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ARTICLE

Effect of Heat Treatment on Microstructure and Tensile Properties of Ultrasonically Treated As-extruded Mg-6Zn-0.5Y-2Sn Alloy

Xue Hansong[1,2], Chen Yangyang[1], Huang Jie[1], Li Xinyu[1], Rao Jinsong[1], Pan Fusheng[1,2]

[1] College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China; [2] National Engineering Research Center for Magnesium Alloys, Chongqing University, Chongqing 400044, China

Abstract: The effect of heat treatment on the microstructure and tensile properties of an ultrasonically treated as-extruded Mg-6Zn-0.5Y-2Sn alloy was investigated. The results show that after the aging treatment and solid solution+aging treatment, the grain size of the test alloys becomes more uniform. The MgSnY and Mg_2Sn phases are much more dispersed and increased in amount. In addition, both the aging and solid solution+aging treatment improve the yield strength of the test alloys, especially, the aging treatment significantly improves the yield strength from 165 MPa to 269 MPa, increased by 63.1%. The precipitation strengthening, which is derived from the MgSnY and Mg_2Sn phase, is the major factor for improving the yield strength of the test alloys with heat treatment. Moreover, the aging treatment more effectively improves the comprehensive tensile properties of test alloy compared with the solid solution+aging treatment. The mechanisms for the experimental observations were also discussed.

Key words: Mg-6Zn-0.5Y-2Sn alloy; ultrasonical treatment; heat treatment; yield strength; tensile properties

In Mg alloys, the rare earth (RE) elements have been used for improving the mechanical properties [1]. For example, Yttrium (Y) is beneficial to the tensile properties[2-4] of Mg alloy. Sn can enhance the precipitation strengthening of Mg-Zn alloy due to the formation of Mg_2Sn phase[5,6]. In addition, the Mg-Zn-Y-Sn quaternary alloy was investigated because the combined addition of Sn and Y in the Mg-Zn alloy can form several phases such as $MgZn_2$, Mg_2Sn and MgSnY, which result in superior comprehensive tensile properties [7, 8].

Ultrasonic treatment (UST) is an effective way to modify the microstructure and to improve the properties of the light alloy [9]. In our previous study, the coarse dendrites are changed into roundish equiaxed grains, and the second phases become fine and dispersed in the Mg-6Zn-0.5Y-2Sn alloy with ultrasonic treatment. Additionally, the UTS, YS and elongation reach 246 MPa, 83 MPa and 20.2%, increased by 30%, 43% and 67%, respectively[7].

Heat treatment is also effective in improving the tensile properties of the Mg alloy. In a Mg-Zn based alloy, double aging (T5) exhibits better age-hardening response than single aging. Pre-aging provides more GP zones to be nucleation sites in the subsequent aging process, and thus the precipitates after double aging become finer and more dispersed [10-12].

However, there are few reports concerned with the effect of heat treatment on the microstructure and tensile properties of ultrasonically treated as-extruded Mg-Zn-Y-Sn quaternary alloy. Based on our previous work, we further investigated the effect of heat treatment on the microstructure and tensile properties of an ultrasonically treated as-extruded Mg-6Zn-0.5Y-2Sn alloy. The study might be helpful for the further development of high strength heat-treatable magnesium alloys.

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Corresponding author: Xue Hansong, Ph. D., Associate Professor, College of Materials Science and Engineering, Chongqing University, Chongqing 400044, P. R. China, Tel: 0086-23-65111212, E-mail: hsxue@sohu.com

