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Dynamic recrystallization kinetics and microstructure evolution of an AZ91D magnesium alloy during hot compression

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ABSTRACT

The flow behavior of as-cast AZ91D magnesium alloy was evaluated by isothermal compression tests within the temperatures of 220 °C, 260 °C, 300 °C, 340 °C, and 380 °C, and the strain rates of 0.001, 0.01, 0.1, 1 and $10s^{-1}$. Based on the stress-strain curve and hyperbolic sine function, the thermal activation energy and the strain rate sensitivity of the material were determined to be 142.2726 kJ·mol^{-1} and 0.142, respectively. The S type evolution of the dynamic recrystallization with the increasing strain that dynamic recrystallization (DRX) model obtained from stress and strain data. There was a good consistency between the proposed DRX kinetics model and microstructure observation results. Combining isothermal compression specimens with finite element and scanning electron microscopy (SEM) analysis indicated that the sample can be divided into rarely recrystallized region, partially recrystallized region and completely recrystallized region, which falls within the dense deformation zone, free deformation zone and severe deformation zone, respectively. The different forms of the β-$Mg_{17}Al_{12}$ phase induced by compression tests were confirmed by SEM. Nucleation mechanisms of the DRX were discussed with reference to the transmission electron microscope (TEM) analysis results.

1. Introduction

Because of their low density, high strength ratio, good impact load resistance, and shock absorption, magnesium alloys are widely used in aerospace, automotive, and chemical fields [1–4]. Magnesium alloys have densely packed hexagonal close-crystal (HCP) structures with low stacking fault energy, with fewer sliding and shifting systems at room temperature. Therefore, magnesium alloys processed at low temperature are liable to crack. At present, commercial components of magnesium alloys are usually fabricated by die-casting, while only 10% are fabricated by forging. However, as-cast magnesium alloys have low strength due to defects such as eutectic structure, porosity, voids, microcracks, and coarse grains. Therefore, microstructure and mechanical properties of the as-cast magnesium alloys are generally improved by secondary processing [5,6].

Wrought magnesium alloys are mainly divided into Mg-Al-Zn, Mg-Zn-Zr, Mg-Mn, Mg-Li, and Mg-Re categories according to chemical composition. Recently, Mg-Re alloys have been received great interest because of their high strength, good creep resistance and thermal stability. It has been confirmed in literatures that the rare earth element has a significant effect on the microstructure refinement and mechanical properties improvement of the magnesium alloys [7–9]. A Mg-3Gd-3Al-1Zn alloy was prepared by B. Pourbahari et al. [10]. They found that the original coarse microstructure with an average grain size of 1250 μm was refined to a fine and uniform microstructure with an average grain size of 36 μm by adding Gd in the as-cast Mg alloy, and the tensile strength and elongation was improved by 60% and 250%, respectively. The mechanical behavior of the ZK60-(0–3%) RE alloy was investigated at 300 °C–420 °C by C.J. Ma et al. [11]. Their results showed that the optimal tensile properties were obtained by adding 3% RE into the as-cast ZK60 alloy with a subsequent extrusion process. Compared with the as-received ZK60 alloy, the ultimate tensile strength of the extruded ZK60–3%Re alloy increased from 290 MPa to 340 MPa, and the yield strength increased from 228 MPa to 324 MPa. The Mg-Al-Zn alloys are also a typical class of the wrought magnesium alloys, which have good workability, mechanical properties, and comprehensive performance, and therefore have many industrial applications [12]. The as-cast AZ91 magnesium alloy has much higher specific strength and corrosion resistance compared with pure magnesium [13]. Strengthening β-$Mg_{17}Al_{12}$ precipitates distributed along the boundaries and in the interior of α-Mg matrix with a body-centered cubic (BCC) crystal structure.

During the hot processing, a large number of dynamic recrystallized grains develop due to severe strain of the deformed structure, which

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can effectively refine the coarse as-cast structure by eliminating porosity, voids, and microcracks defects. Critical conditions in dynamic recrystallization (DRX) are difficult to determine experimentally. Recent dynamic recrystallization kinetic models are more accurate in predicting the recrystallized volume fraction of the alloy in thermal deformation. The dynamic recrystallization kinetic model of as-cast AZ61 and AZ91D was established by the thermal deformation test conducted by Xu et al. [14,15], which is consistent with the actual dynamic recrystallization volume fraction. The dynamic recrystallization kinetic model was modified and proposed by O. Beltran [16]. In their subsequent testing of 304 L stainless steel, the model predictions are in good agreement with the experimental observations under different conditions. In the as-cast AZ91D alloy, dynamic recrystallization provides effective material refinement by continuous and discontinuous nucleation [17], which increases the strength of the material. Dynamic recrystallization kinetic model provides a convenient prediction of recrystallization.

We used the as-cast AZ91D magnesium alloy after homogenization treatment as the original material. The aim was to investigate the dynamic recrystallization behavior of the homogenized AZ91D magnesium alloy during the hot deformation by applying isothermal compression tests at various deformation temperatures and strain rates. The constitutive equation of the material was obtained based on stress-strain data under different conditions. The kinetics model describing the DRX behavior was proposed by analyzing the work hardening rate curves to predict the softening behavior induced by DRX. The variations of the DRX volume fraction affected by increasing strains were quantitatively analyzed, and the consistency between the predicted and experimental results was discussed. Nucleation mechanisms of the recrystallized structure during the isothermal compression process were clarified.

