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Microstructure and Tensile Properties of Mg-6Zn-3Sn-0.5Mn Alloy with Different Extrusion Temperature

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Abstract

The microstructure and mechanical properties of as-extruded Mg-6Zn-3Sn-0.5Mn alloy with different extrusion temperature before and after aging treatment are investigated systematically by the means of OM, SEM, TEM and tensile property test. The results indicate that the grain size of the extruding alloy increases with extrusion temperature increasing. At the same time, the grain size uniformity of the extruded tissue is significantly improved with the extrusion temperature increasing. A subsequent aging heat treatment was carried out on as-extruded Mg-6Zn-3Sn-0.5Mn alloy. During aging process many fine particles precipitate which result in the improvement of the yield strength. The best combination of strength and ductility of Mg-6Zn-3Sn-0.5Mn alloy is from the 400 oC extruded bar after ageing treatment of 150 oC for 64 h and the ultimate tensile strength (UTS), the yield strength (YS) and elongation (EL) are 412.4 MPa, 383.2 MPa and 8.4%, respectively.

Keywords

Mg-6Zn-3Sn-0.5Mn; Extrusion; Ageing Treatment; Microstructure; Mechanical Properties.

1. Introduction

Because of the growing demands for lightweight components thereby reducing energy consumption and air pollution of the automobile and aerospace industries, magnesium alloys and their composites have great potential as structural metallic materials due to their low density, high strength to weight ratio, good machinability and superior damping capacity [1-7]. However, the application of magnesium alloys is still limited because of the lack of commercial grades of alloys, most of which have moderate strength, poor ductility at room temperature and high cost comparing with steels and aluminum alloys [8-11]. In order to overcome these disadvantages, great efforts have been made to improve the mechanical properties of magnesium alloy both at room and elevated temperature, such as adding the other alloy elements [12-14], heat treatment [15-17], and some plastic deformation processes [18-20].

Recently, there has been an interest in Sn-containing magnesium alloys because of their good mechanical properties and deformability [21,22]. T.T. Sasaki et al. [23] report enhanced room-temperature tensile and creep properties of extruded Mg–Sn–Zn alloys by solution heat-treatment and

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artificial aging. Zhou Jixue et al. [24] studied the constitution and tensile Properties of semi-continuous cast Mg-6Al-6Zn-3Sn-1Y-0.5Mn alloy at casting state and heat treatment state. Dongqing Zhao et al. [25,26] reported the dynamic microstructural evolution in Mg–4Zn–2Al–2Sn alloy during hot deformation and carried out constitutive modeling for dynamic recrystallization kinetics of Mg−4Zn−2Al−2Sn alloy.

In our previous work [27], a new Mg-6Zn-3Sn-0.5Mn alloy with good mechanical properties and formability was developed. The microstructure and mechanical properties of as-cast and heat-treated Mg-6Zn-3Sn-0.5Mn alloy was analyzed. In order to furthermore confirm this alloy whether is suitable for extruding deformation and aging strengthening, this paper studied the microstructure and mechanical properties of Mg-6Zn-3Sn-0.5Mn alloys with different extrusion process and aging process. The study will be helpful for the future development of the high strength and low cost magnesium alloy.

2. Experimental Procedures

Commercial pure Mg ingot (99.9 wt.%), pure Zn ingot (99.99 wt.%), pure Sn ingot (99.99 wt.%) and Mg-10Mn (Mg-10 wt.% Mn) intermediate alloys ingot were used as raw materials to prepare Mg-6Zn-3Sn-0.5Mn (Mg-6wt.%Zn-3wt.%Sn-0.5wt.%Mn) alloy. The Mg-6Zn-3Sn-0.5Mn rod bar with the diameter of 120 mm was prepared by semi-continuous casting. The mould height is 450mm, and the casting speed is 80-100mm / min, and the secondary cooling water flow is 25L / min. The melting process was in a protective atmosphere (mixed gas of 1vol.% SF_6 and 99 vol.% CO_2).

The ingots were heat treated at 340^{o}C for 2 h then at 420^{o}C for 8 h to homogenized followed by air cooling. The ingots were cut to cylinder billet with the diameter of 110 mm, and then extruded at 300^{o}C, 350^{o}C, 400^{o}C with the extrusion ratio of 25 and followed by air cooling.

In order to obtain the aging hardening curves, the as-extruded alloys were aged at 150℃, 175℃, 200℃ and 225℃ for 2 hours, 4 hours, 8 hours, 16 hours, 32 hours, 64 hours and 128 hours respectively.

The metallographic samples were cut from the same positions and the microstructural observations were conducted on the midsections parallel to extrude direction (ED). Both the as-extruded and as-aged samples were etched with a mixture of 4.25g picric acid, 70ml ethanol, 10ml water and 10ml aceic acid. And microstructure observations were carried out by ZEISS2000-C optical microscope (OM) and SX2-12-10Y scanning electron microscope (SEM). The metallographic samples were further observed by Zeiss LIBRA 200 FE transmission electron microscopy (TEM). The hardness test was carried out by a micro-Vickers apparatus with 200 gram load and 30 seconds loading time. The tensile test was performed on microcomputer controlling electronic universal testing machine (the model DW-200E) at a strain rate of 2 mm/mim at room temperature. And the values of the ultimate tensile strength (UTS), the yield strength (YS) and elongation (EL) were the average of at least three specimens.

3. Results and discussion

3.1 Microstructure of as-extruded Mg-6Zn-3Sn-0.5Mn alloy

Fig. 1 shows the optical microstructure of as-extruded Mg-6Zn-3Sn-0.5Mn alloy taken from the normal plane to the extrusion direction. It can be seen that the grain size increases gradually with the extrusion temperature increasing. And the grain size of the alloy extruded at 300 ^{o}C, 350 ^{o}C and 400 ^{o}C is about 11.2μm, 16.8μm and 24.7μm respectively. The fine microstructure is attributed to the dynamic recrystallization and precipitation that occur during the extrusion. With the increase of extrusion temperature, recrystallization grains forming in the extrusion process of alloy tend to grow up which contribute to the coarser grains.

Fig. 2 shows optical micrographs taken from the parallel plane to the extrusion direction (ED) at 300 ^{o}C, 350 ^{o}C, 400 ^{o}C. It can be found that there are a lot of second phases with bright globular-liked appeared along the grain boundary and inside grains after extrusion deformation. According to the

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EDS analysis results in Table 1, the main components of irregular phases mainly contain Mg element and Sn element. It can be inferred that the irregular phases are the residue Mg_2Sn particles after solution treatment before extruding[27].

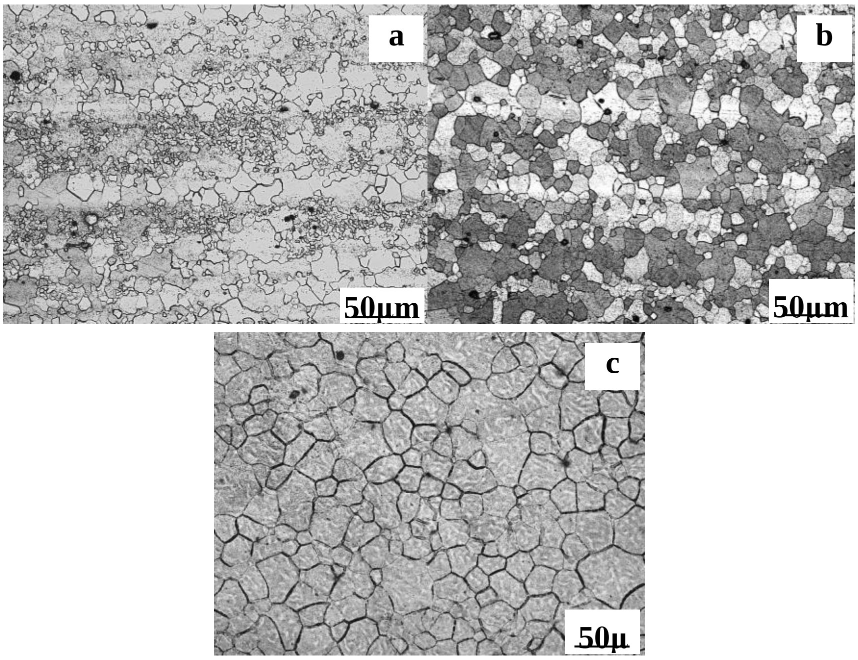

Fig. 1 Optical microstructures of Mg-6Zn-3Sn-0.5Mn alloy extruded at 300 oC (a), 350 oC (b), 400 oC (c)

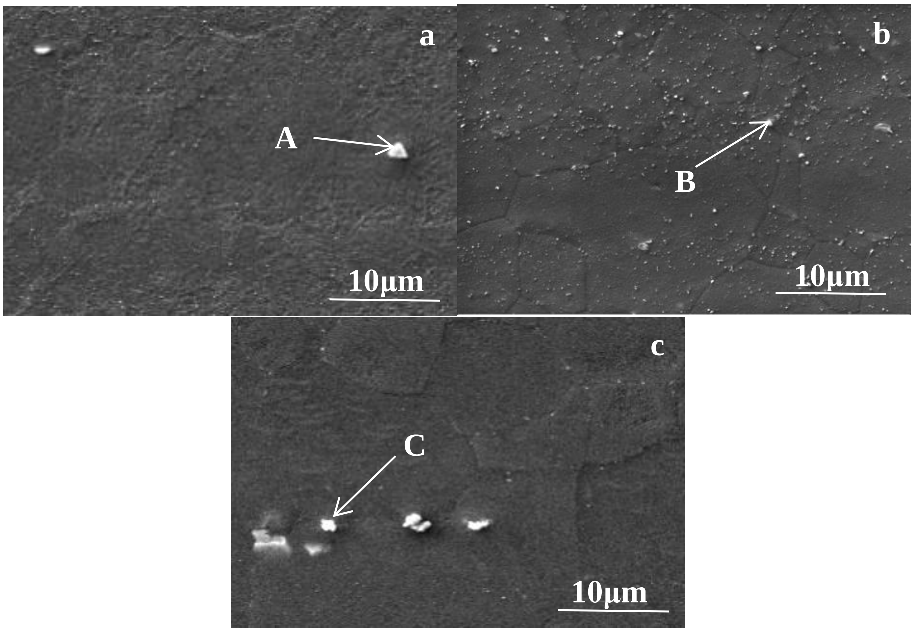

Fig. 2 SEM micrographs of as-extruded Mg-6Zn-3Sn-0.5Mn alloys at 300 oC (a), 350 oC (b), 400 oC (c)

Table 1. EDS analysis of areas in Fig. 2

Area	Mg (at. %)	Zn (at. %)	Sn (at. %)
A	69.03	0.93	30.04
B	64.52	0	35.48
C	73.66	0.95	25.40

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3.2 Mechanical properties of as-extruded alloys

The regular diagram of tensile properties of as-extruded Mg-6Zn-3Sn-0.5Mn alloys with various extrusion temperature are shown in Fig. 3.

It indicates that the ultimate tensile strength and the yield strength of the as-extruded Mg-6Zn-3Sn-0.5Mn alloy decrease with the extrusion temperature increasing. As the extrusion temperature is 300ºC, 350ºC, 400ºC, the ultimate tensile strength and the yield strength are 329.0 MPa and 243.6 MPa, 316.3 MPa and 233.8 MPa, 302.5 MPa and 228.1 MPa, respectively. However the elongation of as-extruded Mg-6Zn-3Sn-0.5Mn alloy increases with the extrusion temperature increasing, which arrives to 14.2 %, 14.5 % and 15.1 % as the extrusion temperature is 300ºC, 350ºC and 400ºC. It means that the decreasing of the extrusion temperature can enhance the ultimate tensile strength and the yield strength in some degree.

From the above conclusions, the reasons for the improvement of mechanical properties is attributed to the following aspects. The increasing of the extrusion temperature results in coarsening grain size (Fig. 1), which are all harmful for improving mechanical properties. Additionally, according to the Hall-Petch relation: $\sigma \propto 1/d^2$, grain size has an important role in the yield strength, that is the finer of the grain size, the higher of the yield strength. The refined grains prevent the motion of dislocation to improve the strength. It can be found form Fig. 1, with the increase of extrusion temperature, it is easier to be deformed during the extruding deformation process, which can improve the microstructure homogeneity of the alloy. And the improved microstructure homogeneity contribute to the higher elongation of as-extruded Mg-6Zn-3Sn-0.5Mn alloys, because the number and morphology of the equiaxed crystal and the plastic deformation organization play a decisive role in the elongation of the sample during recrystallization.

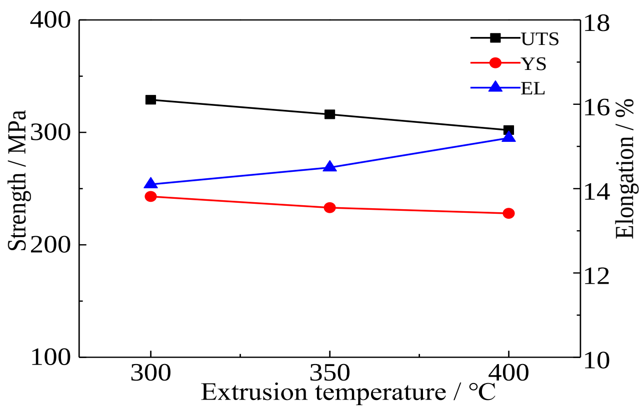

Fig. 3 Tensile properties of Mg-6Zn-3Sn-0.5Mn alloy at 300oC, 350oC, 400oC

3.3 Age-hardening behaviors of as-extruded alloys

Fig. 4 shows the microhardness curves of as-extruded Mg-6Zn-3Sn-0.5Mn alloy after aging treatment at 150 ℃, 175 ℃, 200 ℃ and 225 ℃ for 2 hours, 4 hours, 8 hours, 16 hours, 32 hours, 64 hours and 128 hours respectively. It can be seen that Mg-6Zn-3Sn-0.5Mn alloy has good aging hardening effect, which shows a typical aging hardening phenomenon. With the increase of aging time, the hardness of the alloy first increased and then decreased, which accorded to the characteristics of "under-aging, peak-aging, over-aging" during aging hardening process.

The effect of aging treatment on Mg-6Zn-3Sn-0.5Mn alloy is analyzed by microhardness test. Fig. 4a shows the aging hardening curves of the alloy extruded at 300 ºC. With different aging temperature, the peak-aging value appeared at the 225 ºC for 4 h, 200 ºC for 8 h, 175 ºC for 16 h, 150 ºC for 32 h and the the maximum peak-aging value reached 84.2 HV at 175 ºC for 16 h. Fig. 4b shows the aging hardening curve of the alloy extruded at 350 ºC. With different aging temperature, the peak-aging value appeared at the 225 ºC for 4 h, 200 ºC for 8 h, 175 ºC for 32 h, 150 ºC for 64 h and the the

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maximum peak-aging value reached 83.6 HV at 175 ºC for 32 h. Fig. 4c shows the aging hardening curve of the alloy extruded at 400 ºC. With different aging temperature, the peak-aging value appeared at the 225 ºC for 4 h, 200 ºC for 8 h, 175 ºC for 16 h, 150 ºC for 64 h and the the maximum peak-aging value reached 90.1 HV at 115 ºC for 64 h.

The best aging process is selected under three extrusion temperatures, as shown in Table 2. The optimal aging process for the alloy extruded at 300ºC is 175ºC for 16h, and the best aging process for the alloy extruded at 350ºC is 175ºC for 32h, while the best aging process for the alloy extruded at 400ºC is 150℃ for 64h.

It can be concluded from the above conclusions that the the peak value appears earlier with the increase of aging temperature for Mg-6Zn-3Sn-0.5mn alloy.

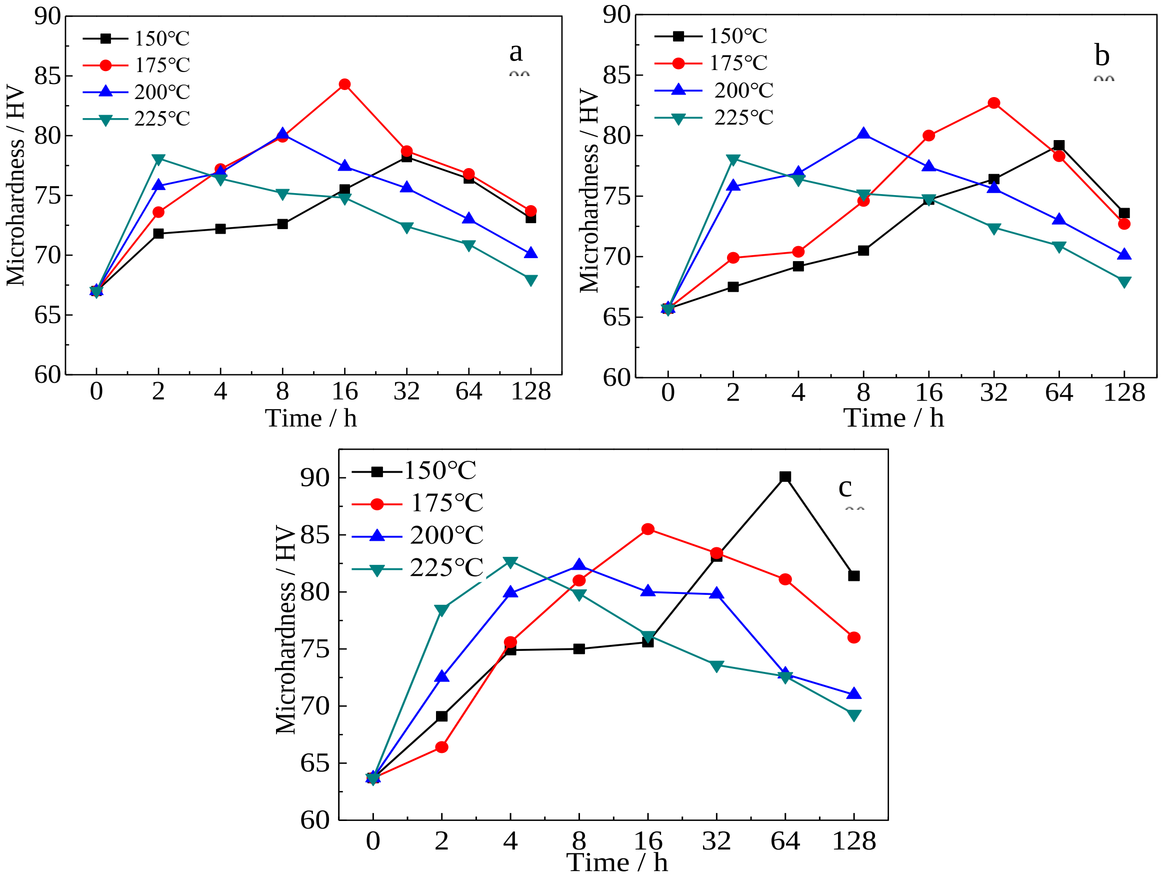

Fig. 4 Age-hardening curves of Mg-6Zn-3Sn-0.5Mn alloy extruded at 300oC (a), 350oC (b), 400oC (c)

Table 2. The optimal aging process of Mg-6Zn-3Sn-0.5Mn alloy under different extrusion temperature

Extrusion temperature / oC	Peak aged process
300	175 ºC × 16 h
350	175 ºC × 32 h
400	150 ºC × 64 h

3.4 Microstructure of peak-aged alloys

Fig. 5 shows typical SEM images of Mg-6Zn-3Sn-0.5Mn alloy under the peak aging process of Table. 2 at different extrusion temperatures. It reveals that a large number of secondary phases precipitate at the grain boundaries and grain interior presenting a dispersion distribution. These precipitates have

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very small grain size, ranging from a few to hundreds of nanometers. The size of precipitates appears smaller, while the density becomes larger and the dispersion distribution becomes more uniform with the increase of the extrusion temperature of Mg-6Zn-3Sn-0.5Mn alloy. This is because the higher extrusion temperature contributes to high solid solubility and better microstructure homogeneity during extruding process, which makes the second phases precipitate in large quantities and present fine granular and dispersed distribution.

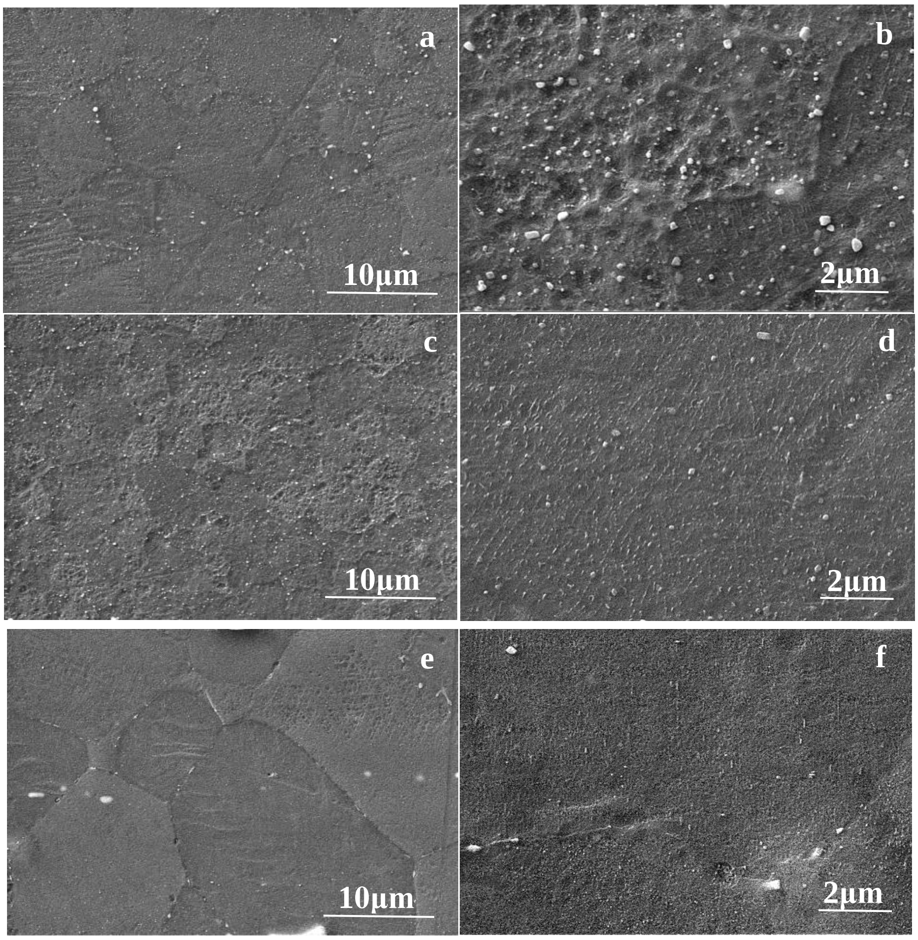

Fig. 5 SEM images of Mg-6Zn-3Sn-0.5Mn extruding at aging under different extrusion temperature, 300oC(a, b)350oC(c, d)and 400oC(e, f)

Fig. 6a shows the bright field TEM image of Mg-6Zn-3Sn-0.5Mn alloy aging at 150 ºC for 64 h taken from [0002] zone axes. A large number of secondary phases precipitate after aging treatment, which is consistence with the SEM results from Fig. 5. There are three kinds of typical morphologies: rod-liked particles (marked as A), plate-liked particles (marked as B) and and sphere-liked particles (marked as C).

The bright filed TEM image of particle A is shown in Fig. 6 (a) and the FFT pattern of particle A is shown in Fig. 6 (b), from which the rod-liked particle A is confirmed as $MgZn_2$ phase. Fig. 6 (c) shows the bright filed TEM image of B and it can be seen that B is rich in Mg and Zn from the EDS result as shown in Fig. 6 (d). The plate-liked particle B is approved to be Mg-Zn phase. Wang et al [15] have reported that plate-liked Mg-Zn particles formed in the peak-aged ZAT422 alloy at T5 heat treatment (150 ºC×40 h). The bright filed TEM image of C is shown in Fig. 6 (e) and the FFT pattern of C is shown in Fig. 6 (f), from which the sphere-liked particles is Mg_2Sn phase. So the secondary precipitates in as-extruded ZTM630 alloy after aging treatment consist of Mg-Zn, $MgZn_2$ and Mg_2Sn phases.

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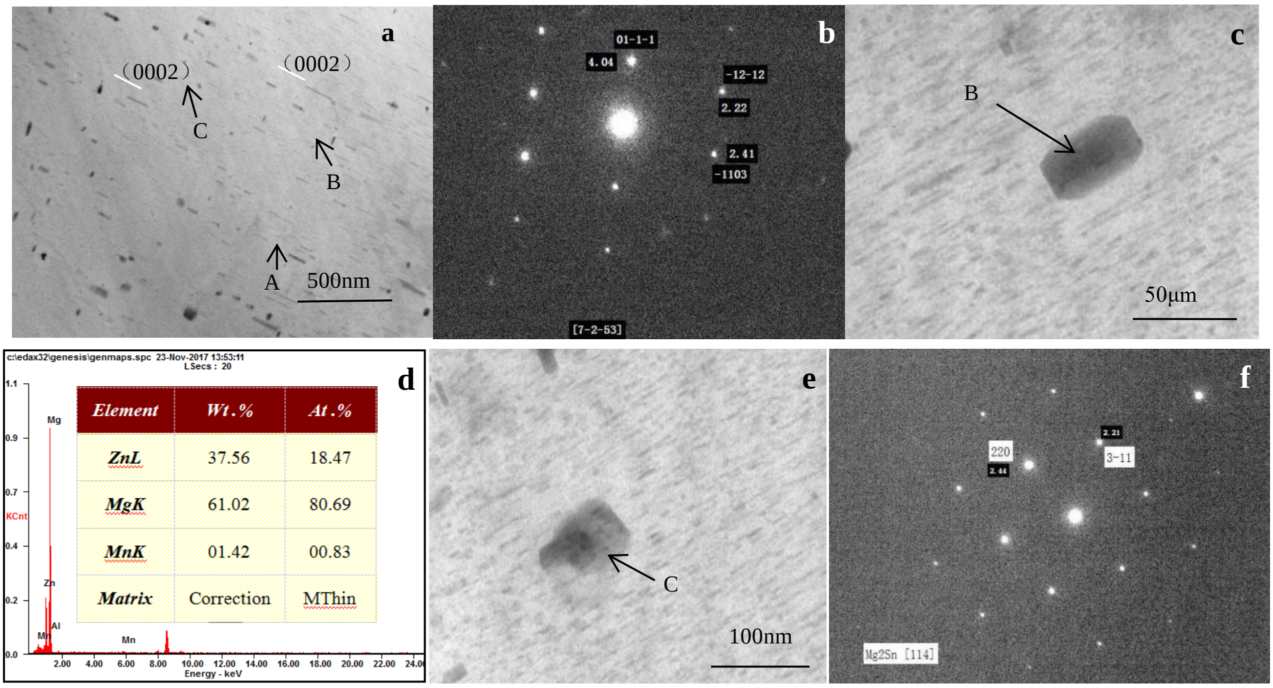

Fig. 6 The bright field TEM images (a), the FFT pattern (b) of particle A, the morphology (c) and the EDS result (d) of particle B, the morphology (e) and the FFT pattern (f) of particle C in the peak-aged Mg-6Zn-3Sn-0.5Mn alloy extruding at 400 oC with aging treatment

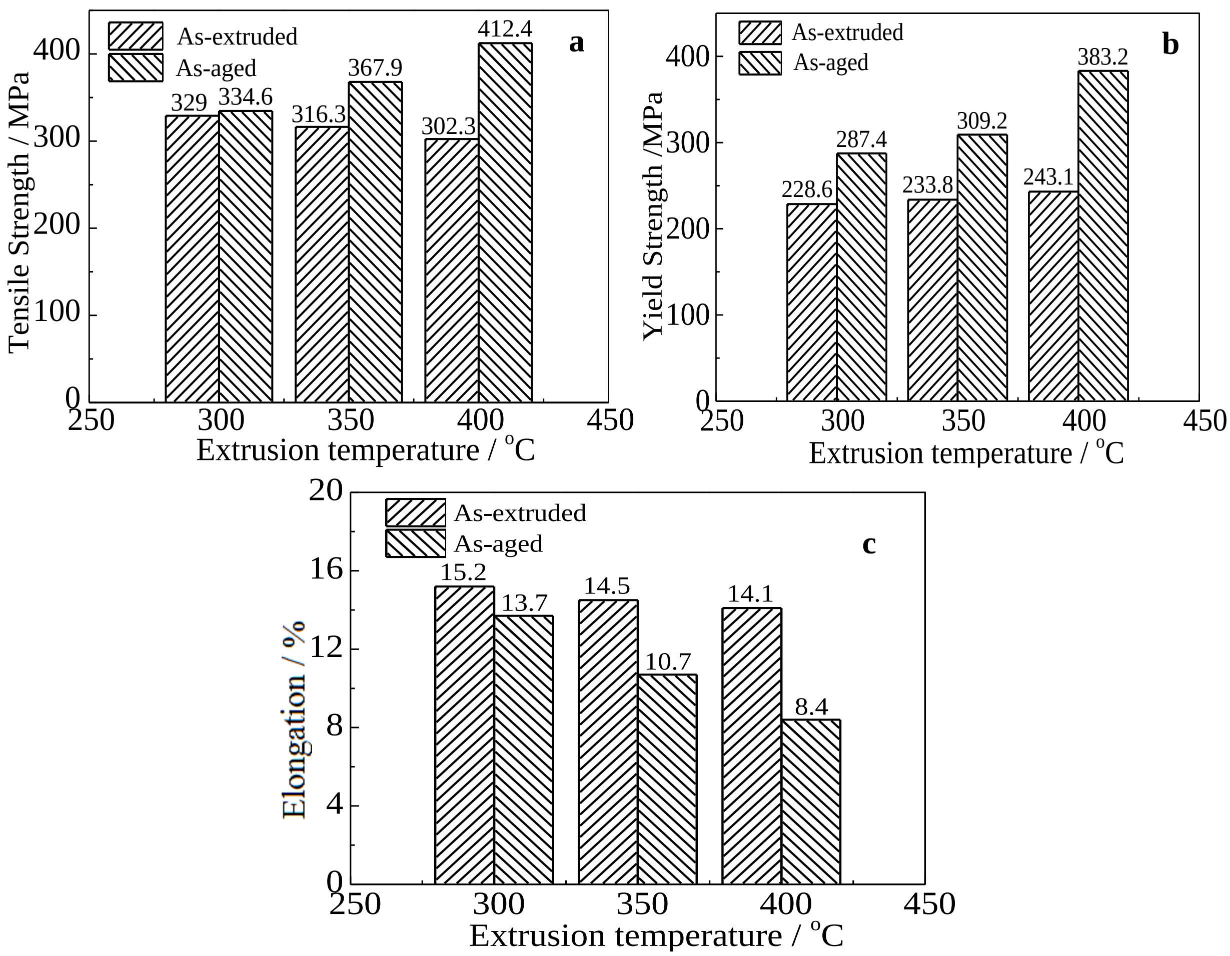

Fig. 7 Mechanical properties of Mg-6Zn-3Sn-0.5Mn magnesium alloy extruding rods before and after aging treatment

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3.5 Mechanical properties of peak-aged alloys

The mechanical properties of peak aged samples of different extrusion temperature are shown in Fig. 7. It reveals that the peak-aged treatments resulted in a significant increase in the strengths of all the as-extruded alloys, while the elongations decrease slightly. The Mg-6Zn-3Sn-0.5Mn alloy extruded at 400ºC with aging treatment of 150ºC for 64h arrived at the highest tensile strength and yield strength, which reached 412.4 MPa and 383.2 MPa respectively and the elongation is 8.4 %. These strengths almost arrived to the strength of RE-containing magnesium alloys treated by T5 or T6, including Mg–Gd–Y–Zn–Zr [28], and Mg–Y–Sm–Zr [29].

During aging treatment, fine Mg-Zn and Mg_2Sn particles precipitate within the matrix and along the grain boundaries. These particles have large effect on the mechanical properties because they act as obstacles to the dislocation movement, which can improve the tensile strength and yield strength. It has been confirmed that different precipitate shapes and orientations exerted different effects on the strength of Mg alloys [2] and the fine Mg_2Sn precipitates with different dimensions and orientations could improve the strength [30].

It can be found from Fig. 7a that the tensile strength of Mg-6Zn-3Sn-0.5Mn as-extruded alloy increases with the increase of extrusion temperature after aging, which is contrary to the change trend of the tensile strength of the alloy with temperature changing before aging. The aging strengthening effect is closely related to the solid solubility of alloy elements in matrix. High extrusion temperature can make the alloy obtain higher solid solubility of alloy elements during the extruding process, so that more strengthening particles can precipitate in the subsequent aging process.

Therefore, as-extruded Mg-6Zn-3Sn-0.5Mn alloy extruded at 400ºC and aged at 150ºC for 64 h exhibits the preferably mechanical properties at room temperature.

4. Conclusions

The microstructure and mechanical properties of as-extruded Mg-6Zn-3Sn-0.5Mn alloy with different extrusion temperature before and after aging treatment were investigated in this work. The following conclusions were summarized.

(1) The grain size of the as-extruded alloy increases with extrusion temperature increasing, which contributes to the tensile strength and the yield strength decreasing. The elongation increases with the increase of extrusion temperature because the grain uniformity of the extruded alloy is significantly improved by higher temperature extrusion.

(2) Mg-6Zn-3Sn-0.5Mn alloy has typical aging treatment hardening characteristics. The aging treatment results in a significant increase of the tensile strength and yield strength due to the fine precipitate particles act as obstacles to dislocation movement.

(3) The best mechanical properties were obtained at the 400ºC extrusion temperature after 150ºC for 64 h aging treatment. The tensile strength, yield strength and elongation reached 412.4 MPa, 383.2 MPa, and 8.4 %, respectively.

Acknowledgments

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