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Hot workability of nanocrystalline AZ91 magnesium alloy

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A B S T R A C T

This study examined the hot deformation behavior and workability characteristics of nanocrystalline AZ91 Mg alloy by performing hot compression tests with a Gleeble-3800 machine. To this end, a nanocrystalline alloy powder with a crystallite size of 25 nm was synthesized via mechanical milling of a pre-alloyed AZ91 Mg alloy powder for 14 h. The mechanically milled (MM) AZ91 powder was subsequently cold pressed at 600 MPa into cylindrical compacts measuring 10 mm in diameter and 12 mm in height. Then, the powder compacts with a relative green density of 91% were hot-compressed at temperatures ranging from 150 °C to 500 °C and at true strain rates ranging from 0.001 s^{-1} to 10 s^{-1}.The true stress–true strain curves peaked at low strains, after which the flow stress increased moderately. Processing maps were developed for all of the hot compression tests at strains of 0.1, 0.5, and 0.8, which represented a safe deformation domain at deformation temperatures and strain rates in the ranges of 250–350 °C and 0.1–10 s^{-1}. The crystallite size of the nanocrystalline AZ91 Mg alloy hot-compressed within the aforementioned domain was measured to be 140 nm, which is considered very fine for Mg alloys and resulted in a high hardness value of 133 HV for the hot-compressed alloy.

1. Introduction

Mg alloys have received considerable attention as a structural material in recent years due to their interesting properties, such as low density, high strength-to-weight ratio, good damping characteristics, superior machinability, and excellent castability [1,2]. To date, most of the research and development on Mg alloys has been performed by the automotive industry; and die casting has been the main manufacturing route for Mg products because of the poor workability of Mg at room temperature, which is a result of its HCP crystal structure [3]. However, die cast Mg products have considerable disadvantages, such as pin holes, porosity, cold shuts, and low strength. In fact, the market for Mg products continues to grow, but many opportunities remain untapped because of the relatively low stiffness and strength of these products. Due to the limited number of slip systems of Mg and its correspondingly large Taylor factor, grain refinement remarkably improves the mechanical properties of Mg and its alloys. It is well known that the fine-grained Mg alloys exhibit an interesting combination of high strength and high ductility at room temperature and superplasticity at elevated temperatures [4]. Superplastic flow occurs by grain boundary sliding, with the sliding accommodated by some limited dislocation slip in the adjacent grains [5]. Consequently, the processing of fine-grained Mg alloys has drawn great interests in the past few years.

Mechanical properties of Mg alloys can effectively be improved by refining their grain structures through dynamic recrystallization (DRX), which generally occurs during hot deformation of these materials. Recently, grain refinement due to DRX during hot deformation has been demonstrated in pure Mg [6] and in conventional wrought Mg alloys such as Mg–Al–Zn [7–9] and Mg–Zn–Zr alloys [10,11]. The grain sizes of different Mg alloys processed by conventional forming processes, such as extrusion and rolling, and also by severe plastic deformation techniques, such as equal channel angular pressing (ECAP) and high pressure torsion (HPT), have been reported to range from 800 nm to 20 μm. However, it has been well established that nanocrystalline metallic alloys with grain sizes less than 100 nm generally exhibit significantly improved mechanical properties over their microcrystalline counterparts. So far, most of the research work on nanocrystalline materials has been carried out on face-centered cubic (FCC) and body-centered cubic (BCC) metals, and studies on nanocrystalline Mg alloys are relatively scarce.

Mechanical milling has proven to be an effective technique for processing nanocrystalline metallic alloys. Most of the previous works on the mechanical milling of Mg alloys concern the processing of Mg matrix composites [12–14] and high performance Mg-based hydrogen storage alloys [15–17]. A number of studies have also examined the microstructural characteristics, thermal

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stability, and mechanical properties of nanocrystalline Mg alloys processed by mechanical milling. It has been reported that the grain sizes of Mg and its alloys can be refined to 30–50 nm through mechanical milling, and nanocrystalline Mg alloys produced by high-energy milling show excellent resistance to grain growth during isothermal annealing and/or subsequent consolidation processes at high temperatures, leading to superior mechanical properties for the bulk alloys consolidated from mechanically milled Mg-based powders [18–22].

The powders processed by mechanical milling are then consolidated by powder metallurgy (PM) routes, such as cold pressing and sintering, powder extrusion, powder forging, and powder rolling. The surfaces of Mg-based powders are covered with a thin stable oxide layer, which strongly inhibits sintering of these powders. Hence, the sintering of Mg-based powders is generally regarded as unfeasible and problematic [23]. Compared with other PM routes such as sintering and hot pressing, the shear stresses involved in powder extrusion and powder forging make them ideal processes for the production of bulk Mg products from powder mixtures [24]. These shear stresses break the oxide layer covering the particle surfaces of Mg-based powders, which can lead to a well-bonded microstructure and superior after-consolidation mechanical properties. A good understanding of the hot deformation behavior of a material is extremely important in hot deformation processes such as extrusion and forging [25]. Thus, several studies have been performed to investigate the effect of processing parameters on the hot deformation behavior of microcrystalline Mg alloys.

Among all of the existing Mg alloys, AZ91 is the most widely used alloy in industry [26]. However, studies on the hot deformation behavior of AZ91 alloy are relatively scarce; and very limited data are available on this alloy's workability characteristics [27–29]. Furthermore, there are no systematic studies on the hot deformation behavior of nanocrystalline Mg alloys. Therefore, this study focused on the hot deformation behavior and workability characteristics of nanocrystalline AZ91 Mg alloy with a view to finding the optimum hot working parameters and evaluating the mechanisms of hot deformation. For this purpose, the approach of processing map has been adopted.

