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Effect of grain size on cyclic microplasticity of ECAP processed commercial pure magnesium

G.D. Fan^a, M.Y. Zheng^{a,*}, C.H. Ju^a, X.S. Hu^a, K. Wu^a, W.M. Gan^c, H.G. Brokmeier^{b,c}

^a School of Materials Science and Engineering, Harbin Institute of Technology,
150001 Harbin, P. R. China

^b Institute of Materials Science and Engineering, Clausthal University of Technology,
D-38678 Clausthal-Zellerfeld, Germany

^c Institute of Materials Research, Helmholtz-Zentrum-Geesthacht (HZG), Outstation at FRM-II,
D-85748 Garching, Germany

* zhenghe@hit.edu.cn, Tel: 86-451-86402291, Fax: 86-451-86413922

Abstract

Equal channel angular pressing (ECAP) was performed on the extruded commercial pure magnesium at 250 °C for 4 passes. Heat treatments were carried out to modify the microstructures. The cyclic plastic deformation behavior of pure Mg with different grain sizes in microstrain region was studied by tensile loading and unloading experiments. The microplastic deformation process of pure Mg can be divided into two stages. In the first stage pronounced plastic deformation associated with dislocation motion on basal plane is initiated at several MPa. The materials are softened and characterized by low friction stresses and hardening exponents. The microplastic deformation enters into region II above the strain of about 8×10^{-4} . Annihilation and tangle of dislocations lead to the increase of hardening exponents and friction stresses. Pure Mg shows a very pronounced anelastic behavior during cyclic microplastic deformation, which results in a rapid increase of modulus defect, effectively decreasing the elastic modulus by up to 60%. Grain size has a marked effect on microplastic deformation behavior of pure Mg. With increasing the grain size, the specimen shows a more pronounced microstrain and anelastic behavior.

Keywords: Pure magnesium; ECAP; Cyclic loading; Microplasticity; Annealing; Grain size

1. Introduction

Microplasticity is one of the most important characteristics of structural materials. The microplastic strain determines various properties of the metals, such as fatigue property [1-3], dimensional stability [4, 5], and various relaxation phenomena, such as damping behavior [6, 7]. The microplastic domain of the mechanical behavior of structural materials can be defined as the region where the plastic strain is smaller than 0.2% [8-12]. However, the plastic strain amplitudes resulting in the fatigue failure of materials are sometimes only in the range of 10^{-5} – 10^{-4} , and are therefore generally smaller than the elastic strain amplitudes. Cyclic deformation leading ultimately to fatigue failure is thus always a sequence of repeated microyielding in tension and in compression [3]. Therefore, the researches on the factors determining the microplastic deformation resistance of metals with various processing history are of great importance, on the one hand, for understanding the microplastic deformation mechanism, and, on the other hand, for both static and dynamic mechanical design of safety

related components which may suffer repeated microplastic strain. Measurement of the plastic deformation amplitude is possible by loading and unloading in a constant extension rate tensile test. The construction of the complete mechanical hysteresis loop provides a convenient way to study the elasto-plastic deformation mechanism of structural materials in the microplastic region [13-15]. However, the nature and mechanism of the transition from micro- into macroplastic deformation have been studied to a much lesser extent.

The multiplicity of deformation modes of magnesium [16-18] makes it difficult to predict its microplastic deformation behavior, which is, therefore, still debatable. The cyclic microplasticity of Mg and Mg-Zn alloys was interpreted in terms of the partial reversal of $\{10\bar{1}2\}$ twins upon unloading by Caceres [19] and Mann [20]. Recently, Zhou [21] explained the microyielding and high damping of magnesium by invoking the motion of incipient kink bands (IKBs), which were fully reversible, dislocation-based loops. But the most commonly accepted microplastic theory of Mg and its alloys is still that the deformation occurs primarily by basal slip [13, 14, 22-24]. It was reported that microplastic deformation in polycrystalline Mg alloys was accommodated entirely by basal slip, which was independent of the initial texture [16, 23, 25]. However, the microplastic deformation process in magnesium, characterized by the movement, interaction and multiplication of the dislocations, is still ambiguous. And very few researches on the effects of microstructures, such as grain size, on microplasticity of magnesium are reported.

