% at 200 °C [1]. Consequently, Gd is expected as a promising candidate for producing high strength Mg alloys because of its effective solution and precipitate strengthening [2–7]. Zn addition in Mg can significantly enhance the ageing hardening response and mechanical properties of Mg–RE alloys due to the fact that it can reduce the solubility of RE in α-Mg matrix [8]. In addition, Zn is an essential element for the formation of long period stacking ordered (LPSO) phase in Mg–RE–Zn alloys [9]. The strengthening mechanism of LPSO phase has been attracting increasing attentions because of its strong plastic anisotropy and layered structure [10–13]. The deformation behavior of LPSO phase largely depends on the loading direction. Therefore, the deformation behavior and mechanical properties of alloys containing the LPSO phase are considered to be sensitive to microstructure and loading direction. The deformation behaviors and strengthening mechanisms of the LPSO phase are well studied under compression, but need to be further investigated under tension.

Up to now, the most successful commercial Mg–RE alloys have been realized as the use of Mg–Y–Nd–Zr alloys, identified as WE54 (Mg–5.1 wt% Y–3.3 wt% RE–0.5 wt% Zr) and WE43 (Mg–4.0 wt% Y–3.3 wt% RE–0.5 wt% Zr) alloys [14–16]. The high strength of these alloys is attributed to the precipitation of metastable β' phase and equilibrium Mg_5RE phase [14,15]. Another major contribution is the development of high strength Mg–Y–Zn alloys which was produced by rapidly solidified powder metallurgy (RS P/M) [17]. This alloy exhibits remarkable mechanical properties with an yield strength (YS) of 610 MPa and an elongation of 5%. The ultra-fine grain size and LPSO phase are mainly responsible for the improvement of the strength. However, the production process is more complicated compared to the conventional wrought processes, such as extrusion and rolling [18,19].

Hot extrusion is an effective technique to refine the microstructure and to improve the mechanical properties of Mg alloys [18,20]. A high-strength Mg–10Gd–5.7Y–1.6Zn–0.7Zr (wt%) alloy with an yield strength of 473 MPa and an elongation of 8% has

* Corresponding author. Tel.: +86 431 85262030; fax: +86 431 85698041.
E-mail address: jmeng@ciac.jl.cn (J. Meng).

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been successfully fabricated by hot extrusion and subsequent ageing [18]. Its excellent mechanical properties are attributed to the fine precipitates formed during ageing and thermomechanical processing. Warm rolling is another effective process to refine the microstructure and to produce large Mg alloy sheets with good mechanical properties [21–23]. Normally, a large total rolling reduction is required for producing a high strength Mg–RE alloy. For example, a high-strength Mg–8.2Gd–3.8Y–1Zn–0.4Zr (wt%) alloy with a YS of 426 MPa and an elongation to failure of 4.5% was produced via warm rolling and ageing [21]. The final reduction is 96%. However, for the cast magnesium alloys, due to their poor workability caused by their intrinsic crystal structure (hcp, hexagonal-closed packed), large grain size, coarse secondary phases and possible casting defects [24–26], normally, the warm rolling are carried out at high temperatures with a small rolling reduction per pass in order to avoid the cracking [23]. An initial extrusion prior to cold rolling can refine the microstructure and reduce the casting defects [27,28]. As a result, the extruded Mg alloy can achieve a high workability and avoid the cracking in the following cold rolling process [27]. Thus, a combination process of hot-extrusion and cold rolling may be an appropriate method to produce high strength Mg–RE alloys.

The purpose of the present work is to produce an ultra-high strength Mg–RE–Zn–Zr alloy via a combination of hot-extrusion, cold rolling and ageing treatment. The mechanical properties of the present alloy and the microstructural evolution during thermomechanical processes will be investigated. Furthermore, the deformation behavior and the strengthening mechanisms of LPSO phase under tension will be discussed.

2. Experimental procedures

Mg–11Gd–4.5Y–1.5Zn–1Nd–0.5Zr alloy was cast at 760 ± 3 °C under gas protection of mixed SF_6/CO_2 (1:99) into a cylindrical steel mold having a diameter of 92 mm. The mold was preheated to 200 °C. Homogenization annealing was performed at 535 °C for 24 h followed by quenching in water (25 °C). The homogenized billet was machined into a round bar with a diameter of 82 mm and length of 120 mm for subsequent extrusion at 410 °C with a ratio of ~30:1 under a ram speed of 1.0 mm s^{-1}. The extruded bar was machined into slabs with a dimension of 120 mm × 8 mm × 2.8 mm for cold rolling. The cold rolling sample appeared to crack as soon as the total reduction was more than 14.5%. Therefore, the cold rolling was carried out in the extrusion direction (ED) with a thickness reduction of 5% per pass up to the total reduction of 14.5%. In order to systemically investigate the effect of cold rolling on microstructural evolution and mechanical properties, three samples were selected including the as-extruded sample and two cold rolled samples with a reduction of 10% and 14.5% respectively. Ageing treatment (*T5*) for the selected samples was carried out at 200 °C for 48 h. Table 1 shows the detailed specimen information. The extruded sample was donated as E sample. E-10R represented the extruded and rolled sample with a rolling reduction of 10%, and E-14.5R represented the one with a rolling reduction of 14.5%. These samples were donated as E-T5, E-10R-T5 and E-14.5R-T5 after T5 treatment.

Table 1
History of the studied samples.

Sample	History
E	Hot extrusion at 410 °C with a ratio of 30:1
E-10R	E sample + cold rolling at room temperature with a total thickness reduction of 10%
E-14.5R	E sample + cold rolling at room temperature with a total thickness reduction of 14.5%
E-T5	E sample + ageing treatment at 200 °C for 48 h
E-10R-T5	E-10 R sample + ageing treatment at 200 °C for 48 h
E-14.5R-T5	E-14.5 R sample + ageing treatment at 200 °C for 48 h

Tensile specimens with a gauge dimension of 20 mm × 4 mm × 2 mm were tested in the ED or rolling direction (RD) using Instron 5569 tension tester with a speed of 1 mm min^{-1} at room temperature. Each alloy state was tested with three specimens in order to obtain the average value and standard deviation. The microstructures were observed using optical microscopy (OM), scanning electron microscopy (SEM) at 15 kV on Zeiss Ultra 55, transmission electron microscopy (TEM) at 200 kV on Philips CM 200 and high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) at 300 kV on FEI-TECNAI G^2. The average grain size and particle size were measured using liner intercept method. More than 300 grains, 30 LPSO phase particles and 1300 irregular particles were collected for the measurement. The volume fraction of the secondary phases was calculated from the average area of their particles. The foils for TEM were prepared by mechanically grinding to a thickness of 120 μm and then thinned by electropolishing with a twin jet system. The electropolishing was performed in a solution of 2.5% $HClO_4$ and 97.5% ethanol at −45 °C under a voltage of 40 V. The second phases were also identified by X-ray diffraction (XRD) (D8 FOCUS X-ray diffractometer) with Cu radiation. The global texture information was obtained using X-ray diffraction (Panalytical, 40 kV and 40 mA) with rectangular slat (15 mm × 8 mm × 2 mm). (0001) and $(10\bar{1}0)$ pole figures of E, E-10R and E-14.5R samples were selected to analysis the texture evolution during thermomechanical process.

3. Results

3.1. Tensile deformation behavior

Fig. 1 shows the typical tensile strain–stress curves of the E, E-10R and E-14.5R samples with and without ageing treatment. The curves shown in Fig. 1 are obtained from the specimens with the best tensile properties among these three tested specimens for each state. The mechanical properties with average values and standard deviations are summarized in Table 2. The subsequent cold rolling can increase both the YS and the UTS, but reduce the ductility with increasing the rolling reduction. The E sample has an average YS of 305.7 ± 1.0 MPa, an average UTS of 362.5 ± 3.2 MPa and an average elongation to failure of 6.2 ± 0.9%. The average YS and UTS of E-10R sample and E-14.5R sample increase to 357.5 ± 23.6 MPa, 400.6 ± 2.6 MPa and 413.7 ± 28.4 MPa, 443.3 ± 13.0 MPa, and the elongation to failure decrease to 2.9 ± 0.8% and 2.4 ± 0.6%, respectively. After cold rolling, the subsequent ageing treatment greatly enhances the strength. The increments in the average YS and UTS range from 70.8 MPa to 89.2 MPa and from 58.3 MPa to 74.1 MPa. The influences of ageing treatment on the ductility of E sample and E+R sample are different. For the E sample, the average elongation to failure decreases to 2.8 ± 0.4% from 6.2 ± 0.9% after ageing treatment. In comparison, the changes in ductility of the E-10R and E-14.5R samples are negligible. The E-14.5R-T5 sample has an high average YS of 481.6 ± 19.9 MPa and an average UTS of 517.4 ± 27.5 MPa, but a poor ductility of 2.0 ± 0.4% after ageing treatment. One sample shows a YS of 502.0 MPa and an UTS of 546.8 MPa and an elongation to failure of 2.6% (Fig. 1b).

Fig. 2 shows the microstructure of E sample at the fracture surface and in the vicinity of fracture surface. The cracks in LPSO phase particles are marked by black arrows and labeled with numbers from 1 to 6. They are roughly normal to the ED. Number 1–3 cracks are found in the region next to the fracture surface