PHYSICAL METALLURGY AND HEAT TREATMENT

Relationships between the Process Conditions and Microstructures Evolution for Extrusion-Shear of Magnesium Alloy¹

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Abstract—It is widely recognized that grain refinement has significant influences on the strength and ductility of metals. To improve the industrialization of the severe plastic deformations (SPD) technologies for magnesium alloy, a new extrusion-shear (ES) method has been explored and widely investigated because it can manufacture ultra-fine grained microstructures in magnesium alloys. It is crucial to research the effects of process parameters on the deformation behaviors of ES process. Three-dimensional (3D) finite element modeling of ES processes with different billet temperatures and channel angles have been researched. Different billet temperatures have been regarded as the initial conditions in DEFORMTM-3D software. The strain rates evolutions have been gained with different ES process conditions. The microstructure evolution has been analyzed by the simulation and experimental results. The decreases of billet temperatures and channel angles could improve the grains refinements of magnesium alloys which have been prepared by ES process. The simulation and theoretical analysises results showed that reasonable billet temperatures and channel angles could promote grains refinements. Research results could present the fundamentals and practical guidelines for the designs of ES dies and optimization of process parameters to manufacturing high-performance magnesium rods.

Keywords: magnesium alloys, microstructures, grains refinement, billet temperature, channel angle **DOI:** 10.3103/S1067821215040069

1. INTRODUCTION

Magnesium (Mg) alloy is a light, power-saving and recyclable alloy which is 35% and 77% less dense than those of aluminum alloy and steel, respectively. Therefore the use of Mg alloy has gradually increased in automotive and aerospace industries [1, 2]. In recent years bulk microstructure materials processed by methods of severe plastic deformation (SPD) have attracted the growing interest in materials processing. Previous studies have reported some SPD methods to reduce grain size and improve the formability of Mg alloys including equal channel angular rolling [3], accumulative back extrusion [4], and friction stir processing [5]. Gong et al. [6, 7] applied the different speed rolling for the manufacturing ZK60 and Mg-4.5Al-1.0Zn alloys and the large plastic deformation leads to the great grain refinement and subsequent superior mechanical properties of Mg alloy thin sheets. For the manufacturing of extruded components, equal channel angular extrusion (ECAE) is effective to process Mg alloys components owing to great refinement by shear stress, in which as the crosssection of the material remains unaltered it can be processed over and over again to produce large plastic strains. Matsuyama et al. [8] used a new processing procedure to extrude a cast Mg-9% Al alloy involving the sequential application of extrusion and ECAE. Experiments showed that the Mg-9% Al alloy has an initial grain size of $\sim 50 \ \mu m$ after casting but this was reduced to $\sim 12 \ \mu m$ after extrusion and it was further reduced to ~ 0.7 µm when the extruded alloy was subjected to ECAE for 2 passes at 473 K. Although the cast alloy exhibits extremely limited ductility and the extruded alloy was only moderately ductile, it was demonstrated that processing by EX-ECAE (extrusion firstly and many ECAE steps following) could produce excellent ductility including the occurrence of both low temperature superplasticity and high strain rate superplasticity, but the ECAE process was only used in the lab scale processing and preparation for nano-crystalline material, there exit

an unbridgeable gap between the experimentation and applications of industry [9]. In addition, the EX-ECAE usually includes more than 2 steps, and the Mg alloy material may be oxidized. The authors' research team has engaged in the research on extrusion-shear (ES) process by experiments and simulation since 2008 [10], and the technology has been granted patents for state inventions. Optimum billet temperatures and die structures with channel angles for ES process could obtain satisfactory mechanical properties of the extruded products. The effects of bil-

¹ The article is published in the original.



Fig. 1. 3D Finite element model of ES dies and billet.

let temperatures and die structures on deformation characteristics can play a significant role in the formation of ultra-fine grains (UFG) for Mg alloy by affecting the dislocation density and evolution of dislocation cells and their subgrain structures [11, 12]. Finite element method (FEM) can be used to reduce the amount of try-out to manufacture the acceptable products.