2. Materials and Experimental

The as-cast AZ91D magnesium alloy with chemical composition of 9.19 Al-0.623 Zn-0.254 Mn-0.0253 Si-0.0015 Fe-0.005% Cu-0.001 Ni-0.001Be (wt%) was used as the raw material in this study. The as-cast ingots were homogenized in a LK-JS vacuum resistance heating furnace at 420 °C for 5 h with a heating rate of 5 °C/s, and then were cooled down to room temperature by adopting a furnace-cooling method. The OM images and XRD patterns of the As-cast and the homogenized alloys are shown in Fig. 1(a)–(d).

The as-cast microstructure is composed of matrix α-Mg and second β-$Mg_{17}Al_{12}$ phases with a mean grain size of 120.72 μm (Fig. 1a and b). Some second phases with network structure are located along the grain boundaries of the dendritic matrix, and others are distributed within the dendritic grains (shown as the bright white ones). No obvious grain boundaries are discovered in the as-cast structure because the α-Mg grain boundaries are covered by the eutectic phase. It is quite similar to the microstructure of as-cast AZ61 alloy reported by AliakbariSani et al. [18]. Fig. 1c and d reveals that massive lamellar β phases were developed from the initial coarse β-$Mg_{17}Al_{12}$ phases in the homogenized AZ91D alloy, which are distributed inside the matrix and grain boundaries. The precipitated phases dissolve into the matrix during solid solution process, leading to the formation of supersaturate solid solution. Then the β phases reprecipitate from the supersaturated solid solution and appear inside the matrix and on grain boundaries through continuous precipitation (CP) or discontinuous precipitation (DP) behavior. With the decrease of temperature, the solid solubility of alloying elements in α-Mg matrix decrease. Precipitates discontinuously nucleate and grow at intersections between second phase and near-equilibrium matrix with large angle boundaries. This heterogeneous reaction leads to the formation of lamellar structure on both sides of migrated grain boundaries (Fig. 1c). During the aging process, CP forms in all the remaining regions of the supersaturated matrix, and the precipitates are evenly dispersed in α-Mg crystals (Fig. 1c) [19,20].

Cylindrical compression specimens were machined with a height of 15 mm and a diameter of 10 mm after aging treatment. Before compression tests, two ends of the samples were polished with #1500 sandpaper, and graphite plates were attached at both ends to reduce the friction between the sample and punch. The side of the specimen was polished to remove the surface oxide layer. Two platinum crucibles were welded in the middle region of the polished side surface for measuring the temperature. The compression tests were carried out on a Gleeble-3500 thermo-mechanical simulator in the temperatures range of 220°–380 °C with an interval of 40 °C and the strain rate range of 0.001–10s^{-1}. The samples were heated to schedule temperature with a rate of 5 °C/s and kept for 180 s to eliminate thermal gradient (Fig. 2) after compression, the sample was immediately quenched to retain the deformed microstructure and then sliced along the axial section for cold inlaid. Inlaid specimens were sanded, mechanically polished, followed by etching with a solution of 5 g picric acid, 5 ml acetic acid, 10 ml water, and 100 ml ethanol. Finally, the optical microstructure, secondary electron (SE) images and TEM analysis were examined by Scope A1 Optical Microscope (OM) and Sigma 500 scanning electron microscope (SEM) and JEOL JEM-2010 TEM.

3. Results and Discussion

3.1. Constitutive Equation for Flow Behavior

The typical compression curve obtained at 220 °C and 0.001 s^{-1} is shown in Fig. 3. The flow stress curves can be divided into four stages: (I) linear hardening stage; (II) strain hardening stage; (III) softening stage; (IV) steady state stage. At the first stage, the alloy undergoes an elastic deformation governed by work hardening (WH), therefore the curve shows a linear increase. During the initial deformation, dislocations are aggregated and tangled, which leads the resistance to dislocation movement. The direct manifestation is shown that the flow stress values and the WH effect are increased continuously. As much larger strain is applied, the DRV gradually strengthens and the stress increases gradually to a critical value (corresponding to a critical strain of ε_c) with a power-law function.

At the second stage, when the critical strain is reached, the dislocation density for DRX nucleation is reached inside the alloy. Since the softening effect of DRX and DRV counteracts the WH effect, the stress gradually increases to the peak stress. At the third stage, the softening effect is enhanced when a large number of recrystallized nucleation (mainly) and DRV (secondary) are induced during the thermal deformation, and the stress curves exhibit slow drop in the leading role of strain softening. At the fourth stage, the DRX softening and WH effects compete with each other. A small fluctuation of the stress level is presented, and a dynamic balance is maintained (corresponding to a steady state stress of σ_{ss}).

The stress and strain curves obtained at various temperatures and strain rates are shown in Fig. 4. At a given strain rate, the flow stress gradually increases when the temperature is decreased. Increased temperature causes the decrease in the critical resolved shear stress (CRSS) of basal system, and the activation of prismatic and pyramidal slip systems [18]. At a constant temperature, the peak stress increases as the increasing strain rate increases. It is concluded that the AZ91D alloy is a positive strain rate sensitive material.

During the process of thermal deformation, the relationship between the flow stress, deformation temperature, and strain rate can be described by the hyperbolic sine law proposed by Sellars and Tegart [21]. The expression formula is

$$\dot{\varepsilon} = A[\sinh(\alpha\sigma)]^n \exp\left(-\frac{Q}{RT}\right) \tag{1}$$

It can also be expressed as the following formula: