IOP Conf. Series: Materials Science and Engineering **182** (2017) 012051 doi:10.1088/1757-899X/182/1/012051

Microstructure evolution and mechanical properties of biomedical Mg-Zn-Gd alloy wires

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Abstract. In order to manufacture the Mg-Zn-Gd alloy fine wires for the development of new biomedical Mg alloy implant devices, a hot extrusion and cold drawing process which is used to develop the Mg-Zn-Gd alloy fine wires were investigated. The results demonstrate that the Mg-Zn-Gd alloy has good formability. The microstructure and properties of the Mg-Zn-Gd alloy wires were studied by the observations of optical microscopy and scanning electron microscopy. The results show that the process is successfully developed to manufacture the high-quality wires with 3.00 mm diameter. The achievement of the high-quality Mg-Zn-Gd alloy wires is ascribed to the refined microstructure due to dynamic recrystallization during hot extrusion. Additionally, the grain morphology can play an important role in affecting the subsequent cold drawing performance.

1. Introduction

Mg alloys have attracted more and more attentions as promising implant materials due to their good biocompatibility, biodegradability and appropriate mechanical properties, etc.. Mg alloy fine wires are considered to be one of the huge application potential of Mg alloys in medical devices [1-3]. However, since Mg with the hexagonal close packed crystal structures displays poor ductility and cold formability, it is difficult to produce Mg alloy wires. In order to enhance the workability of Mg alloys, rare earth (RE) elements such as Gd and Y are usually added to significantly improve the comprehensive mechanical properties of Mg alloys. Furthermore, these rare earths are considered to be suitable for the preparation of medical Mg alloy wires as major alloying elements due to relatively high bio-safety [4–6]. And Gd is usually used for improving the properties of Mg alloys as one of important rare earth elements. It has been demonstrated that Gd as the addition of biomedical Mg alloys is feasible [7].

In the present study, Gd was added to the Mg-Zn alloy to fabricate the high-quality wires through a combination of hot extrusion and cold drawing. In addition, the microstructures and mechanical properties of the experimental Mg-Zn-Gd alloy wires were examined at different states. It is anticipated that the present experimental results will be helpful for future efficient production of Mg alloy fine wires by cold drawing processing.

2. Experimental procedures

2.1. Fabrication of wires

The chemical compositions of the experimental Mg-Zn-Gd alloy by chemical analysis can be listed in table 1.

Table 1. Chemical compositions of the experimental alloy.

Alloy	Zn	Gd	Mg
Mg-Zn-Gd	1.18	1.02	Bal.

Based on the previous works [8-11], we carefully selected the processing parameters for Mg-Zn-Gd alloy wires as follows: i) The Mg alloy was melted at 750 °C for 20 min, and then the molten Mg alloy was cooled to 720 °C and poured into an iron mold; ii) The obtained billets were machined into a size of 100.0 mm in diameter and 250.0 mm in length, and extruded into wires with 4.0 mm in outer diameter at 400 °C with the extrusion rate of 20:1; iii) The extruded wires were then drawn at ambient temperature with an area reduction between 9.5% and 12.5% every pass. After every two passes of cold drawing, annealing is performed at 400 °C for 60 min to remove the work hardening. In the drawing processes, vegetable oil was used as the lubricant. Eventually, the Mg alloy wires, whose outer diameter was reduced to 3.00 mm, were fabricated. These working procedures can be summarized as follows: melting→hot extrusion(4.0 mm in diameter)→cold drawing(3.8 mm in diameter)→cold drawing(3.6 mm in diameter, as-drawn 1#)→annealing(3.6 mm in diameter)→cold drawing(3.2mm in diameter)→cold drawing(3.0 mm in diameter, as-drawn 2#).

2.2. Microstructure analysis

The wires for microstructure observation were cut from the longitudinal sections, and ground with different grits of silicon carbide papers starting from 200 grit to 1500 grit and polished with 1 μm and 3.5 μm diamond pastes, and then chemically etched by a solution of 1.5 g picric acid, 25 ml ethanol, 5 ml acetic acid and 10 ml distilled water. The grain structures and morphologies of the samples were examined using the optical microscope (LEICA-DMI3000M).

2.3. Mechanical property test

The tensile mechanical property test was carried out on a universal material test machine (DNS200) at a strain rate of $1\times10\ s^{-1}$ at room temperature. For each group of tests, three wires were chosen as a function of Chinese standard (GB/T 228.1-2010) with an over length of 150 mm and a gage length of 100 mm. The fracture surfaces of the wires were examined using the scanning electron microscopy (JSM-5610LV).

3. Results and discussion

3.1. Processing of wires

Figure 1 shows the processing product from the extruded billets of the Mg-Zn-Gd alloy to drawn wires. The extruded wires and drawn wires exhibit no deformation. It can be further observed that the wires have smooth surfaces and high straightness, which are suitable for subsequent processing.

Figure 1. Experimental Mg-Zn-Gd alloy wires: extruded wire and drawn wire.

3.2. Microstructure observation

Figure 2 shows the microstructures from longitudinal sections of the wires for the Mg-Zn-Gd alloy under different conditions. For figure 2a, equiaxed, completely recrystallized grains of the as-extruded alloy are observed with the average grain size of approximately 6.9 μm. It demonstrated that the dynamic recrystallization occurred in the hot extrusion working processing of Mg-Zn-Gd alloy. When cold drawing deformation of 19% for the as-extruded alloy was employed, the microstructure of as-drawn wire was remarkably different from that of the as-extruded alloy, as shown in figure 2b, where the tendency of the fibrous structures along drawing direction was developed and grains became smaller in comparison with original equiaxed grains. Meanwhile, the work-hardening rate of the Mg alloy with the hexagonal close packed crystal structures was quickly increased during cold drawing deformation. Therefore, static recrystallization through annealing treatment is necessary to achieve subsequent cold drawing. Thus, annealing was carried out at 400 °C for 60 min in the present study. The results showed that equiaxed, static recrystallized grains formed during annealing treatment, as displayed in figure 2c. Measurement of grain size revealed that there was a fully recrystallized microstructure of the annealed Mg-Zn-Gd alloy wire with the mean grain size of approximately 12.7 μm, which was about twice as big as that of the as-extruded wires. The annealing treatment leads to the improvement in regularity and uniformity of recrystallization grains while there is a rise in average grain size. It hints that the microstructure of the wires grows enough during the present annealing treatment. Apparently, it is relatively easy for the Mg-Zn-Gd alloy wires with static recrystallized grains to further draw into finer wires with 3.0 mm in diameter, whose microstructure can be seen in figure 2d.

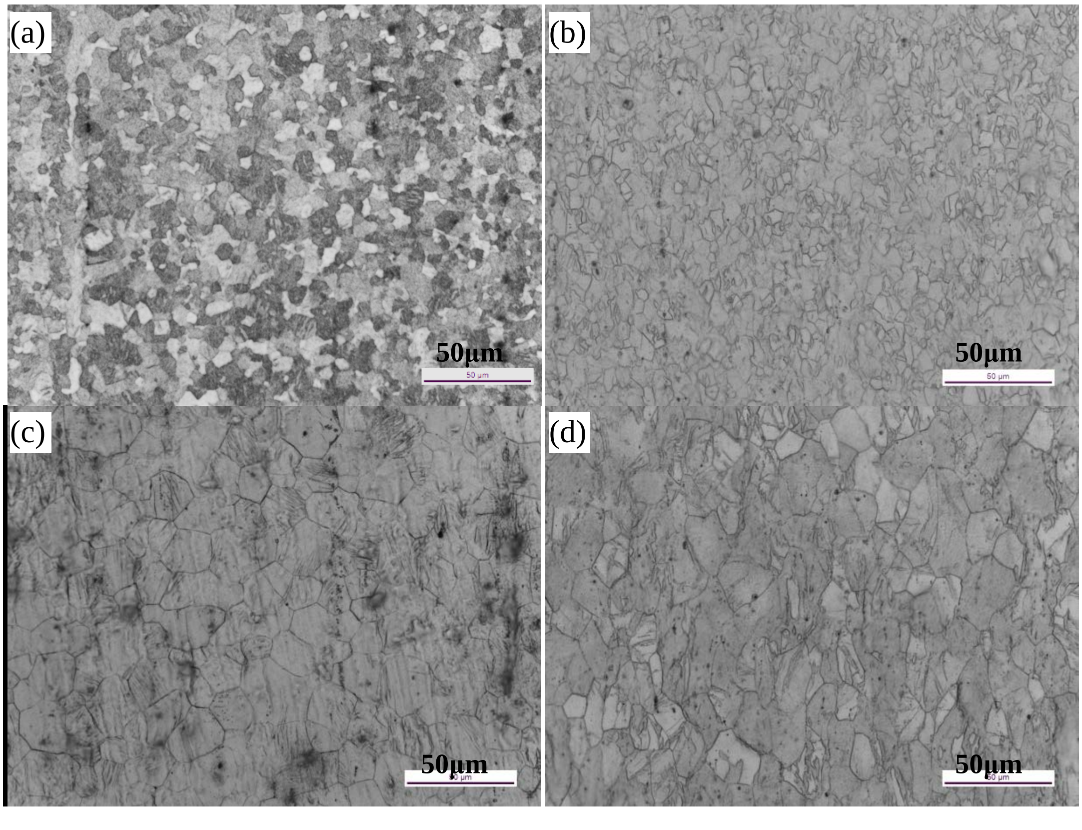

Figure 2. Optical microstructure of Mg-Zn-Gd alloy wires under different conditions. (a) as hot extruded(4.0 mm in diameter); (b) as-drawn 1# (3.6 mm in diameter); (c) as-annealed(3.6 mm in diameter); (d) as-drawn 2# (3.0 mm in diameter).

3.3. Tensile mechanical property of wires

Figure 3 illustrates the tensile mechanical property of Mg-Zn-Gd alloy wires under different conditions. The corresponding mechanical properties including ultimate tensile strength (UTS), 0.2% yield strength (YS) and breaking elongation (ε) are depicted, respectively. It is revealed that the yield-tensile ratio of as-drawn 1# and as-drawn 2# wires retains 0.58 and 0.79, respectively. A higher yield-tensile ratio of as-drawn 2# wires can be beneficial to application of implant materials. After annealed at 400 °C, the Mg alloy wires exhibit a significant decrease in the UTS and YS, but a significant increase in the breaking elongation. The elongation of as-drawn 1#, as-drawn 2# and as-annealed wires reaches 8%, 5% and 23%, respectively.

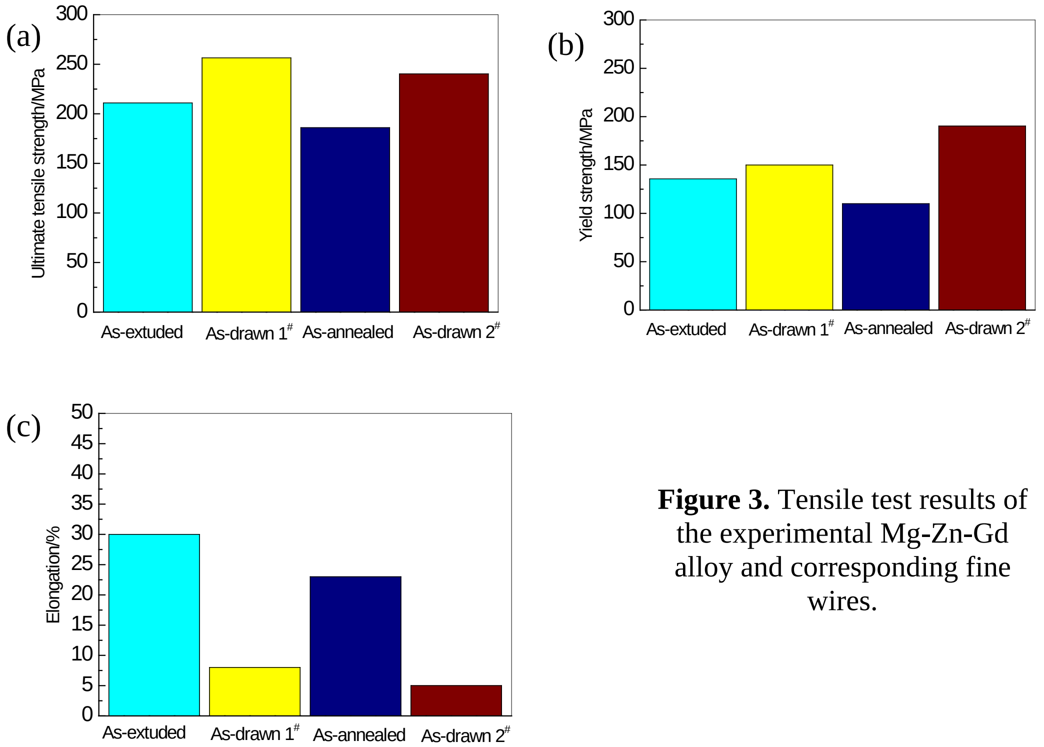

Figure 3. Tensile test results of the experimental Mg-Zn-Gd alloy and corresponding fine wires.

Further analysis shows that the room temperature tensile strength and elongation of as-extrude and 400 °C annealed wires assume the highly significant difference. The extruded wires have higher ultimate, yield strength, and even much higher elongation in comparison than as-annealed alloys, suggesting that the refinement of grains may be still a primary controlling factor on the mechanical properties of Mg-Zn-Gd wires during metal working.

3.4. Fractography

Figure 4 reveals the SEM images of the tensile fracture surfaces of the Mg-Zn-Gd alloy wires under different conditions. For all the Mg-Zn-Gd wires, the fracture surfaces appear to vary from one to another. The SEM image of as-extruded wires in figure 4a shows a complicated fracture surface, where there are shallow dimples with or without particles. After the as-extruded wires were drawn at room temperature, dimples become shallow, and brittle rupture surfaces are also observed (figure 4b). This is an explanation that the elongation of the as-drawn 1# wire was reduced. When the wires were annealed at 400 °C for 1 h, the dominant deep dimples came into being in the failure surfaces (figure 4c), which maybe account for the high ductility of the as-annealed wires. Figure 4d depicts the rupture surfaces of as-drawn wires. It can be seen that, the as-drawn wires have the combination of unevenly distributed brittle and ductile fracture characteristics. Therefore, it can be concluded that the Mg-Zn-Gd alloy wires have the combination of brittle and ductile fracture characteristics irrespective of the heat treatment and deformation.

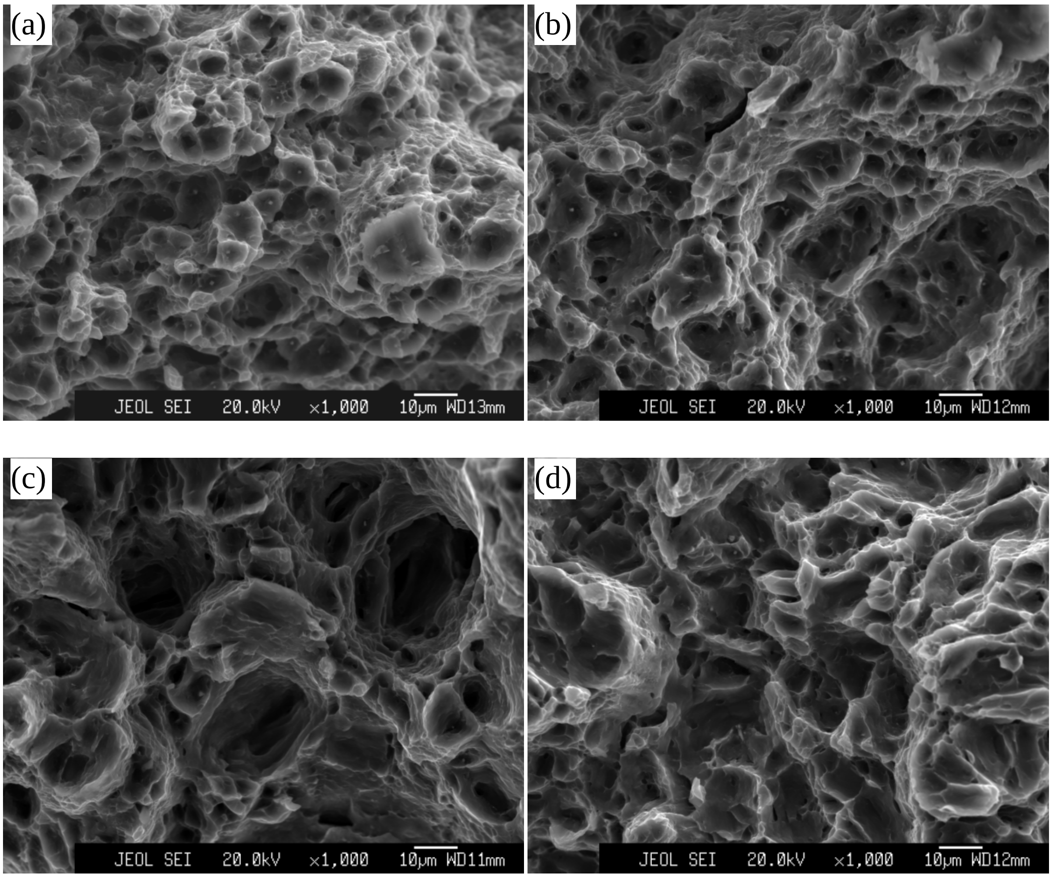

Figure 4. SEM images of the tensile fracture surfaces of Mg-Zn-Gd alloy wires under different conditions. (a) as-extruded(4.0 mm in diameter); (b) as-drawn 1# (3.6 mm in diameter); (c) as-annealed(3.6 mm in diameter); (d) as-drawn 2# (3.0 mm in diameter).

4. Conclusions

1) In comparison with the as-extruded wires, the as-annealed wires exhibit a greater average size of 12.7 μm.
2) The extruded wires exhibit nearly the breaking elongation of 30%. The as-drawn 1#, as-drawn 2# and as-annealed Mg-Zn-Gd alloy wires respectively show the YS of 150 MPa, 190 MPa and 110 MPa, and the breaking elongation of 8%, 5% and 23%. The yield-tensile ratio of as-drawn 1# and as-drawn 2# wires retains 0.58 and 0.79, respectively.
3) The tensile fracture surfaces of Mg-Zn-Gd alloy wires reveal that fracture mechanisms under different conditions are brittle and transgranular ductile fracture.

Acknowledgments

The present research has been fully supported by Guangdong province science and technology plan projects (Nos. 2013B010102025, 2014B010105009, 2014B070706027, 2014B090908006, 2014B090907008, and 2013B091602002), Guangdong provincial key laboratory for technology and application of metal toughening (No. 2014B030301012) and Guangzhou science and technology plan project (No. 201509010003).

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