1 Experiment

The raw materials used were highly pure Mg (>99%), Zn (>99%), Sn (>99%) and Mg-30%Y master alloy. The alloy was melted in an electrical furnace protected by CO_2+0.5% SF_6 mixed gas with a volume ratio of 99:1. The melt was maintained at 720 °C for 20 min to fully homogenize the alloying elements. Then it was poured into a stainless steel mold at 680 °C, and an ultrasonic treatment (UST) was employed with an ultrasonic horn immersed in the melt for 20 s under a power of 700 W. The ultrasonic system consisted of an ultrasonic transducer, an ultrasonic horn and an ultrasonic probe. The frequency was 20 kHz and the maximum power was 1 kW. The ultrasonically treated ingots were homogenized at 420 °C for 12 h and then extruded at 360 °C with an extrusion ratio of 25:1. The ultrasonically treated as-extruded specimens were subjected to the aging treatment (T5) and the solid solution+aging treatment (T6). We have previously found that pre-aging at 90 °C for 24 h results in dispersed GP zones in Mg-6Zn alloys, and its yield strength after pre-aging at 90 °C for 24 h followed by secondary aging at 180 °C for 8 h reaches 281 MPa, which is 9% higher than that with single aging at 180 °C for 8 h. Accordingly, T5 treatment consisted of pre-aging at 90 °C for 24 h, followed by secondary aging at 180 °C for 8 h and final quenching in water. For T6 treatment, the specimen was solid-solution treated at 420 °C for 2 h, followed by two-step aging as above. Herein, the ultrasonically treated as-extruded Mg-6Zn-0.5Y-2Sn alloy is referred as the test alloy in the following paper. The actual chemical composition of the Mg-6Zn-0.5Y-2Sn alloy is listed in Table 1.

The microstructure was observed by an optical microscope (OM, XJP-6A), and a scanning electron microscope (SEM, Tescan Vega Ⅱ LMU) equipped with an energy dispersive X-ray spectrometer (EDS, Oxford Inca Energy 350). The specimens were mechanically polished and etched in a 4% nitric acid in ethanol solution. The phase composition was examined with X-ray diffraction (XRD, D/MAX-2500PC) using Cu Kα radiation at a scanning angle of 10°~90° and a scanning rate of 4°/min. And the actual chemical composition was tested by an X-ray fluorescence spectrometer (XRF-1800 CCDE). The tensile test was performed on an electronic universal testing machine (SANS CMT-5105) at a rate of 2 $mm \cdot min^{-1}$ at room temperature.

Table 1 Actual chemical composition of the Mg-6Zn-0.5Y-2Sn alloy (wt%)

Mg	Zn	Y	Sn
Bal.	5.54	0. 47	1.91

2 Results and Discussion

2.1 Microstructure of the alloys before heat treatment

Fig.1 shows that in the as-extruded Mg-6Zn-0.5Y-2Sn alloy without ultrasonical treatment, large grains along with small grains coexist in the matrix with uneven grain size. In contrast, with the application of the UST, the grains become finer with an average grain size of approximately 15 μm (standard deviation: 0.4 μm). The coarse dendrite α-Mg grains are changed into roundish equiaxed grains. During the process of UST, a large quantity of cavitation bubble is formed and continues to expand in the melt. The expansion of the bubbles requires heat from the local melting region. Thus, the temperature of bubbles/melt interface decreases to enhance the heterogeneous nucleation in the melt. Moreover, these bubbles finally collapse, which induces a powerful pressure pulse as large as 100~1000 MPa[13]. According to the Clausius-Clapeyron equation[14]:

$$\Delta T_{\mathrm{m}} = \frac{T_{\mathrm{m}} \Delta P (V_{\mathrm{L}} - V_{\mathrm{S}})}{\Delta H} \quad (1)$$

where T_{m} and P are the freezing point and pressure, respectively; V_{L} and V_{S} represent the volume of the liquid and solid phases, respectively; ΔH is the latent heat of freezing. The rapid increase in pressure leads to a rise in ΔT_{m}, which enhances heterogeneous nucleation. This mechanism explains why the grains become finer after the UST.

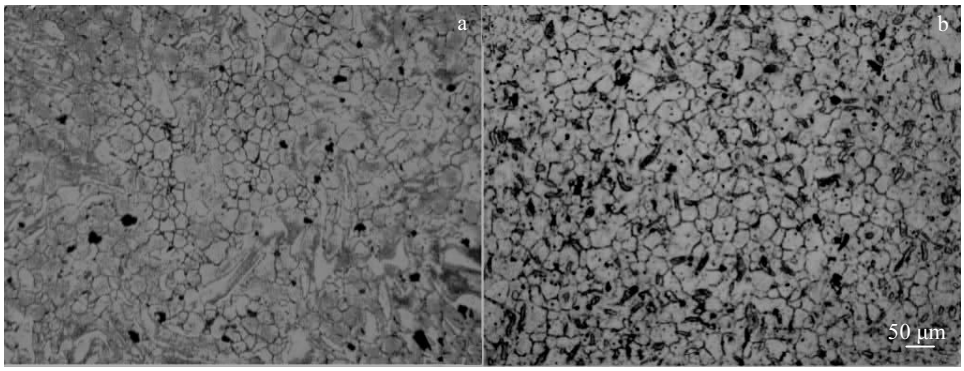

Fig.1 Metallographic structure of the as-extruded Mg-6Zn-0.5Y-2Sn alloy without (a) and with (b) ultrasonical treatment

Fig.2 shows the XRD pattern of the test alloy. The test alloy mainly consists of α-Mg, MgSnY and Mg_2Sn phase. Some I phase (Mg_3Zn_6Y) dissolved in α-Mg matrix during homogenization process, and other I phase was broken into smaller particles, which are then dissolved in α-Mg matrix during extrusion process. This is why the I phase cannot be detected in the XRD pattern of the test alloy.

According to the mathematical expression of effective distribution coefficient k_e:

$$k_e=C_s/C_l=k_0/[k_0+(1-k_0)\ e^{-R\delta/D}] \quad (2)$$

where C_s is solute concentration in solid phase and C_l is that in liquid phase, k_0 is equilibrium distribution coefficient, R represents the moving speed of solidification interface, δ is the thickness of boundary layer, and D is diffusion coefficient. With the presence of ultrasonic field, the cooling rate increases[15], thereby the moving speed of solidification interface increasing, so $e^{-R\delta/D}$ decreases, and then k_e in the mathematical expression increases, namely the element concentration in solid phase increases. The solid solubility of solute elements in the matrix is improved after UST. As a result, the UST can promote the dissolution of the second phases into the matrix.

From Fig.3, it can be seen that the MgSnY phases are dispersed, suggesting that the UST can promote the dissolution of the second phases into the matrix, and the extrusion process can crush the Mg_2Sn particles. Thus, the Mg_2Sn phase is broken into very small particles which are uniformly dispersed into the matrix. Therefore, it is difficult to identify the Mg_2Sn phase after the UST.

2.2 Effect of heat treatment on the microstructure of the test alloys

Fig.4 shows the metallographic structures of test alloys with heat treatment. The results show that the grain size all becomes more uniform. The average grain size of the test alloy with T5 treatment slightly increases from 15 μm (standard deviation: 0.4 μm) to 18 μm (standard deviation: 0.8 μm), while the average grain size of test alloy with T6 obviously increases from 15 μm (standard deviation: 0.4 μm) to 29 μm (standard deviation: 1.8 μm). After T6 treatment, the grains are larger

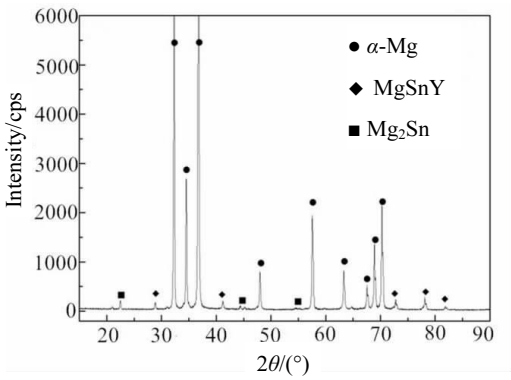

Fig.2 XRD pattern of the test alloy

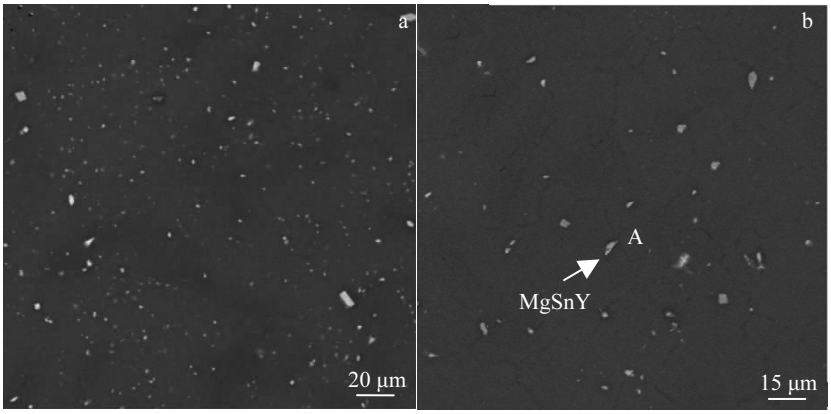

Fig.3 SEM morphologies of the Mg-6Zn-0.5Y-2Sn alloy (a) and MgSnY phase (b)

because the solid solution process can drive the grains to grow more rapidly. The UST and heat treatment promote the second phases to dissolve into the matrix, so the eutectic compounds in grain boundaries nearly disappear.

Fig.5 shows the SEM morphologies of test alloys with heat treatment. From Fig.3a, Fig.5a and Fig.5c, the volume fraction of second phases was calculated by Image-Pro Plus software. The results of sample without heat-treatment, and T5 and T6 treated samples are 1.0%, 3.95% and 3.50%, respectively. It can be seen that the amount of second phases obviously increases after T5 and T6 treatment. Compared to the test alloy with T6 treatment, there are more second phases in the test alloy with T5 treatment. The EDS results (Table 2) of Fig.5b and Fig.5d show that the second phases are unchanged. The UST can promote Sn and Y dissolve into the matrix; therefore the MgSnY and Mg_2Sn phases precipitate in subsequent aging process. As a result, these precipitated phases are still the MgSnY phase and a small amount of the Mg_2Sn phase. After heat treatment, these MgSnY and Mg_2Sn phases are uniformly dispersed.

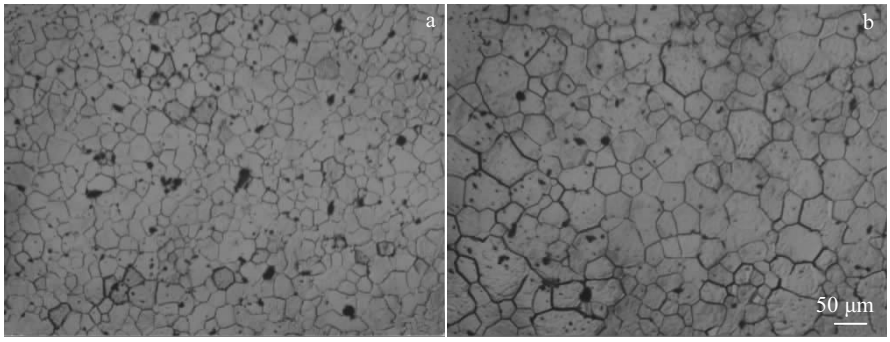

Fig.4 Metallographic structure of the test alloys with T5 (a) and T6 (b) treatment

