Microstructures and mechanical properties of pure magnesium bars by high ratio extrusion and its subsequent annealing treatment

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Abstract: Pure magnesium bars were prepared by two-pass cumulative high ratio extrusion with as-cast Mg as the original material and then the final as-extruded bars were annealed. The effect of extrusion deformation and annealing treatment on the microstructure, mechanical properties and fracture behaviour of the Mg were investigated by optical microscopy (OM), mechanical properties test and scanning electron microscopy (SEM), respectively. The results show that the grain size is obviously refined by the effect of dynamic recrystallization during the extrusion deformation. Thus the room-temperature mechanical properties and fracture behaviour of the material were significantly improved. After the first extrusion, the coarse as-cast grain size was reduced to 35 μm, and the yield strength (YS), ultimate tensile strength (UTS) and elongation of the bar achieved 84 MPa, 189 MPa, and 12%, respectively. After the further extrusion, the YS of as-extruded bar was over 120 MPa; however, the elongation decreased due to work hardening. Finally the grain size of the as-extruded bar was 9−10 μm after annealing treatment, and its YS, UTS and elongation of the bar achieved 124 MPa, 199 MPa, and 10.7%, respectively. The microstructures and mechanical performance of the material were enhanced obviously.

Key words: pure magnesium; high ratio extrusion; mechanical properties; grain refinement; dynamic recrystallization

1 Introduction

Magnesium alloy with advantages of high specific strength and specific stiffness is the lightest structural material which has broad application prospects in the field of automotive and aerospace industry [1−4]. In addition, with advantages of excellent biocompatibility and biodegradability, magnesium and magnesium alloy especially the high-purity pure magnesium almost do not contain any harmful impurity elements, and have great potential in the biomedical fields [5−7]. At present, as a new generation of biomedical materials, magnesium alloy suffered researchers to favor fully.

However, as we all know, magnesium alloys process close-packed hexagonal crystal structure and less slip systems at room temperature, which limit their cold deformation [8]. Studies have shown that grain size of magnesium alloy can be refined greatly by severe plastic deformation (SPD) and their improved plasticity [9,10].

At present, many SPD methods have been adopted by researchers to obtain fine-grained, ultrafine-grained or even nanostructured bulk materials, such as equal channel angular extrusion (ECAE), high pressure torsion (HPT), multiple forging (MF) [11]. SOMJEET et al [12] have studied equal channel angular extrusion process of pure magnesium and found the main deformation mechanism of ultrafine crystalline pure magnesium is slip. The microstructure and thermal stability of ultra-fine grained pure Mg and Mg10Gd alloy treated by HPT were investigated by CIZEK et al [13], and the microstructures of HPT-treated Mg and Mg10Gd and its grain growth with temperature were characterized. The fracture behaviors of AZ80 magnesium alloy during multiple forging processes have been investigated by GUO et al [14]. It was found that the mechanisms of cracking on the surface and inside the specimens are different. SUN et al [15] have studied microstructure stability of AZ31 magnesium alloy wire via cold drawing during annealing process and they reported that a large

amount of shear deformation contributes to grain growth.

On the plastic deformation of magnesium alloy, many researchers have done lots of work [16−18], but the research about the plastic deformation behavior of magnesium alloys, especially cold and warm plastic deformation behaviors, are not enough. More deeper and detailed researches needed to be done. In this work, the microstructures, mechanical properties and fracture behaviors of pure magnesium by multi-passes accumulated high ratio extrusion and annealing process were studied with as-cast pure magnesium as original material.

2 Experimental

The commercial as-cast pure magnesium (99.95% purity) billets with a diameter of 52 mm, height of 40 mm were used as the starting material of extrusion process. After two-pass extrusion, the secondary extruded pure magnesium bars with a diameter of 5 mm were obtained. The major extrusion process parameters are shown in Table 1. To investigate the effect of annealing process on microstructures and mechanical performance of the extruded rods, the secondary extruded bars were annealed at 250 °C for 20 min in a resistance furnace without any protect gas.

Table 1 Process parameters for extrusion of pure magnesium

Extrusion pass	Extrusion temperature/°C	Extrusion ratio	Diameter after extrusion/mm
1	300	27	10
2	150	4	5

The microstructures of pure magnesium with different treatment processes were observed on an optical microscope and their mechanical properties were performed at room temperature by an INSTRON−5569 standard testing machine. The geometry and size of tensile specimen is shown in Fig. 1. For all experiments, strain rate was 1×10^{-3} s^{-1}. To investigate fracture behaviors of pure magnesium under different processes, the tensile fracture surfaces were observed on a scanning electron microscope.
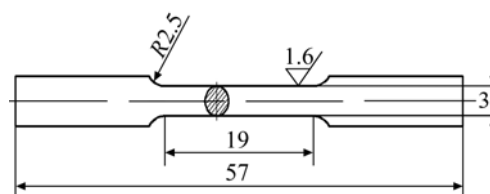

Fig. 1 Geometry and dimension of tension specimen (unit: mm)

3 Results and discussion

3.1 Microstructures

Figure 2 shows the microstructures of the material by different process states. It can be seen from Fig. 2 (a) that the initial as-cast billet is characterized by non-uniform and coarse grains, with the grain size in the range from several microns to several millimeters.

