

Microstructure and Mechanical Properties of Pure Magnesium Subjected to Hot Extrusion

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Abstract

The as-cast pure magnesium (Mg), with a purity of 99.99%, was hot-extruded at 300 °C to prepare a Mg bar with a diameter of 8 mm. The microstructure and mechanical properties of the sample before and after extrusion were obviously refined with a large number of subgrains rather than equiaxed grains. The results show that the as-extruded microstructure has $\{10\bar{1}2\}$ tensile twins can be observed significantly in the microstructure at this temperature. Mechanical properties including yield strength (YS), ultimate tensile strength (UTS) increased greatly but uniform elongation (UE) decreased slightly as a result of work hardening.

Keywords: Microstructure, Pure mg, Mechanical Properties, Hot Extrusion

Introduction

Mg and its alloys, with high strength and low density, are the lightest structural material at present, which have been applied widely in the fields of automotive, aerospace and biomedicine [1-3]. However, they possess poor formability and less slip systems at room temperature as to the close-packed hexagonal (HCP) crystal structure, highly limiting their cold deformation.

Severe plastic deformation (SPD), which provides a very large strain and significant rearrangement of dislocations, is considered commonly to be an effective way to refine grains and improve mechanical properties for Mg and its alloys [4,5,6]. There are many SPD methods including equal channel angular pressing (ECAP), multiaxial forging (MAF) and high pressure torsion (HPT) etc., which have been used to produce ultrafine and even nanostructured grains for materials. Biswas et al. [7] carried out ECAP on Mg at higher temperatures for seven passes and nearly room temperature for final 8th pass, indicating slip is the main deformation mode. Huang et al. [8] processed AZ31 Mg alloy using HPT, the results showed that significant grain refinement was achieved and grain size is in the sub-micro range at temperatures of 296 K and 373 K. Guo et al. [9] also indicated that grains in as-cast AZ80 Mg alloy were refined during the deformation induced by hot multiaxial forging (MAF).

Moreover, S. Biswas et al. [10] investigated the microstructure and texture during extrusion of pure magnesium at 400 °C, indicating dynamic recrystallization (DRX) contributed to the grain refinement

and texture rotation. S. Suwas et al. [11] prepared extruded pure Mg at different temperatures, the results showed that mechanical properties including YS and UE increased with more refined grains at a lower temperature. S. Suwas et al. [12] analyzed texture evolution of Mg processed by equal channel angular extrusion (ECAE), arguing this texture may be advantageous to cold rolling. Generally, to speak generally, lots of work has been done with respect to the plastic deformation of Mg and its alloys in recent years. However, relative researches about warm plastic deformation for them are still not so enough that more studies need to be done. In this paper, a commercially pure Mg ingot was hot-extruded and then the microstructure, texture and mechanical properties of as-extruded pure Mg were studied in detail.

Experimental Procedures

A cylindrical pure Mg (99.99% purity) billet with a diameter of 32 mm, height of 50 mm was used as the original material for extrusion process. Graphite and medical vaseline were mixed as a lubricant. First it was heated to 300 °C and kept for 20 min in a die using a thermocouple to control the temperature, and then extruded into a Mg bar with a diameter of 8 mm at a speed of 5 mm/s.

A sample (5 mm × 4 mm × 3 mm) was cut along the extruded direction for electron back-scattered diffraction (EBSD) measurement. Samples were mechanically ground by sandpapers, followed by electro-polishing in a solution containing perchloric acid (10 ml) and ethanol (190 ml) at 243K. EBSD measurements were performed by using a MR1A3 scanning electron microscope (SEM) with an EBSD camera fitted on it. Samples were tilted by 70° before EBSD and EBSD data were processed with Channel 5 analysis software. Tensile tests were conducted at room temperature using a CMT5205

tensile machine at a speed of 0.5 mm/min. The gage length of the samples for tensile test is 15 mm, with their cross-sectional area of 4 mm in width and 2 mm in thickness.

Results and Discussion

Fig. 1 shows the microstructure before and after hot extrusion. The as-cast pure Mg shows very large grains (appropriately 1500 μm). But after hot extrusion, these coarse grains were refined obviously, with an average grain size of $\sim 40 \mu\text{m}$. When a pure Mg rod was hot-extruded at a high strain rate (with a extrusion ratio of ~ 16) and a short deforming time, coarse grains were smashed into a large number of subgrains, generating many low-angle grain boundaries. Consequently, the as-extruded microstructure becomes finer than the initial one.