The processing map technique used in this study is based on the dynamic materials model (DMM), and the efficiency of the power dissipation (η) through microstructural changes during deformation is given by [30]:

$$\eta = 2m/(m+1) \tag{1}$$

$$m = (\partial \log \sigma)/(\partial \log \dot{\varepsilon}) \tag{2}$$

where m is the strain rate sensitivity of flow stress, σ is the flow stress, and $\dot{\varepsilon}$ is the strain rate. The extremum principles of irreversible thermodynamics as applied to the continuum mechanics of large plastic flow were explored to define a criterion for the onset of flow instability, given by:

$$\xi = (\partial \ln[m/(m+1)]/\partial \ln \dot{\varepsilon}) + m \leq 0 \tag{3}$$

2. Experimental procedure

The raw material used for this study was a pre-alloyed Mg–Al–Zn powder (Ecka Granules, Germany) with a chemical composition equivalent to that of AZ91 D Mg alloy (8.8 wt% Al, 0.6 wt% Zn, 0.2 wt% Mn, 0.03 wt% Si, and the balance Mg). The mentioned powder was mechanically milled in a horizontal attritor mill (CM01 Simoloyer, ZOZ, Germany) using the following milling parameters: ball-to-powder weight ratio: 20/1; ball diameter: 5 mm; ball material: AISI 420 stainless steel; milling time: 14 h; milling speed: 700 rpm; and milling atmosphere: Ar. Stearic acid (2 wt%) was also employed as the process control agent (PCA).

The mechanically milled (MM) AZ91 powder was uniaxially cold pressed at 600 MPa into cylindrical billets measuring 10 mm in diameter and 12 mm in height using no pressing lubricant. The green densities of the powder compacts were measured to be 91% of the theoretical value, or 1.80 g/cm^3.

A graphite foil with a thickness of 0.05 mm was placed between the ends of the powder compacts and the anvils to minimize friction during the hot compression test. Prior to the hot compression tests, the samples were resistance heated in an Ar. atmosphere to the required temperature at a heating rate of 3 °C s^{-1}, then maintained at the test temperature for 1 min to minimize thermal gradients along the sample. Single-hit compression tests were performed using a servo-controlled Gleeble-3800 system (Dynamic Systems Inc., USA) at strain rates of 0.001 s^{-1}, 0.01 s^{-1}, 0.1 s^{-1}, 1 s^{-1}, and 10 s^{-1} and deformation temperatures ranging from 150 °C to 500 °C, which cover the entire hot working range of Mg alloys. After hot compression, the samples were air-quenched to room temperature. The load–stroke data were converted into true stress–true strain curves using standard equations, which in turn used to compute the efficiency (η) and instability (ξ) parameters as a function of temperature and strain rate.

Scanning electron microscopy (SEM) and X-ray diffractometry (XRD) were employed for the characterization of the as-received and MM AZ91 powders. Besides, differential scanning calorimetry (DSC) study was conducted on the MM AZ91 powder using a simultaneous thermal analyzer (STA) 6000 manufactured by Perkin–Elmer. The MM AZ91 power was heated in an alumina pan at a rate of 5 °C/min up to 500 °C under flowing Ar. For baseline subtraction, the empty alumina pan was initially tested in the same fashion.

The densities of the compacts hot-compressed to different strain levels were measured by the Archimedes method in accordance with the MPIF 42 standard. The relative densities of hot-compressed specimens were calculated by dividing the measured densities by the theoretical density of MM AZ91 powder, i.e. 1.8 g/cm^3. The hot-compressed specimens were then sectioned in the center parallel to the compression direction for the microstructural and mechanical characterization. The sectioned samples were then ground, polished, and examined using SEM, XRD, and Vickers hardness testing (HV1).

3. Results and discussion

3.1. Starting microstructure

Fig. 1(a) illustrates the cross-section microstructure of an AZ91 Mg alloy powder particle, showing that the microstructure of the as-received AZ91 powder was comprised of a dark grey matrix in which a grey precipitate was randomly distributed. The EDS analyses (data not shown) determined that the dark grey matrix was enriched in Mg while the grey precipitate was enriched in Mg and Al. As the atomic numbers of Mg and Al are 12 and 13 respectively, SEM in backscattered electron (BSE) mode was not capable of making an obvious contrast between the matrix of powder particle and the precipitate containing Al. As a result, the grey precipitate is highlighted using a black oval. After the milling process, no second phase precipitates were visible in the microstructure of the MM AZ91 powder (Fig. 1(b)), and the particles of the MM AZ91 powder possessed a grey matrix with no specific microstructural feature.

The XRD patterns of AZ91 and MM AZ91 powders are presented in Fig. 2. The microstructure of AZ91 powder particles was composed of the β-$Mg_{17}Al_{12}$ precipitates and the α-Mg solid solution matrix. This structure is typical of AZ91 castings [31]. The diffraction peaks of the β-$Mg_{17}Al_{12}$ phase are barely detectable in the XRD pattern of MM AZ91 powder, which can be related to the dissolution of the β-$Mg_{17}Al_{12}$ phase in the α-Mg matrix of the powder particles. Another possibility is that mechanical milling caused the β-$Mg_{17}Al_{12}$ phase to be refined into very fine dispersoids distributed in the α-Mg phase, which are hardly detectable by XRD. The milling process was also found out to affect the intensities and widths of the diffraction peaks of the α-Mg phase, and the mentioned peaks became weaker and wider through mechanical milling. This phenomenon can be attributed to the reduction in particle size, the refinement of crystallite size, and the enhancement of lattice strain, all promoted by the severe plastic deformation of AZ91 powder particles during mechanical milling. The grain sizes of AZ91 casting products normally range between 10 μm and 150 μm [31,32]. The AZ91 powder used for this study had been