In the present research, attempts have been made to simulate the ES process which would manufacture Mg alloy rods. The main objective of this study is to investigate the influences of process parameters on microstructures evolutions during ES process. The components of ES die have been designed and manufactured and installed to industrial horizontal extruder. The microstructures of AZ31 Mg alloy samples from ES have been observed. The geometrical and numerical models based on simulation theories have been built to predict the evolution of the strain rates by using DEFORM-3D software. The simulation could characterize the thermo-mechanical response of Mg alloy during ES process in term of strain rates affected by different billets temperatures and channel angles. The experimental results of ES are also compared with the simulation results with different conditions.

2. SIMULATION CONDITIONS

Figure 1 presents the schematic diagram of the ES process including direct extrusion and two

continuous simple shearings. An oblique angle Φ between two channels is called die channel angle, which is 135° or 120° in this research. ES process not only makes continuous forming possible but also allows the various kinds of rods into the desired final dimensions. The material for Mg alloy AZ31 is considered as a plastic body during the ES process. And the punch and die are assumed to be rigidity. The punch is moved in X-axial direction. The geometries of the billet and tooling are selected based on an actual extrusion condition. The simulation parameters applied in FEM are shown in table.

The flow stress-strain data of the AZ31 alloy are obtained through hot compression tests using Gleeble1500D. To take the effect of deformation heating during hot compression at strain rates, a set of flow stress-strain curves have been done including the actual specimen temperature with a range of $250-400^{\circ}$ C and a strain rate range of $0.01-10 \text{ s}^{-1}$. The flow model of stress is implemented in the

commercial finite element code DEFORMTM-3D using the user subroutine, in which the yield stress is dependent on the strain, strain rate and temperature. Contact boundary conditions have been applied to the nodes of billet in order to specify the contact between those nodes and the surface of ram. The friction between workpiece/tool interfaces has been considered to be of shear-type.

Simulation parameters

Preheat temperature for billet (°C)	420, 400, 370
Preheat temperature for ES die and punch (°C)	400, 380, 350
Billet length (mm)	250
Billet diameter (mm)	80
Container insider diameter (mm)	82
Container outside diameter (mm)	90
Die bearing length (mm)	5
Channel angle (°)	135, 120
Extrusion ratio	11.6
Ram speed (mm/s)	20
Friction factor of the container-billet interface	0.4
Friction factor between the billet and die.	0.4
Heat transfer coefficient between tooling and billet $(N/^{\circ}C \text{ s mm}^2)$	11
Heat transfer coefficient between tooling/billet and air $(N/^{\circ}C \text{ s mm}^2)$	0.02
ES Die material	H13



Fig. 2. Strain-rates distribution at time 3.03 s with ES die channel angle 135° and different billet temperatures (a) 370° C, (b) 400° C, (c) 420° C.

3. EXPERIMENTAL PROCEDURE

In order to validate the results of FEM, ES dies have been designed and manufactured to perform the actual ES processes, cone-shaped ES dies with two shearing and different channel angles have been designed and manufactured. ES experiments have been carried out to analyze the results obtained from computer simulation. Real ES experiments have been carried out by employing a 300 ton horizontal extrusion press with a resistance heated container and a heater. The die material, die dimensions, billet dimensions and extrusion conditions were set as the same as those used in numerical simulation, as described in table. The billets have been heated in an external furnace up to 420°C, 400°C, and 370°C respectively. The ram speed was kept as 20 mm/s during experimental verification. The casted AZ31 billet was used in this study. All the observation samples have been taken from the center of extruded rods. Microstructures in the as-received, and ES extruded materials have been examined following standard metallographic procedures. The polished surface has been etched using either a solution of 1 vol % HNO₃, 24 vol % C₂H₆O₂, and 75 vol % H_2O , or of 10 mL acetic acid, 4.2 g picric acid, 10 mL H_2O , or 70 mL ethanol. Microstructure observations have been carried out using PME OLYMPUS TOKYO-type optical microscope (OM). The average grain size has been measured by line-intercept method [13].

4. RESULTS AND DISCUSSIONS

Effects of Billet Temperatures on Strain of Mg Alloy Billets

The strain rate for ES process is directly related to the ram speeds and billet temperatures. The process parameters of ES process have a lot to do with strain rates. The predicted strain rates provide quantitative insight into the deformation behaviors of billets to gain the deformation characteristics of ES deformation with different billet temperatures.

