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Microstructure and superplasticity of the as-cast Mg–9Al–1Zn magnesium alloy after high-ratio differential speed rolling

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ABSTRACT

The direct application of high-ratio differential speed rolling (HRDSR) on the cast microstructure of Mg–9Al–1Zn (AZ91) alloy resulted in significant refinement of grain size and divorced eutectic β-phase with semi-network structure. Nanosized β-phase particles (50–100 nm) precipitated at grain boundaries and within grains from the Al-rich α-phase, which formed as a result of fragmentation and decomposition of the *divorced eutectic* β-phase during severe plastic deformation by HRDSR. The HRDSR-processed AZ91 alloy exhibited excellent superplasticity at low temperatures below 573 K. The optimum superplasticity (with a maximum tensile elongation of 830%) was achieved at 573 K at 1×10^{-3} s^{-1}. Beyond 573 K, serious deterioration in superplasticity occurred due to a loss of thermal stability of grain size caused by the dissolution of the fine β-phase particles into the matrix. Models that can explain the high-temperature flow behavior of the ultrafine grained AZ91 alloy were proposed.

1. Introduction

The as-cast microstructure of the Mg–9Al–1Zn (AZ91) alloy has a high volume fraction of the β-$Mg_{17}Al_{12}$ phase that covers the grain boundaries and interdendritic interfaces in a continuous manner [1,2]. The network structure of the β phase is beneficial for improvements in the corrosion resistance due to its role as a barrier against corrosion attack [1,2], but results in poor ductility because it is hard and brittle at room temperature, thereby providing a path for crack or cavity nucleation and growth [3]. Many researchers have tried to improve the room temperature mechanical properties (strength and ductility) and high temperature properties (superplasticity) of the AZ91 alloy by reducing the matrix grain size and breaking up the coarse β phase [3–16], and they found that severe plastic deformation (SPD) is effective in achieving this goal. SPD has been applied to the AZ91 alloy using the three different major methods. The first method is the application of a solid-solution heat treatment before SPD (by equal channel angular pressing (ECAP) [9,14], high ratio differential speed rolling (HRDSR) [15] or friction stirring [4]). Very fine β particles precipitated from the solutionized matrix during the process. The second method is a combination of extrusion (or hot rolling) and SPD for achieving ultrafine grains [3,8,16]. Matsubara et al. reported [8] that after hot extrusion followed by ECAP for two passes at 473 K, the initial grain size of the Mg–9%Al alloy prior to extrusion was reduced from 50 to 0.7 μm. Kim et al. [16] applied HRDSR to the extruded AZ91 alloy with micron size β-phase particles at 473 K and observed that intensive shear can plastically deform the β-phase particles and then break them into smaller particles. The third method is the application of SPD on the as-cast AZ91 alloy. This method is most cost effective because the number of processing steps can be reduced, but the refinement of coarse β phase to fine particles is challenging [6,10–13]. Chung et al. [6] applied ECAP to the as-cast AZ91 alloy at 593 K. The grain size of the alloy after six passes of ECAP was less than 10 μm. The yield stress of the alloy increased from 65 MPa to 135 MPa after the first pass of ECAP, but did not show much change with further ECAP. Friction stirring has been proved to be an effective method of refining the as-cast microstructure of AZ91 alloy [10–13]. Depending on the cooling media used during the process, a grain size between 0.5 and 8 μm could be achieved. During the process, the coarse β-phase particles were broken into small particles and uniformly dispersed over the matrix.

In this study, we applied HRDSR to an as-cast microstructure of AZ91 alloy for exploring the possibility of refining its microstructure to a high level. Superplasticity of the HRDSR-processed AZ91 alloy was examined and the models that can explain its high-temperature flow behavior were proposed.

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2. Experimental methods

The present alloys were fabricated using the following procedures. The cast billets were prepared by melting commercial purity Mg and Zn along with Mg–Al alloy in an electric resistance furnace using a steel mold with a cylindrical cavity 98 mm in diameter and 120 mm in height under the protection of the mixed gas of CO_2/0.5 wt% SF_6. After holding for 10 min at 963 K, the melt was poured into a steel cylindrical mold with an inside width of 90 mm, a length of 160 mm and a thickness of 40 mm. The plates, with thicknesses of 4 mm, were cut out from the cast billet and then subjected to equal speed rolling (ESR, i.e., conventional rolling) at 673 K with 3 passes. The 2 mm thick sheet was then subjected to HRDSR for an additional 2 passes at a speed ratio of 2. The final thickness of the sheet was 0.5 mm. Between every rolling pass, the samples were reheated in a furnace at 673 K for 10 min. The surface temperature of the rolls was maintained to be 473 K through the entire rolling process by using the heat elements embedded beneath the surface of rolls.

The microstructures of the as-cast and HRDSR-processed AZ91 alloy were examined by optical microscopy (OM), scanning electron microscopy (SEM) and electron probe micro-analysis (EMPA) after grinding, polishing and etching using a solution of 100 mL ethanol, 6 g picric acid, 5 mL acetic acid and 10 mL water. The microstructure of the HRDSR-processed AZ91 alloy was also examined in detail using a field emission transmission electron microscope (FETEM) (JEM 2001 F, 200 keV) equipped for energy dispersive X-ray spectroscopy (EDS). The samples were jet-polished with a solution composed of 60% methyl alcohol (CH_3OH), 30% glycerin ($C_3H_8O_3$) and 10% nitric acid (HNO_3), and then ion milled.

