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Ultrafine-grained Mg–Zn–Zr alloy with high strength and high-strain-rate superplasticity

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ABSTRACT

We report the feasibility of fabricating ultrafine-grained ZK60 magnesium alloy sheets with a combination of superior mechanical properties at room temperature and excellent superplasticity at high strain rates by means of high-ratio differential speed (HRDSR). The ZK60 alloy processed under the optimum HRDSR condition showed a high yield strength of 378 MPa and a reasonably high ductility (a total elongation of 12.3%). The major strengthening mechanisms could be attributed to the grain size and particle strengthening. Its microstructure was quite thermally stable up to 573 K, such that high strain rate superplasticity could be achieved at 10^{-2} and 10^{-1} s^{-1}. The development of high-density shear bands that occurred uniformly over the entire volume of a sample and dynamic precipitation of spherical or irregular shaped particles with the sizes of 20–32 nm along the lamellar boundaries in the shear bands and on the boundaries of grains newly formed within the shear bands via continuous dynamic recrystallization, resulted in an ultrafine-grained microstructure with high thermal stability. The room-temperature mechanical properties and superplastic ability of the HRDSR-processed ZK60 were comparable to those of a magnesium alloy of a same or similar composition processed via powder metallurgy. Because the currently developed HRDSR technique is based on a rolling process and only a few processing steps are involved, its transfer to industry that demands high-performance magnesium alloy sheets is promising.

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1. Introduction

Magnesium alloys with a combination of high strength and high formability represent ideal materials for a wide range of applications in the automobile and aerospace industries, medicine, energy and sports products. The economical production of these materials in large quantities is also important for their widespread use.

Equal channel angular pressing (ECAP), which is a severe plastic deformation (SPD) method, has been the most widely studied for improving the mechanical properties of Mg alloys through grain refinement and texture modification [1–7]. Shear deformation repeatedly imposed at the sharp ECAP die corner often increases the ductility of Mg alloys by rotating a high portion of basal planes such that they have high Schmid factors for the test loading direction. This texture change, however, often results in a strength decrease below the level of the starting material due to texture-softening [2,5]. For example, the ECAP-processed ZK60 alloy with 4–8 passes via route B_c [4] exhibited yield stresses of 120–125 MPa, which were lower than that of the starting material by ~40 MPa. To overcome this problem, Orlov et al. [7] proposed an integrated process that combined conventional extrusion and ECAP into a single process that produced a fine-grained microstructure and favorable texture that promoted basal slip, yet not to an extent that strength was largely sacrificed. The ZK60 alloy processed by this method exhibited a good balance of strength and tensile ductility, showing a yield stress (YS) of 310 MPa and a total elongation of 17.1%. Another method for simultaneous improvement of strength and ductility in Mg alloys is surpassing the texture-softening effect by significantly reducing the grain size to a sub-micron level through the use of low processing temperatures below 473 K. Yoshida et al. [8] showed that the grain size of the AZ61 alloy could be reduced to 0.5 μm by ECAP at 448 K. The resultant material exhibited a YS of 351 MPa with a total elongation of 33%.

Though the improvement of ductility at room temperature by ECAP is impressive, the extent is often insufficient to realize the formation of components with complicated shapes at room temperature. For this reason, formation of most of the wrought Mg alloys is conducted at elevated temperatures. It is well known that grain refinement enhances superplasticity by increasing the optimum strain rate for superplasticity because the contribution of the grain boundary-sliding (GBS) mechanism to the total plastic flow increases as the grain size decreases. Conventional superplastic flow, however, often occurs at low strain rates, which reduces the forming rate and leads to low productivity in manufacturing

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components. High strain rate superplasticity (HSRS) is the ability of a material to sustain large plastic deformation in tension at high strain rates [9]. In HSRS, the peak values of tensile elongations over 500% are obtained at strain rates above 10^{-2} s^{-1}, and high strain-rate sensitivity exponents (m) of 0.5 are measured at corresponding strain rates. For this reason, HSRS is attractive for commercial applications since it enables the formation of components with complex shapes at high forming rates. HSRS has been observed in ultrafine-grained magnesium alloys and magnesium composites processed by powder metallurgy (PM) technique combined with a high ratio extrusion. For example, the PM Mg–Zn–Zr (ZK61) alloy with a grain size of 0.65 μm, which was processed through powder sintering and subsequent extrusion with a reduction ratio of 100:1, exhibited tensile elongations of 400–650% at 1×10^{-2}–1×10^{-1} s^{-1} [10]. The enhancement of superplasticity in Mg alloys through grain refinement by SPD has been also extensively studied [11–15]. There are many reports of excellent low temperature superplasticity (at temperatures less than 0.5 T_m, where T_m is the melting temperature) in Mg alloys processed by ECAP [11–14], but there has been little success in achieving HSRS in Mg alloys. This is most likely due to the poor grain-size stability of the ingot-processed Mg alloys compared to the PM counterparts. The superior thermal stability of the PM Mg alloys was attributed to precipitates that were more densely and uniformly dispersed over grain boundaries and the matrix, and a considerable amount of thermally stable oxide particles in the matrix, which were inevitably introduced during the atomization process for making the powders [10].

In a previous study [16], we succeeded in producing ultrafine-grained (UFG) ZK60 sheets capable of exhibiting HSRS via high-ratio differential speed rolling (HRDSR). This HRDSR technique, which has been studied by one of the current authors [17,18], has the advantage of being suited to cost-effective mass production of UFG sheets because the process is continuous and requires only a few rolling passes. In addition, unlike in ECAP, scale-up is easy and no special die is required. Here, we report the development of Mg alloy sheets with excellent mechanical properties at room temperature and HSRS at elevated temperatures. We examined the microstructure of the HRDSR-processed ZK60 alloy in detail and analyzed its mechanical behavior to understand the physical mechanisms underlying the improvement of strength and HSRS in Mg alloys.

2. Materials and methods

The material used in this study was a 6 mm-thick ZK60 plate (5.16% Zn-0.57% Zr-bal Mg in weight percent) with a width of 110 mm extruded from an ingot with a diameter of 192 mm. The plate was conventionally rolled to 2 mm with 2 passes at 573 K. In the following HRDSR process, the speed ratio between the upper to lower roller speed was set at 3. The top roll rotated with a speed of 3 rpm in the counterclockwise direction while the bottom roll rotated with a speed of 1 rpm in the clockwise direction. The roll surfaces were maintained at 473 K throughout the rolling process using heating elements embedded within the rolls. In the first trial, a cold sample was fed into the hot rolls under non-lubricated condition for a thickness reduction of 68% by a single pass (referred to as the HRDSR ZK60C, hereafter). The second attempt was made with sample preheating. A sample preheated to 473 K was subjected to HRDSR under non-lubricated condition for a similar thickness reduction of 70% by a single rolling pass (referred to as the HRDSR ZK60H, hereafter). In both cases, defects, such as edge cracks or surface cracks, were not formed.

According to the computer simulation results [19] performed for AZ31 Mg alloy under the similar experimental conditions used in this study, rapid temperature rise in a deforming body took place in the case where the cold sheet (298 K) and hot rolls (473 K) were used, due to heat generation by friction and severe plastic deformation. At the beginning of plastic deformation, temperature of the sheet was much lower than for the case where the hot sheet (473 K) and hot rolls (473 K) were used, but they became similar near the exit (630 K). In both cases, effective strains reached the similar level of ~3.6, which is comparable to that obtained by ECAP after 4 passes.

For tensile testing to evaluate the mechanical properties at room temperature, dog-bone specimens with a gauge length of 32 mm and gauge width of 6 mm were cut from the rolled materials along the rolling direction (RD). The testing was conducted at an initial strain rate of 10^{-3} s^{-1}. For tensile testing to evaluate the superplasticity at elevated temperatures, dog-bone specimens with a 5 mm gauge length and gauge width of 4 mm were used. The tensile testing was carried out at temperatures between 473 K and 593 K at different strain rates under constant cross-head speed conditions. Strain-rate change (SRC) tests were performed in tension between 453 K and 593 K to measure the strain-rate sensitivity that differed depending on the investigated temperature and strain-rate ranges. A strain of ~0.03 was given between the strain rates following the pre-straining of 0.15. In each tensile test, a tensile specimen was heated to the test temperature within 5 min and was then held for an additional 5 min prior to the tensile loading. Vickers microhardness was measured using an Akashi MVK-H3. A load of 100 g was applied for 10 s for the measurements.

Optical microstructures were observed in the longitudinal sections (the normal direction (ND)–RD planes) of the samples after etching in a solution of picric acid (4.2 g), water (10 ml), acetic acid (10 ml) and ethanol (70 ml). Phase analyses were performed with a X-ray diffractometer (XRD) using Cu (K-alpha) source. Electron back-scatter diffraction (EBSD) analyses of the HRDSR ZK60C and HRDSR ZK60H were conducted on the ND–RD sections with a scanning step size of 50 nm. For the as-extruded ZK60 plate, a scanning step size of 1 μm was used. To identify a thickness layer of the sheet, a parameter $s = \pm 10\Delta x/(d_0/2)$ was introduced, where Δx represents the distance between the respective layer and sample center and $d_0/2$ is the half-thickness (i.e., $s = 0$ for the center layer, $s = 10$ for the upper surface layer in contact with the fast speed roll and $s = -10$ for the lower surface layer in contact with the slow speed roll). The top ($s = 9$), middle ($s = 0$) and bottom ($s = -9$) regions were examined in the HRDSR ZK60C and HRDSR ZK60H, and the middle region ($s = 0$) was examined in the as-extruded ZK60. The TSL version 5.31 was used as the EBSD analysis software, and data points with confidence index values lower than 0.1 were removed. The mean grain sizes of the HRDSR-processed alloys were determined by EBSD using a grain tolerance angle of 15°.

The sections for TEM observation were made along the ND–RD and the transverse direction (TD)-RD planes of the HRDSR ZK60C and the HRDSR ZK60H. The TD-RD samples were mechanically polished and then thinned by jet polishing, followed by ion milling. Field emission TEM (JEM 2001F, 200 kV) with energy dispersive spectroscopic (EDS) system was used for imaging microscopic features and identifying the precipitates. Solutions composed of 60% methyl alcohol (CH_3OH), 30% glycerin ($C_3H_8O_3$), and 10% nitric acid (HNO_3) were utilized for the jet polishing, and a BAL-TEC RES 101 operated at 4 kV and a tilting angle of 6° was used for ion milling. The ND–RD TEM samples, on the other hand, were mechanically polished and then thinned to perforation by ion milling. The particle distribution in a TEM image of the HRDSR ZK60C was analyzed by using an image analysis software (Olympus analySIS TS Material®).

3. Results

3.1. Microstructures and hardness

Fig. 1(a)–(c) shows optical micrographs of the as-extruded ZK60, the HRDSR ZK60C and the HRDSR ZK60H taken at the

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