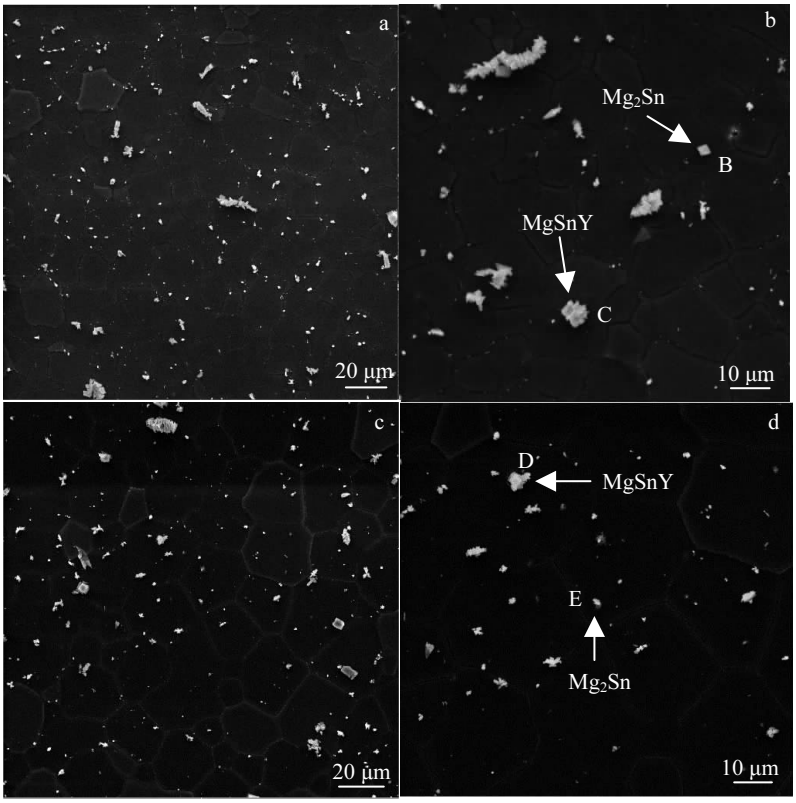

Fig.5 SEM morphologies of the test alloys with T5 (a, b) and T6 treatment (c, d)

Table 2 EDS results of the points indicated in Fig.3 and Fig.5

Point	Element content/at%				Phase
	Mg	Zn	Y	Sn	
A	37.38	3.39	31.8	27.42	MgSnY
B	65.14	2.25	0.68	31.93	Mg_2Sn
C	33.89	2.74	33.62	29.75	MgSnY
D	36.3	1.89	30.59	31.22	MgSnY
E	67.06	0.57	0.2	32.17	Mg_2Sn

Double aging can result in a more uniform distribution of precipitates[10, 16]. As a result, the MgSnY and Mg_2Sn phases are much more dispersed in test alloys with heat treatment. From Fig.5, the MgSnY and Mg_2Sn phases in the test alloy with T5 treatment are more dispersed than those in the test alloy with T6 treatment. The high temperature during the heat treatment brings about a small number of coarsened MgSnY particles in the test alloys with heat treatment. And it can be noticed that the test alloys with T5 and T6 treatment have similar size of the MgSnY and Mg_2Sn phase. In a word, after the heat treatment, the grain size of the test alloys becomes more uniform, and the second phases are more dispersed and also increased in amount. Meanwhile, the second

phases in the test alloys with T5 and T6 treatment have similar size. But the test alloy with T5 treatment has more second phases and they are more dispersed than those in the test alloy with T6 treatment.

2.3 Effect of heat treatment on the tensile properties of the test alloys

The tensile property results of the test alloys are listed in Table 3, and Fig.6 shows the tensile test results of the test alloys at room temperature.

For the test alloy without heat treatment, the yield strength σ_y can be estimated as[1]:

$$\sigma_y=\sigma_{Mg}+\sigma_{gb}+\sigma_{ss} \quad (3)$$

where σ_{Mg}=21 MPa for pure Mg. The parameter σ_{gb} represents the grain boundary strengthening, which can be estimated as:

$$\sigma_{gb}=\sigma_{Mg}+kd^{-1/2} \quad (4)$$

where k is 298 MPa·μm$^{1/2}$ for the Mg-6Zn alloys[17] and d is the average grain size. The parameter σ_{gb} is calculated to be 97.9 MPa and σ_{ss} represents the solid solution strengthening. Considering three elements (Zn, Y, Sn) were added into the test alloy, σ_{ss} can be estimated as[1]:

$$\sigma_{ss}=(k_{Zn}^{3/2}C_{Zn}+k_{Y}^{3/2}C_{Y}+k_{Sn}^{3/2}C_{Sn})^{2/3} \quad (5)$$