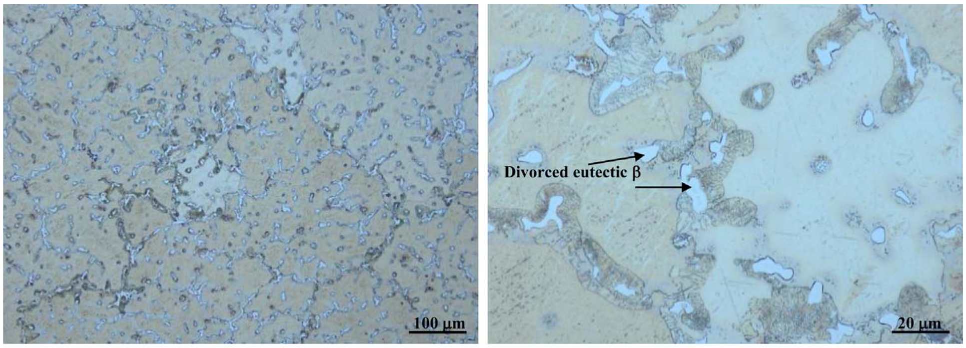
Room temperature tests were conducted on the as-cast and HRDSR-processed samples with dog-bone geometries using a 10-mm gauge length, 4-mm width and 4-mm shoulder radius (the gauge length was parallel to the rolling direction) at an initial strain rate of $1 \times 10^{-3}\ s^{-1}$.

The strain rate change (SRC) tests at high temperatures were performed in the strain rate range between 10^{-4} and $10^{-2}\ s^{-1}$ at 473–653 K. For tensile testing, the tensile specimens with a dog-bone geometry and a 5-mm gauge length were used. A pre-strain of ~0.15 was imposed at an initial strain rate of $1 \times 10^{-3}\ s^{-1}$. After the prestraining, the strain rate was reduced to 10^{-4} and then increased up to $10^{-2}\ s^{-1}$ through several steps. A plastic strain of ~0.03 was applied between the strain rates followed by the pre-straining. The SRC test at each temperature was consecutively repeated three times to confirm that the strain-rate–stress relation obtained from the SRC test was reliable. Through the third round, the accumulated strain was 0.64. The elongation-to-failure tests were conducted at 473 – 653 K and at initial strain rates of 5×10^{-4}, $1 \times 10^{-3}\ s^{-1}$ and $1 \times 10^{-2}\ s^{-1}$. In both the elongation-to-failure and the SRC tests, the tensile jig was preheated to a set testing temperature, and the sample was then mounted onto the sample holder in the heated jig. In total, a 5 min period was required to reach the testing temperature again. Then, the sample was allowed to equilibrate at the testing temperature for an additional 5 min before initiating tensile straining.

3. Results

Fig. 1**(a)** and **(b)** show the typical microstructures of an as-cast AZ91 at different magnifications, in which a high volume fraction of the divorced eutectic β-$Mg_{17}Al_{12}$ phase is distributed along the grain and dendrite interfacial boundaries. The $\alpha+\beta$ lamellae, which may have formed on the divorced α eutectic phase during cooling to room temperature after solidification, interconnects disconnected β phase, leading to the formation of a continuous network of β phase along the boundaries. The grain size determined by using electron backscattering diffraction was 53 μm (not shown here). According to the EDS analysis result, the average concentrations of aluminum in the β phase and the as-cast matrix were 42.5 and 2.8 wt. %, respectively. The EMPA elemental mapping of Al, Zn and Mn for the as-cast AZ91 (Fig. 2) indicates that Zn is also enriched in β phase, suggesting that a part of Al in $Mg_{17}Al_{12}$ is substituted by Zn to form $Mg_{17}(Al, Zn)_{12}$ [17].

Fig. 3(a)–(d) show the SEM micrographs of the HRDSR-processed (HRDSRed) AZ91 alloy viewed on the transverse planes at different magnifications. The following results were observed. First, the microstructure was highly refined after HRDSR. As a result, the coarse β phase almost disappeared, only leaving a small amount of residue (marked as 'C'). The refined microstructure was macroscopically composed of two regions (Fig. 3**(a)**). One region was almost free of particles (marked as 'A') and the other region contained many particles (marked as 'B'). Fig. 3**(b)** shows that the 'B' region was filled with thin and strip-like (sliced by shear) β phase (marked as 'C'), which were the remnants of the divorced eutectic β phase, and surface water droplet-like particles with sizes of 3–5 μm (marked as 'D'). At higher magnifications (Fig. 3**(c)** and **(d)**), it is recognized that many fine β particles with a typical size of 0.2–0.5 μm (marked as 'E') were dispersed within these droplet-like particles (marked as 'D'). According to the EPMA mapping results on the HRDSRed AZ91, shown in Fig. 4, and the EDS analysis results from Fig. 4 (Table 1), the droplet-like particles were Al-rich α phase with Al content of ~ 9 wt%. The matrix also had a high concentration of Al (7.0–8.0 wt%). The Zn distribution in the matrix was uniform (Zn mapping in Fig. 4). In the previous study [16], it was found that during HRDSR on the extruded AZ91 alloy with micron-sized β-phase particles, the β phase was



(a) (b)

Fig. 1. SEM micrographs of the as-cast AZ91 alloy at a (a) low magnification and (b) high magnification.