The effective strain rates distributions caused by ES die with channel angle 135° are illustrated in Fig. 2 and billet temperatures are 370°C and 400°C and 420°C, respectively, which provide the important information regarding the strain rates distribution. The strain rate



Fig. 3. Microstructures of AZ31 Mg processed by ES with channel angle of 135° with different billet temperatures (a) 370° C, (b) 400° C, and (c) 420° C.

distributions for different preheated temperature of billets are significantly different and not even. The maximum strain rates slightly decrease with the rise of billet temperatures. The deformation of the initial extrusion is nonuniform and the highest strain (65.7 s⁻¹) in Fig. 2a and bigger than which of the other two figures. The shear deformation can be obtained at the intersection of the two channels. It indicates that the largest strain still exists in the corner region, where the simple shear occurs. The billet temperatures of billets have significant effects on the strain rates distribution in the rods of Mg alloy.

The Levy–Mises flow rule can be used to relate the stress to the strain rate during bulk metal forming. This flow rule is shown in equation (1). The equation describes the relationship between the stress and strain rate.

$$\sigma_{ij} = \frac{1}{\dot{\lambda}} \dot{\varepsilon}_{ij}.$$
 (1)

Where σ_{ij} and ε_{ij} are flow stress and strain rate for point *ij* respectively. The coefficient λ is a constant.

Extrusion temperature would affect the amount of flow stress. The flow stress of Mg alloy decreases with the increase of temperature, and strain rate decreases with drop of flow stress from equation (1), so strain rates decrease with temperature rise.

Effects of Billet Temperatures on Grains Refinements of Mg Alloy

The AZ31 Mg alloy is worked under high pressure during ES extrusion where the occurrence of fracture is usually prevented. With the increase of preheated temperature of billets, the temperature of rod surface rises due to intense plastic deformation and storage heat in billets and frictional heat, especially at the surface where friction affects the crack phenomenon. This leads to damage mechanical properties of the rods and to formation of micro-cracks at the surfaces of the billet. So it is very important to choose the appropriate preheated temperature of billets and die to obtain ideal surface qualities under the condition that extrusion speed is high relatively, and preheated temperature of billets is appropriate, the capabilities of the equipment meet the requirements of force during ES process.

Microstructures for ES Extrusions with Different Billet Temperatures (370°C, 400°C and 420°C) and channel angle 135° are shown in Figs. 3a, 3b and 3c, respectively. There are no even equiaxed grains in ES hot-extruded rods as shown in Fig. 3. After the hot ES process, grains are refined and more homogenous. But in Figs. 3b and 3c, there are many original grains and their sizes are bigger than 100 μ m, while the size of fine grains is around 10 μ m owing to dynamic recrystallization (DRX), and the distribution of grains is nonhomogeneous. It is clear the DRXed grains have grown up.

Effects of Channel Angles on Grains Refinements of Mg Alloy

Strain-rates distributions at time 3.03 s with ES die channel angle of 120° and different billet temperatures of 400°C and 420°C have been shown in Fig. 4. It is obvious that the maximum strain rates caused by ES die with channel angle 120° is bigger than which caused by ES die with channel angle 135° at extrusion time 3.03 s by comparing the Figs. 2 and 4. The strain rates distributions for different channel angles of ES die are significantly different and not even. The maximum strain rates obviously decrease with the rise of channel angles. The deformation of initial ES process is nonuniform, and the highest strain (55 s⁻¹) in Fig. 4a. The shear deformation can be obtained at the intersection of the two channels. Distributions of the strains are lamellar with distinct deformation gradients in the deformation zone. The deformation of this position is close to the simple shear deformation. From the Figs. 2 and 4 the maximum strain rates increase with the drop of channel angles.

Microstructures for ES processes with different billet temperatures 370°C, 400°C and 420°C are shown in Fig. 5. There are even equiaxed grains with an average grain size of about 10 μ m in ES hot-extruded rods, as can be shown in Fig. 5a. It indicates that intensive and full DRX has taken place during ES process when the preheated temperature of billet is 370°C. After the



Fig. 4. Strain-rates distribution at time 3.03 s with ES die channel angle 120° and different billet temperatures (a) 400° C, (b) 420° C.