where k_{Zn}, k_Y, and k_{Sn} are the strengthening constants, k_{Zn} = k_{Sn} =905 MPa/%$^{2/3}$, and k_Y=1249 MPa/%$^{2/3}$. The solid solution content of Zn, Y, Sn in the matrix are calculated to be 5.54 wt%, 0.026 wt% and 1.05 wt%, respectively. C_{Zn}, C_Y and C_{Sn} are the concentration of the solution in terms of mole fraction, which are 2.1%, 0.007% and 0.2%, respectively. The σ_{ss} is calculated to be 75 MPa. As seen, σ_{Mg}, σ_{gb} and σ_{ss} all contribute to the yield strength in the test alloy. Among them, grain boundary strengthening is the primary factor. The calculated YS of the test alloy without heat treatment is 194 MPa, which is close to the experimental YS of 165 MPa. There are two primary reasons for the discrepancy between two values. First, the dispersion strengthening should be considered in the expression of σ_y, which needs to be further improved. Second, the k coefficient used in the Hall-Petch equation may not be totally accurate.

With T5 treatment, the YS of the test alloy increases from 165 MPa to 269 MPa, increased by 63.1%. For the T5 treatment, the yield strength σ_y can be estimated as[18]:

$$\sigma_y=\sigma_{Mg}+\sigma_{gb}+\sigma_{ppt} \quad (6)$$

The σ_{gb} is calculated to be 91.2 MPa. The average grain size of the test alloy with T5 treatment increases from 15 μm (standard deviation: 0.4 μm) to 18 μm (standard deviation: 0.8 μm), $\Delta\sigma_{gb}$=–6.7 MPa, so the grain boundary strengthening indeed slightly weakens.

Table 3 Tensile properties of the test alloys with different heat treatments

Treatment	UTS/MPa	YS/MPa	EL/%
As-extruded	327.2	165	18
T5	330.8	269	11.4
T6	318	218.3	13

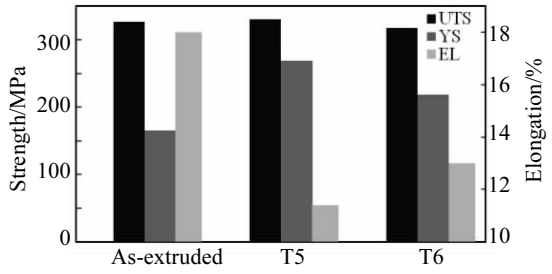

Fig.6 Tensile test results of the test alloys at room temperature

The σ_{ppt} represents the precipitation strengthening, and according to the Orowan mechanism, σ_{ppt} can be estimated as:

$$\sigma_{ppt}=\frac{Gb}{2\pi\sqrt{1-\upsilon}(0.825\sqrt{(d_t t_t/f_v)}-0.393d_t-0.886t_t)}\cdot\ln\frac{0.886\sqrt{d_t t_t}}{b} \quad (7)$$

where G is the shear modulus of pure Mg, b is the magnitude of the Burgers vector, υ is the Poisson' ratio; d_t, t_t, and f_v are the mean diameter, average thickness and the volume fraction of precipitates, respectively. Compared to the experimental YS of the test alloy with T5 treatment, the calculated YS is 112.2+σ_{ppt} MPa. The results show that σ_{ppt} is the dominant factor for improving the YS of the test alloy with T5 treatment. In other words, precipitation strengthening plays the most important role in enhancing the YS for the test alloy with T5 treatment.

With T6 treatment, the YS of the test alloy increases from 165 MPa to 218.3 MPa, increased by 32.1%. For the T6 treatment, the yield strength σ_y can be estimated as[1]:

$$\sigma_y=\sigma_{Mg}+\sigma_{gb}+\sigma_{ppt} \quad (8)$$

σ_{gb} is calculated to be 76.3 MPa. The average grain size of the test alloy with T6 treatment increases from 15 μm (standard deviation: 0.4 μm) to 29 μm (standard deviation: 1.8 μm), $\Delta\sigma_{gb}$=–21.6 MPa. Thus grain boundary strengthening weakens after T6 treatment. Compared to the experimental YS of the test alloy with T6 treatment, the calculated YS is 97.3+σ_{ppt} MPa. Thus, the σ_{ppt} is also proven to be the dominant factor for improving YS of test alloy with T6 treatment.