Equal channel angular pressing (ECAP) can modify the microstructures of Mg alloys, such as the grain refinement and texture modification [26, 27], and has great potential to improve the properties of Mg alloys, such as mechanical property [26, 27] and damping capacity [28, 29]. In the present study, cyclic loading and unloading tests were carried out on pure Mg after ECAP and subsequent annealing treatment. The aim of the present investigation is to gain some insight into the microplasticity, and to study the effect of grain size on microplastic deformation of pure Mg.

2. Experimental procedures

In this research, a rectangular billet for ECAP with a dimension of $10 \times 10 \times 60 \text{ mm}^3$ was machined from the as-extruded commercial pure Mg (99.90%). The ECAP die had characteristic angles $\Phi=90^\circ$ and $\psi=37^\circ$. The ECAP was conducted at 250°C for 4 passes using route B_C , in which the specimen is rotated by 90° in the same direction between each pass. The 4-pass ECAPed pure Mg were then annealed at 200°C for 60 min, 300°C for 30 min, 350°C for 15 min and 400°C for 10 min, respectively. The microstructures of the pure Mg were obtained in an Olympus DP11 optical microscope (OM). Transmission electron microscope (TEM) was carried out in a FEI-TECNAI G2 F30 operating at 200 kV. Neutron diffractometer at TEX-2 (GKSS Research Center, Germany) was used to measure the texture. A bulk sample with gauge of $10 \times 10 \times 10 \text{ mm}^3$ was cut from the center part of the samples for texture measurement.

Tensile tests were performed at room temperature using an Instron 5569 universal test machine at a crosshead speed of 10 MPa/min. Flat tensile samples of dog-bone shape with a gauge dimensions of $6 \times 2 \times 10 \text{ mm}^3$ were prepared. Tensile tests in smaller strain region were also carried out by dynamic mechanical analyzer (DMA)(TA Q800, USA) using tensile mode at a crosshead speed of 8 MPa/min with a strain sensitivity of 1×10^{-6} . The specimen dimension is $15 \times 2 \times 1 \text{ mm}^3$. All the tensile specimens were selected to be parallel to the

extrusion direction of the ECAPed billet.

3. Results and discussion

3.1 Microstructure

Fig. 1 shows optical microstructure of commercial pure Mg after ECAP and subsequent annealing treatments. The average grain size of the ECAPed pure Mg was measured to be about 6 μm (Fig. 1a). The grain boundaries are not clearly defined, which also can be seen from the TEM micrograph in Fig. 2(a), and the dislocation density is high due to the intense strain introduced by the severe plastic deformation. The grain size of the ECAPed Mg is slightly increased to about 15 μm after subsequent annealing at 250 $^{\circ}\text{C}$ for 60 min (Fig. 1b). The grain boundaries are well defined and the grains become equiaxed with low density of dislocations (Fig. 2b), indicating that dynamic recrystallization (DRX) takes place significantly during the annealing treatment. After annealing at 300 $^{\circ}\text{C}$ for 30 min, the grain size of the ECAPed Mg is increased to about 27 μm (Fig. 1c), and similar as the specimen annealed at 250 $^{\circ}\text{C}$, the material is characterized by low dislocation density and long dislocations (Fig. 2c). Due to the absent of second phase in pure Mg to inhibit the migration of grain boundaries, the average grain size is markedly increased to about 58 and 72 μm after annealing at 350 $^{\circ}\text{C}$ for 15min and 400 $^{\circ}\text{C}$ for 10min, respectively (Fig. 1d and e).

3.2 Texture evolution

Fig. 3 displays the complete pole figures of pure Mg after ECAP processing and subsequent annealing. For better visualization, the (0002) and $(10\bar{1}0)$ complete pole figures are presented on the extrusion direction (ED) - normal direction (ND) plane. For ECAP processed pure Mg, the strongest texture component locates at about 40 $^{\circ}$ from ND towards ED and 20 $^{\circ}$ from ND towards TD. In addition, a weak texture component with basal planes parallel to ED can be observed in as-ECAPed Mg. After annealing at 300 $^{\circ}\text{C}$ for 30 min and 400 $^{\circ}\text{C}$ for 10 min, the texture type of pure Mg keeps almost unchanged, except the slightly lower intensity.

3.3 Cyclic microplasticity

Fig. 4 shows the stress-strain curves of pure Mg with different grain sizes measured by DMA, the limit loading force of which is 18 N. It is observed that a deviation from the straight line occurs at a very small stress of about 3 MPa. And the tensile curves separate with each other at the stress between 4 and 7 MPa. The tensile stress decreases with the increase of grain sizes.

Fig. 5 shows cyclic tensile hysteresis loops for the ECAPed and annealed specimens. The cyclic tensile curves with strain larger than 5×10^{-4} were also measured by tensile machine, as shown in Fig. 6. The whole microplastic deformation behavior can be described by combining the both results. It can be seen in Fig. 5 that in loading with the stress below 3 MPa, the unloading and repeated loading curves coincide with a straight line, indicating that the material deforms in a fully elastic manner. This elastic range observed is related to the magnitude of a friction stress which resists the motion of the dislocations. Repeated loading with larger stress leads to the formation of elastoplastic hysteresis loops. The loops grow gradually with increasing the amount of plastic prestrain. For as-annealed pure Mg, larger stress-strain hysteresis loops are observed under cyclic loading after a small plastic strain.

The specimen annealed at 400 °C shows the most pronounced hysteresis loops (Figs. 4c and 5c).

Fig. 7 shows the development of loading–unloading hysteresis loops and defines several relevant parameters. At the peak stress, the applied stress σ_p is the sum of the friction stress σ_f and the back stress σ_b , that is,

$$\sigma_p = \sigma_f + \sigma_b \quad (1)$$

The friction stress σ_f corresponds to the resistance the dislocations have to overcome to keep moving in the lattice, while the back stress σ_b is associated with piled-up dislocations that are created after overcoming the friction stress [30]. The dislocations begin to move under the effect of back stress upon unloading.

$$\sigma_b - \sigma_y = \sigma_f \quad (2)$$

σ_y is the yield stress upon unloading. Consequently, the reverse plastic flow in the unloading of a loaded specimen will begin when the external stress changes by the quantity $\sigma_p - \sigma_y = 2\sigma_f$. If a plastically deformed specimen is unloaded by the quantity $\sigma_p \leq 2\sigma_f$ and again loaded, the unloading and subsequent loading curves will coincide with each other.

Fig. 8 presents the anelastic strain (defined in Fig. 7) as a function of the total strain for pure Mg. Magnesium shows a very marked anelastic behavior. The anelastic strain develops sharply after an applied strain of about 1×10^{-4} , and saturates after the strain of 0.01–0.02, slightly decreases at a larger strain. With increasing the grain sizes, the anelastic strains increase gradually.

As shown in Fig. 9, a simple model is assumed according to Granato–Lücke (G–L) dislocation model [31] to explain the microplastic deformation mechanism of pure Mg. The loading-unloading curves almost coincide with each other or show very small hysteresis loops when the strain is smaller than 1×10^{-4} , the materials almost deform in an elastic manner. The hysteresis loops grow up gradually and the anelastic strain obviously increases above this critical strain of 1×10^{-4} , which is thought to be associated with the breakaway of dislocations from weak pinning point defects (Fig. 9b-c), such as vacancies and impurity atoms in commercial pure Mg. In this case, the dislocations move between the strong pinning points, and sweep large area, leading to the marked increase of anelastic strain. The catastrophic breakaway from the weak pinning points will not cause the microscopic yield of a material at once. Since the dislocation segments elastically return back to their original position when external load is removed, thus the material behaves elastically. The area swept by dislocations and the hysteresis loop areas are closely related to the damping capacities of material, and the strain of unpinning from weak point defects corresponds to the first critical strain amplitude in the curves of strain dependent damping capacity of pure Mg [29]. The relationship between microplasticity and damping will be discussed elsewhere.

The relationship between the cyclic stress and the plastic deformation of pure Mg is

shown in Fig. 10. It can be seen that in the whole plastic deformation region, cyclic stress-strain curves can be expressed in the form

$$\sigma = B \varepsilon_p^n \quad (3)$$

where σ is the stress, ε_p is the plastic strain and B and n are constants. The values of n were obtained by plotting $\log \sigma$ against $\log \varepsilon_p$. Over the range of plastic strain, there are three values of n for pure Mg under different conditions, as shown in Table 1. Thus the plastic deformation process of pure Mg can be divided into three regions: The amount of hardening exponents are between 0.33 and 0.39 in microplastic region I corresponding to the plastic strain below 2×10^{-4} (the total strain of about 8×10^{-4}). When the slopes of straight lines increase to about 0.6, the deformation process enters into microplastic region II; above plastic strain of 1×10^{-3} (the total strain of about $2.5\text{--}2.8 \times 10^{-3}$), the hardening exponents decrease sharply. The first two plastic regions are corresponding to microplastic deformation. And the hardening exponents of pure Mg in present study slightly deviate from the value of 0.5 calculated from the microplastic strain theory proposed by Thomas [9] and Brown [10] for polycrystalline pure metals.

A unique event associated with such a phenomenon as freeing the dislocation from stronger point defects (Fig. 9e) or activating a Frank-Read source (Fig. 9f) occurs when the resolved shear stress exceeds Gb/L_N , where G is the shear modulus, b is the Burgers vector of the dislocation, and L_N is the average length between two near strong pinning points on dislocation segments. Pronounced plastic deformation occurs in materials accompanied by the change of the characteristics of dislocation structure, resulting in the softening of materials. At the beginning of microplastic deformation each dislocation source is considered to be independent of the others, and the piling up of dislocations does not occur, which is in accordance with the lack of strain hardening in the microplastic deformation region I as shown in Fig. 10(a).

According to measured data in single-crystal Mg, the critical resolved shear stress (CRSS) of the basal $\langle a \rangle$ slip system is approximately 1/100 those of non-basal slip systems on prismatic and pyramidal planes [32, 33]. Many studies [16, 23, 25] have shown that the microplastic deformation in Mg alloys is always initiated by basal slip, which is mainly responsible for the plastic strain smaller than the macroscopic yield point. Therefore, dislocation motion occurs only on basal plane in individual grain after the activation of dislocations from strong point defects in microstrain region. Reactions of dislocations with each other inevitably take place. And the dislocation source ceases to be active when the back-stress of the piled-up dislocations reduces the stress at the source to a critical value [9]. So the annihilation and tangle of dislocations lead to the marked decrease of mobile edge dislocations, which is in agreement with the dislocation exhaustion theory proposed by Marschall [4] who stated that the first stage of microplasticity was the activation of dislocation sources and then the exhaustion of mobile dislocations. Therefore, the microplastic deformation enters into region II at the critical plastic strain of about 2×10^{-4} (the total strain of about 8×10^{-4}), exhibiting high strain hardening, as shown in Fig. 10(b).

After the plastic strain of about 1×10^{-3} , i.e., the total strain of about $2.5\text{--}2.8 \times 10^{-3}$, the hardening exponents of pure Mg significantly decrease, as shown in Fig. 10(b), indicating that the other slip systems, such as non-basal slip systems or twinning, may be activated to accommodate the larger plastic strain [16, 23, 34]. Since the limited slip on the basal plane

provides only two independent slip systems, not meeting the von Mises criterion of five independent slip systems for homogeneous deformation. The transition from the microplastic deformation to the macroplastic deformation occurs at this critical strain.

Fig. 11 shows the normalized secant elastic modulus (or apparent elastic modulus) evolvment (E_s , defined in Fig. 7) of pure Mg as a function of total strain, for all the materials tested. The experimental values have been normalized to the initial (elastic) slope of the stress–strain curve of each specimen such that the normalized $E_s = 1$ corresponds to the nominal value of pure Mg, $E = 42$ GPa. For the ECAP processed specimen with grain size of $6 \mu\text{m}$, the secant elastic modulus keeps almost the same as the elastic modulus E below the strain of 1×10^{-4} , but the secant elastic modulus for as-annealed samples decrease quickly. In excess of the strain 1×10^{-4} , the secant elastic modulus significantly decreases, which results from the breakaway of dislocations from the weak pinning points. The E_s reaches a minimum at the strain of 0.01, and slightly increases afterwards. With increasing the grain size, the secant elastic modulus decreases gradually. The smallest value of the secant elastic modulus is only 35% of elastic modulus.

The value of the peak stress can be obtained on the application of a total deformation strain from [35]

$$\sigma_p = \varepsilon(E - \Delta E) = \varepsilon E(1 - \frac{\Delta E}{E}) \quad (4)$$

ΔE is defined in Fig. 7. The inelastic deformation strain has the form

$$\varepsilon_p + \varepsilon_a = \varepsilon - \varepsilon_e = \varepsilon - \sigma_p / E \quad (5)$$

Then the elastic modulus defect can be derived from

$$\frac{\Delta E}{E} = \frac{\varepsilon_p + \varepsilon_a}{\varepsilon} \quad (6)$$

Fig. 12 shows that the elastic modulus defect as a function of total deformation strain. In the coordinates $\log \Delta E / E - \log \varepsilon$, the experimental points fit straight lines so that the experimentally obtained points can be expressed by the equation.

$$\frac{\Delta E}{E} = A\varepsilon^m \quad (7)$$

where A and m are material and experiment sensitive constants. Thus the elastic modulus defect development can be divided into three regions. The elastic modulus defect occurs at a very low strain (10^{-5} – 10^{-4}). The $\Delta E / E$ values increase markedly with increasing strain up to about 8×10^{-4} , and this region is corresponding the microplastic deformation region I in Fig. 10(a); above the critical strain of 8×10^{-4} , the $\Delta E / E$ values increase slightly with lower slopes due to the tangle of dislocations, which is related to the microplastic deformation region II in Fig. 10(b); with entry into region III the deficiency in modulus keep almost constant and the values approach 1.

The development of friction stresses σ_f of all materials are presented in Fig. 13. The friction stress virtually shows a very small value of several MPa at the beginning of deformation. Owing to the release of dislocations from point defects, the friction stresses slightly decrease with increasing strain up to about 1×10^{-3} , then increasing rapidly afterward as a result of the formation of dislocation forests. With the increase of the grain sizes, the

friction stresses decrease gradually.

3.4 The effect of grain size on microplasticity

The present observation suggests that plastic yielding can occur in ECAPed pure Mg at a very small stress of several MPa. With increasing the annealed temperatures, the grain sizes of pure Mg markedly increase, however, the textures almost keep constant. As shown in Fig. 2, the dislocation densities are very low after annealing treatments, and considered to be in the same order [36]. Thus there is no doubt that grain size is the major factor in determining the plastic strain at a given stress for as-annealed specimens. As grain size is increased, the loading-unloading hysteresis loops become more pronounced, and the microplastic strain also increases. It was reported by Thomas [9] that the microplastic strain at a given stress was proportional to the third power of the grain size, i.e. $\varepsilon = Kd^3$, for high-purity polycrystalline copper. This means that larger grain size will contribute to relatively larger microstrains. Moreover, the specimen with larger grain size shows a larger elastic modulus defect (Fig. 12) but smaller secant elastic modulus (Fig. 11) and friction stress (Fig. 13). In fact, the smaller the grain size is, the more grain boundaries the specimen has, also the more strong dislocation pinning points it has. Thus dislocation movement is intensively inhibited in Mg with smaller grain size, in which most dislocations are confined within the cell walls or grain boundaries. For the as-ECAPed pure Mg, the smaller grains and tangle of dislocations both increase the difficulty of dislocation motion for a long distance, so it has the smallest microplastic strain at a given stress. Koike [12] found that large-grained AZ31 Mg alloy facilitated basal slip more easily than small-grained alloy, thus exhibiting a clear microscopic yielding at a very low stress, and the strain hardening rate of alloy with larger grain size decreased with increasing strain from the very beginning of deformation. Furthermore, larger grain size specimen gives more pronounced anelasticity (Fig. 8) during cyclic loading, which is contrary to the observations of Caceres [19] and Mann [20], who pointed out that smaller grain size specimen would give more instable twins, and showed marked anelastic behavior. In the present research such a small stress is not enough to activate twins. In addition, the texture with basal planes inclined about 40° to the ED facilitates the basal slip during tensile test, increasing the difficulty to activate twinning.

4. Conclusions

The cyclic plastic deformation behavior of the ECAPed pure Mg with different grain sizes in microstrain region was studied by tensile loading and unloading experiments, and the following conclusions are drawn:

(1) Heat treatments were carried out on ECAP processed pure Mg to modify the microstructures. The grain size is significantly increased and the dislocation density is decreased after a short time annealing, while the texture almost keeps stable.

(2) Pronounced hysteresis loops and anelastic strain progressively develop after a small strain of about 1×10^{-4} , which corresponds to the breakaway of dislocations from weak pinning points. The microplastic deformation process can be divided into two regions: In the first region pronounced plastic deformation associated with dislocation motion on basal plane takes place at several MPa. The materials are softened and characterized by low friction stresses and hardening exponents. The microplastic deformation enters into region II above the strain of about 8×10^{-4} . Annihilation and tangle of dislocations lead to the increase of

hardening exponents and friction stresses.

(3) Grain size has significant effect on microplastic deformation behavior of pure Mg. Larger grain size will contribute to relatively a larger microstrain and elastic modulus defect, but a smaller friction stress and secant elastic modulus.

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Figure Captions:

Fig. 1. Optical microstructures of pure Mg after (a) ECAP processing and subsequent annealing at (b) 250°C for 60min, (c) 300°C for 30min, (d) 350°C for 15min, (e) 400°C for 10min. The grain sizes are 6, 15, 27, 58, 72 μm , respectively.

Fig. 2. TEM microstructures of pure Mg after (a) ECAP processing and subsequent annealing at (b) 250°C for 60min, (c) 300°C for 30min.

Fig. 3. (0002) and $(10\bar{1}0)$ complete pole figures of pure Mg after (a) ECAP processing and subsequent annealing at (b) 300°C for 30min, (c) 400°C for 10min.

Fig. 4. Tensile curves of the ECAPed and annealed pure Mg of different grain sizes in microstrain region.

Fig. 5. Tensile loading–unloading loops measured by DMA for the ECAPed and annealed pure Mg of different grain sizes.

Fig. 6. Tensile loading–unloading loops measured by tensile machine for the ECAPed and annealed pure Mg of different grain sizes.

Fig. 7. Schematic flow curve of cyclic loading hysteresis loops. E is the elastic modulus of Mg (42MPa), E_s is the secant modulus. ε_p and ε_e are the plastic strain and elastic strain;

ε_a is the anelastic strain during unloading. σ_p is the peak stress at the start of the unloading;

σ_f and σ_b are the friction stress and back stress, respectively; σ_y is the yield stress upon unloading.

Fig. 8. The anelastic strain as a function of the total strain in (a) smaller strain and (b) larger strain region.

Fig. 9. Dislocation model of microplasticity according to Granato-Lücke theory [31].

Fig. 10. The stress as a function of the plastic strain in (a) smaller strain and (b) larger strain region.

Fig. 11. The normalized secant elastic modulus as a function of true strain for pure Mg.

Fig. 12. The modulus defect plotted against strain for pure Mg of different grain sizes during cyclic tensile tests.

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Fig. 13. The development of friction stress as a function total strain for pure Mg of different grain sizes.

Table 1. The average grain sizes and hardening exponents n for pure Mg after ECAP processing and subsequent annealing.

Materials	As-ECAPed	ECAP+ 250 °C-60min	ECAP+ 300 °C-30min	ECAP+ 350 °C-15min	ECAP+ 400 °C-10min
Grain size (μm)	6	15	27	58	72
n_1	0.39	0.38	0.36	0.35	0.33
n_2	0.58	0.60	0.62	0.62	0.61
n_3	0.19	0.21	0.22	0.22	0.21