Fig. 5. Microstructures of AZ31 Mg alloy processed by ES die with channel angle of 120° at different temperatures: (a) 370° C, (b) 400° C, and (c) 420° C.

hot ES process, grains are refined and more homogenous. But in the Fig. 5b there are many original grains with the grain size more than 100 μ m, and the size of fine grains is around 10 μ m indicating of the happening of DRX. In addition, the size distribution of grains is nonhomogeneous. There are almost few fine DRXed grains exiting in the billet in the Fig. 3d for the DRXed grains have grown up. It is clear the ES process with higher billet temperatures grain growth rate is significantly bigger than the grain refinement rates caused by ES process.

It is concluded that average grain size of Mg alloy increase with billet temperature rise. It can be observed that the lower temperatures could inhibit the grains growth up further. Figures 3 and 5 show that the average sizes of microstructures for Mg alloy prepared by ES extrusion increase with the rise of channel angles. The smaller grain size attained from the lower temperature would help to acquire the more superior mechanical properties [14–16].

Theoretical Analysis for Strain Rates during ES Process

The strain rates ε_1 of two shearings for ES process are described in equation (2).

$$\dot{\varepsilon}_{1} = \left[\frac{2\cot\left(\frac{\Phi}{2} + \frac{\Psi}{2}\right) + \psi \operatorname{scs}\left(\frac{\Phi}{2} + \frac{\Psi}{2}\right)}{\sqrt{6}}\right] \qquad (2)$$
$$\times \frac{\sqrt{v_{2}}}{\psi R_{2}} \exp\left(\frac{Q}{RT}\right).$$

Where ϕ is channel angle, ψ outer channel angle, v_2 extrusion velocity during two shearings period.

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 R_2 radius of extruded rod, Q activation energy, and R gas constant, T extrusion temperature.

The relationship between the average recrystallization grain size (d) and the Zener-Hollomon parameter (Z) during DRX is given by equation (3) [17, 18].

$$\left(\frac{d}{d_0}\right)^n = 10^{-3} \times Z^{-1/3}.$$
 (3)

The temperature corrected strain rate Z is given by equation (4) [19-23].

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right). \tag{4}$$

Where $\dot{\varepsilon}$ is strain rate, Q is the activation energy for the deformation, T is the temperature and R is the gas constant.

It can be seen from the equation (2), (3) and (4) that average sizes of Mg alloy decrease with the rise of strain rates. The effective strain rates decrease with the rise of billet temperatures and channel angles. It is obvious that average sizes of grains for lower billet temperatures and smaller channel angles are finer than those prepared by ES process with higher billet temperatures and bigger channel angles.

5. CONCLUSIONS

FEM and experimental results for ES process are conducted. ES experiments with different channel angles and billet temperatures have been applied to fabricate AZ31 Mg alloy rod with ram speed of 20 mm/s. The microstructures of as-received from ES extruded rod have been observed. The results are summarized as follows.

(1) 3D FEM simulation of as-cast AZ31 Mg alloy billets subjected to ES extrusion with different billet temperatures and ES dies with different channel angles have been carried out successfully. The strain rates in the extruded rods have been predicted during ES extrusions.

(2) The strain rates of Mg alloy during ES decrease with the increase of billet temperatures and channel angles. The grain sizes of microstructures for Mg alloy decrease with the rises of strain rates, and increase with the rise of billet temperatures and channel angles for ES dies.

(3) The modeling results are in accordance with the experimental results. The modeling would able to predict the strain and microstructure for ES process.

ACKNOWLEDGMENTS

This work is supported by the open fund for Key Laboratory of Manufacture and Test Techniques for Automobile Parts (Chongqing University of Technology) Ministry of Education in 2003, and National Science Foundation for Distinguished Young Scholars of China (Grant no. 51101176) and foundation of the post doctorate in Chongqing City and Project Number is Xm201327, and China Postdoctoral Science Foundation funded project.

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