In contrast, as shown in Fig. 2(b), the microstructure of as-extruded bar after the first-pass extrusion is greatly reduced and presents relatively homogeneous grain size with the average grain size of about 35 μm. The homogeneous microstructure and grain size refining are
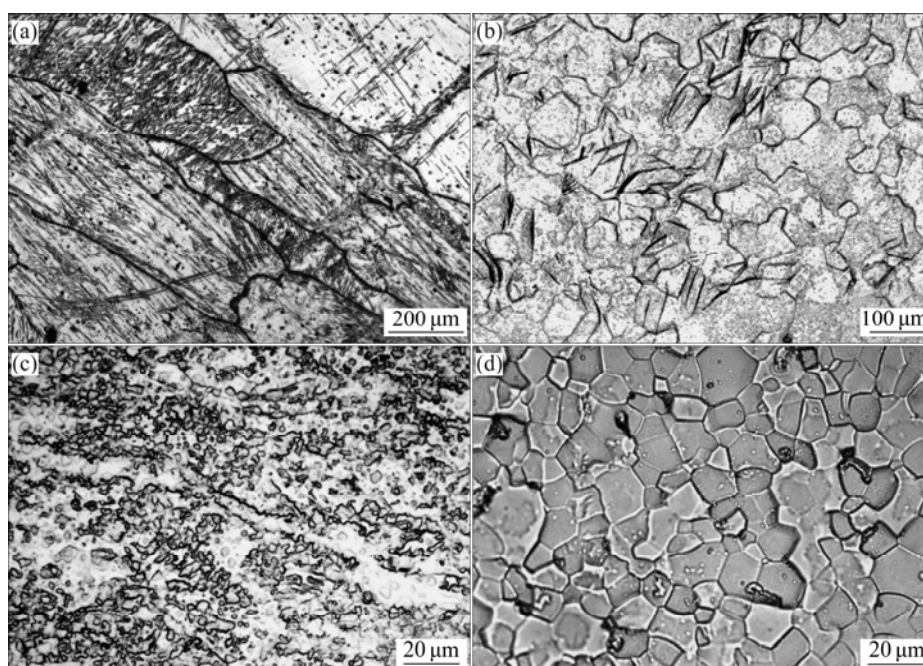

Fig. 2 Microstructures of pure magnesium by different treat processes: (a) As-cast billet; (b) 1-pass extruded bar; (c) 2-pass extruded bar; (d) As-annealed bar

due to dynamic recrystallization during the first-pass extrusion with the relatively high extrusion temperature of 300 °C. In addition, a large number of deformation twining was formed during the deformation process because of the relatively large extrusion ratio of 27, and the severe deformation occurred [19].

The microstructure of as-extruded bar after the second-pass extrusion is shown in Fig. 2(c). Lots of deformation state microstructures can be observed due to the occurrence of partial dynamic recrystallization. In Ref. [20], when the extrusion ratio is less than 5, the inhomogeneous microstructures will occur in the as-extruded product, and the smaller the deformation degree is, the more inhomogeneous the microstructure is. In this experiment, the phenomenon of inhomogeneous microstructure has occurred because the second extrusion ratio was only 4 and the deformation degree was very small. In addition, the extrusion temperature was lower and the energy from outside environment was not sufficient to promote the occurrence of fully dynamic recrystallization. Therefore, the deformation state microstructure was obtained in the bar after the second-pass extrusion.

To improve the microstructures and mechanical performance of the second-pass extruded bar, they were annealed at 250 °C for 20 min, and the impact of static

recrystallization on the microstructures and mechanical performance of them was studied. The as-annealed microstructure is shown in Fig. 2(d) and the average grain size was 9−10 μm because of the occurrence of static recrystallization.

3.2 Mechanical properties

Figure 3 shows the room-temperature mechanical properties of materials in different process states. As shown in Fig. 3(a), the yield strength (YS), ultimate tensile strength (UTS) and the maximum elongation of as-cast billet were only 24 MPa, 86 MPa and 4.8%, respectively.

Obviously, the YS, UTS and the corresponding elongation of the first-extruded bar were 84 MPa, 189 MPa and 12%, respectively, as shown in Fig. 3(b). The basic mechanical properties of the material have been significantly enhanced after hot extrusion.