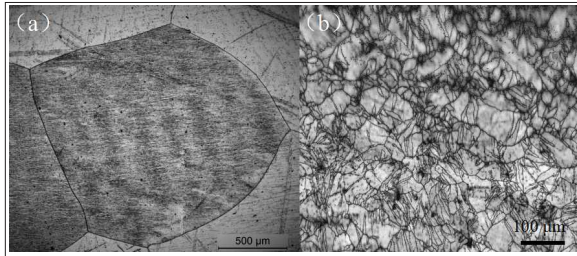


Figure 1: Microstructures of pure Mg (a) before and (b) after hot extrusion characterized by EBSD contrast

Fig. 2a shows the components of the as-extruded microstructure, where blue color represents recrystallized grains while yellow and red indicate subgrains and deformed grains, respectively. It can be found easily (Fig. 2b) that the majority of grains in the as-extruded microstructure are subgrains with a fraction of $\sim 60.5\%$, which is higher than that of recrystallized ($\sim 2\%$) and deformed ($\sim 37.5\%$) grains. Since pure Mg possesses a HCP structure, the stacking fault energy especially in prismatic and pyramidal planes is higher than other structural metals. As a result, recovery instead of recrystallization can proceed sufficiently, though the processing temperature is 300 $^{\circ}\text{C}$. Therefore, stable subgrains are formed in the microstructure, which is different from the result of Fan et al. [13].

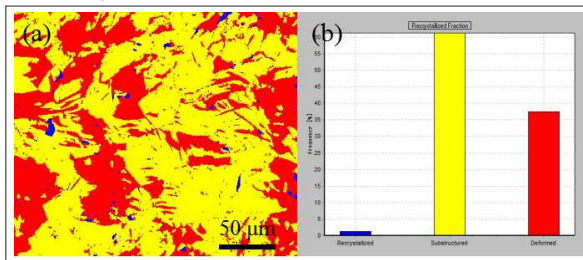


Figure 2: The components (a) of as-extruded microstructure and (b) their percentages

Moreover, it is worth to note that there are a great number of twins (Fig. 3a) of type (10 2) in the microstructure, indicating twinning is also an important deformation mode beside slipping at this temperature. The twin fraction is $\sim 20.2\%$, which was calculated by EBSD analysis software. The morphology of these twins is also obtained by using transmission electron microscope (TEM), as displayed in Fig. 3(b). Sun et al. [14] extruded a pure Mg ingot at 300 $^{\circ}\text{C}$ and also found that some twins appeared after extrusion. Fan et al. [13] reported that a pure Mg bar was hot-extruded at 350 $^{\circ}\text{C}$ with a extrusion ratio of ~ 12 , which is close to the value of ~ 16 in this paper. However, few twins were observed in the as-

extruded microstructure, which is contrary to the result that reported in our work. Therefore, the formation of twins in the as-extruded microstructure is mainly associated with the processing temperature, and 300 $^{\circ}\text{C}$ may be the critical point above which twinning can not be activated during the extrusion, further studies still need to be done.

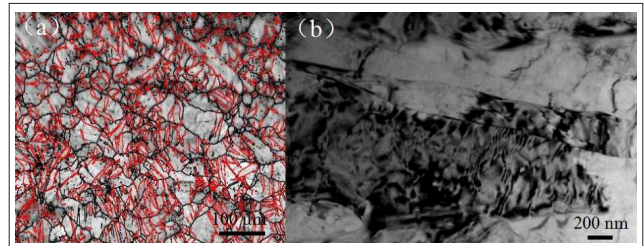


Figure 3: (10 2) twins characterized by (a) EBSD contrast (in red lines) and (b) TEM micrographs of pure Mg after hot extrusion

Fig. 4 displays the tensile curves of as-cast and as-extruded pure Mg. The data of mechanical properties, including YS, UTS and UE are shown in Tab.1.

Table 1: Tensile mechanical properties of the as-cast and as-extruded pure Mg

Samples	YS / MPa	UTS / MPa	UE / %
as cast	18	48	16
as extruded	106	178	10

The as-cast pure Mg yields easily with a very low YS of $\sim 18 \text{ MPa}$ and UTS of $\sim 48 \text{ MPa}$ due to the easy activation of basal slip in coarse grains [15]. After hot-extrusion, the mechanical properties are highly improved as a result of work hardening, with YS of $\sim 106 \text{ MPa}$ and UTS of $\sim 178 \text{ MPa}$. The classic Hall-Petch equation indicates that the YS of a polycrystalline material will be enhanced as a result of the reduction of grain size. As shown in Fig. 1b, the grain size decreases obviously after hot extrusion, so the enhancement of YS is in good accordance with the Hall-Petch equation. Beside it, a large number of twins, which also play an important role in modifying the YS, appear largely in the as-extruded microstructure, as shown in Fig. 3a. In other words, they may act as a barrier to impede dislocation slip and conduce to a better property for pure Mg [16]. Consequently, both the grain refinement and twinning cause the increase of YS. However, UE decreased after hot extrusion because of work hardening. During the process of hot extrusion, large deformation was imposed on the cast Mg, generating many subgrain boundaries and twin boundaries, which further lead to local strain hardening and final reduction of UE. Sun et al. [14] also reported that the UE of the as-extruded pure Mg was dropped to 7% after second pass from initial 13% of first-pass. Similar phenomena can also be found in other plastic deformation of metallic materials [17-19].

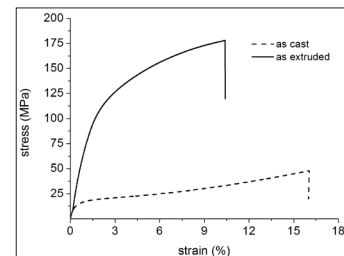


Figure 4: Tensile strain-stress curves of as-cast and as-extruded pure Mg at room temperature

Fig. 5 shows fracture morphology of as-cast and as-extruded pure Mg obtained by SEM. Figs. 5(a) and 5(b) display fracture surface of as-cast pure Mg, river-like pattern can be found easily (Fig. 5a) with some cleavage steps. More details with long band-like tearing edges are shown in Fig. 5(b), which was magnified by 2000.

However, the fracture morphology after hot extrusion is distinctly different from the initial one. River-like pattern is replaced by interrupted short steps (Fig. 5c) with some small and shallow dimples (Fig. 5d) due to the hot deformation.

Generally speaking, micropores appear and then gathered by the plastic deformation, so dimples develop in the fracture surface. Moreover, the dimple size also have an effect on the plastic deformation capability of a material, the larger micropore size is, the better deformation ability is. As a consequence, the dimples with smaller size in the as-extruded fracture of pure Mg result in the poor ductility and thereby UE decreased.

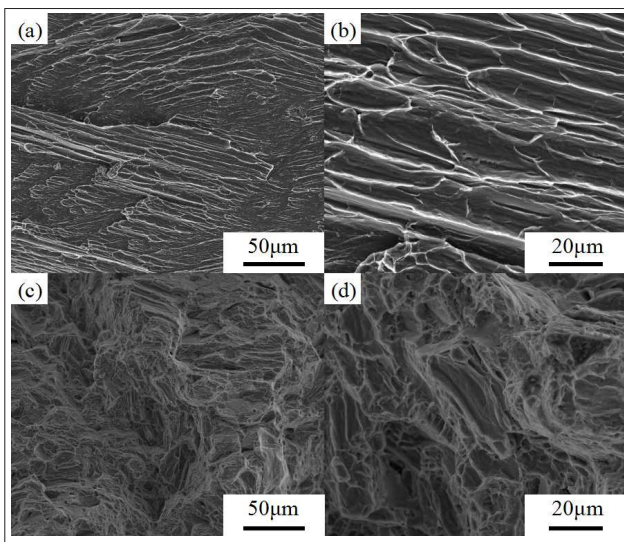


Figure 5: SEM fracture morphology of (a), (b) as-cast and (c), (d) as-extruded pure Mg magnified 500 (a,c) and 2000 (b,d) respectively

Conclusion

In this research, a pure Mg bar was prepared by hot extrusion at 300 °C and its microstructure, texture and mechanical properties were investigated explicitly and conclusions can be drawn as follows

1. The as-extruded microstructure is obviously refined with a large number of subgrains rather than equiaxed grains. Twins of type $(10\bar{1}2)$ appear largely in the microstructure as a result of high extrusion ratio in a short time.
2. After hot extrusion, YS and UTS are greatly improved as a result of refined grains and numerous twins in the microstructure but UE decreased slightly.
3. The fracture morphology of the as-extruded pure Mg sample shows obvious difference compared with that of the as-cast one due to the hot extrusion.

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