However, when the first-pass extruded bar was further extruded at 150 °C, only the YS rose to 123 MPa, while the corresponding UTS and elongation decreased to162 MPa, and 7%, as shown in Fig. 3(c). The combined effect of the fine grain strengthening and deformation strengthening makes the YS of the second extruded bar significantly higher than that of the first-pass extrued bar because of the refinement of grain
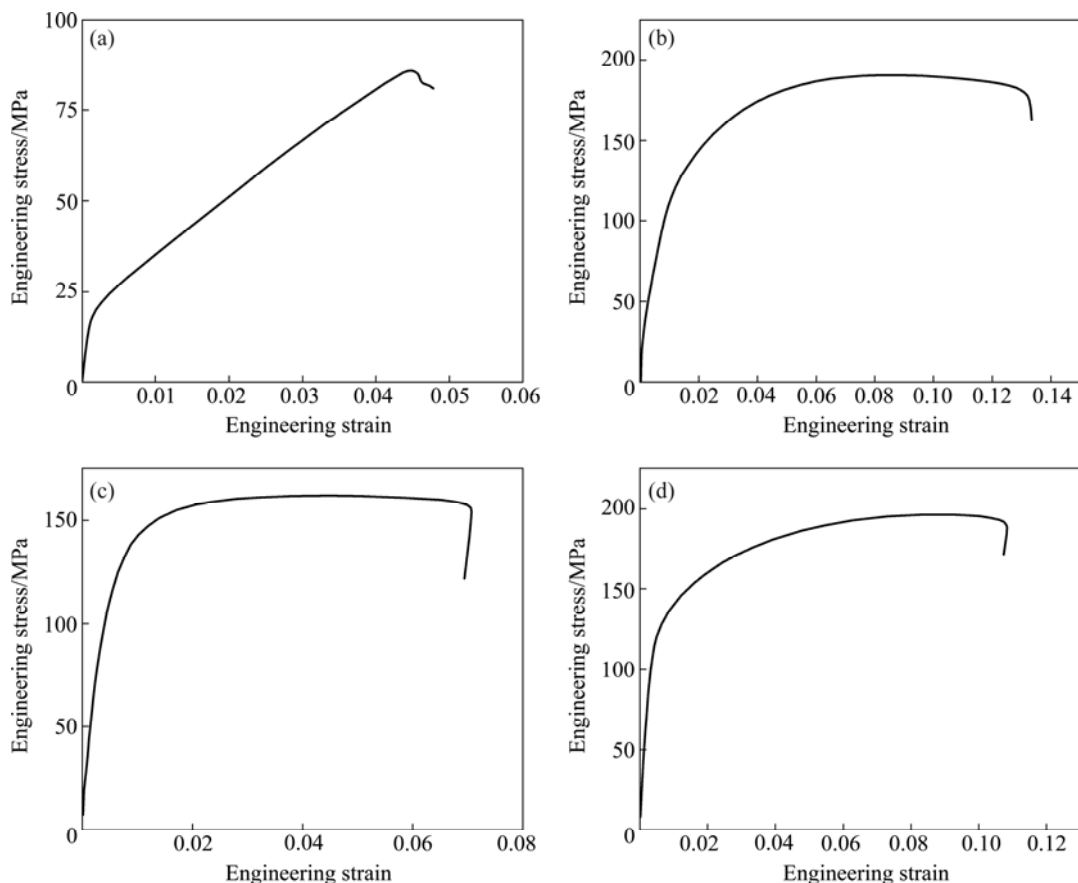

Fig. 3 Stress—strain curves of pure magnesium by different treat processes at room temperature: (a) As-cast billet; (b) 1-pass extruded bar; (c) 2-pass extruded bar; (d) As-annealed bar

size of the further extruded bar.

From Fig. 3(d), it can be seen that the YS of the second extruded bar did not decrease after annealing, about 124 MPa, while the UTS and maximum elongation were significantly improved to 199 MPa and 10.7%, respectively. The mechanical properties of the material were obviously enhanced after static recrystallization.

3.3 Fracture morphology

Figure 4 shows the room-temperature tensile fracture surfaces of pure magnesium rods in different process states. As we all know, to some extent, the size and depth of the dimple of the material depend on plastic deformation capability, and the better the plastic deformation ability of the metal, the more prone the necking is and the greater the size of the micropores is.

From Fig. 4(a), it can be seen that the tensile fracture surface of as-cast billet is featured by typical river-like pattern of the brittle fracture, which indicates its poor room-temperature ductility.

In comparison, after the first-pass extrusion, a large number of dimples were formed on fracture surface, which indicates that the plasticity of as-cast pure magnesium can be greatly improved after the first-pass extrusion, as shown in Fig. 4(b). The fracture pattern of the pure magnesium bars from brittle fracture transformed into the porous coalescence fracture due to the hot extrusion.

However, as shown in Fig. 4(c), after further extrusion, the mixed morphology of shallow dimples and tearing ridges was clearly observed on fracture surface and the depth of dimples was shallower than that of the first-pass extruded bar.

In contrast, as shown in Fig. 4(d), a large number of deep dimples were left on room-temperature tensile fracture surface of the second-pass extruded bar again after annealing, which demonstrates that the plasticity of the material by plastic deformation at low-temperature can be improved by static recrystallization annealing.

According to the above analysis, it can be found that after the two-pass extrusion and subsequent recrystallization annealing process, the fracture pattern of polycrystalline pure magnesium rod was changed because of the grain size refinement and the improved plasticity.

4 Conclusions

1) The grain size of as-cast pure magnesium can be significantly refined by dynamic recrystallization due to hot extrusion deformation, therefore, the room-temperature mechanical properties of the material were significantly improved and their room-temperature tensile fracture behaviors were changed.

2) It is prone to the phenomenon of inhomogeneous deformation when the extrusion ratio was selected to be relatively small (in this experiment the extrusion ratio $\lambda=$ 4). When the extrusion temperature was selected to be relatively low (in this experiment the extrusion temperature is 150 °C), work hardening caused the increased YS of pure magnesium, while the decreased plasticity.

3) The refinement of grain size and the improvement of mechanical performance of pure magnesium after warm plastic deformation can be obtained by the reasonable static recrystallization
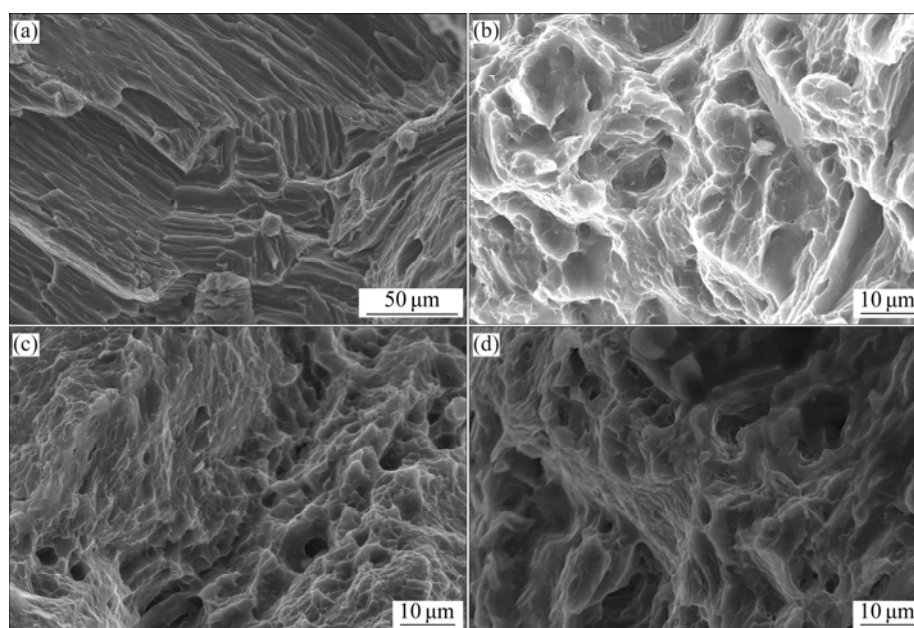

Fig. 4 Tensile fracture morphology of pure magnesium by different treat process: (a) As-cast billet; (b) 1-pass extruded bar; (c) 2-pass extruded bar; (d) As-annealed bar

annealing process. In the present study, the average grain size of the second-pass extruded bar was smaller than 10 μm after the annealing treatment, and their YS, UTS and elongation were 124 MPa, 199 MPa and 10.7%, respectively.

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纯镁大比率挤压棒材的组织与性能及其随后的退火处理

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摘　要：以纯镁铸锭作为坯料，经过两道次累计大比率挤压制备了棒材，并对最终的棒材进行退火处理。通过光学显微镜(OM)、力学性能测试和扫描电子显微镜(SEM)研究了挤压变形和退火处理对纯镁组织、性能以及断裂行为的影响。结果表明：在挤压变形过程中，由于动态再结晶的作用，材料的晶粒尺寸得到明显细化，从而显著地改善了材料的室温力学性能和断裂方式。经过一次挤压后，粗大的铸态晶粒细化到 35 μm，屈服、抗拉强度和伸长率分别达到 84 MPa、189 MPa 和 12%，所得棒材经再次挤压后，屈服强度超过 120 MPa，但是，由于加工硬化的作用，伸长率有所下降。对最终棒材进行退火处理后，平均晶粒尺寸为 9~10 μm，屈服强度、抗拉强度分别达到 124 MPa、199 MPa，伸长率为 10.7%，材料的组织和性能得到明显改善。

关键词：纯镁；大比率挤压；力学性能；晶粒细化；动态再结晶

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