After heat treatment, the amount of second phases increases, and the MgSnY and Mg_2Sn phase distribution is extremely dispersed in the matrix. Precipitation strengthening can be enhanced with more dispersed second phases in the matrix. Thus, these well-dispersed MgSnY and Mg_2Sn phases can substantially improve the YS of test alloys through precipitation strengthening. In a word, the precipitation strengthening derives from the MgSnY and Mg_2Sn phases.

The elongation of test alloys with T5 treatment and T6 treatment is 11.4% and 13%, decreased by 36.5% and 27.8%, respectively. From Fig.5, a part of the MgSnY phase coarsens either in the grain boundary or inside the grain after heat treatment. Usually, the compounds in the grain boundary can prevent grains from moving during deformation process, so the coarsened MgSnY phase in the grain boundary is harmful for the ductility[19]. In addition, the coarsened MgSnY phase easily induces stress concentration at the second phase/matrix interface, so it is more inclined to act as initiation of microcrack. Thus, the elongation of the test alloys with T5 treatment and T6 treatment decreases.

T5 treatment is more effective in improving comprehensive tensile properties of test alloy than T6 treatment because the test alloy with T5 treatment has more second phases and more dispersed distribution of second phases than the test alloy with T6 treatment.

2.4 Fracture analysis

Fig.7 shows SEM morphologies of fracture surfaces of the test alloys. From Fig.7a, after the UST, there are cleavage planes and steps, and river patterns can also be seen. The test alloy without heat treatment has more dimples than the alloys with heat treatment. In addition, the bulky MgSnY phase can act as the origin of microcrack. From Fig.7b and Fig.7c, after T5 and T6 treatment, the cleavage planes become larger, indicating that the plasticity is lowered, which is in line with the experimental value of elongation. However, the test alloy with T6 treatment has more dimples than the test alloy with T5 treatment, which indicates that the former has slightly better plasticity than the latter. All the fracture surfaces demonstrate a characteristic of quasi-cleavage.

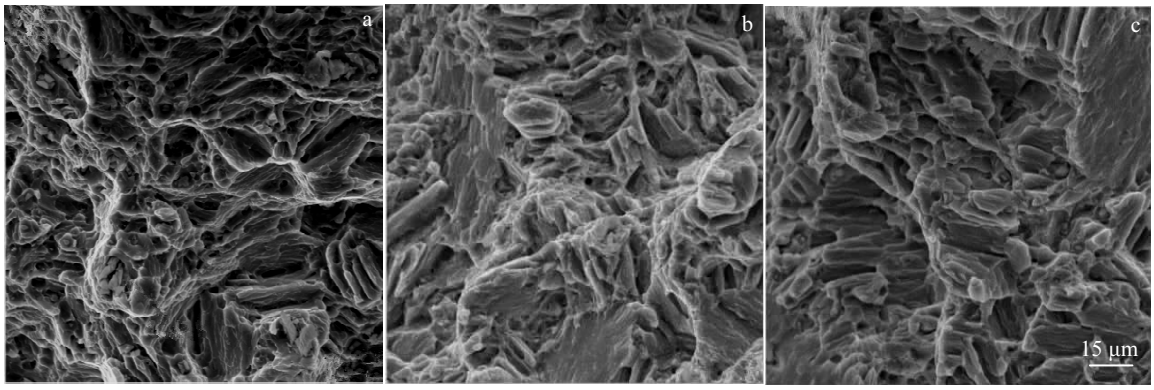

Fig.7 SEM morphologies of fracture surfaces of the test alloys: (a) without heat treatment, (b) with T5 treatment, and (c) with T6 treatment

3 Conclusions

1) The heat treatment results in more uniform grain size and much more dispersed distribution of the second phases with its amount increasing in Mg-6Zn-0.5Y-2Sn alloys.

2) The yield strength of Mg-6Zn-0.5Y-2Sn alloys with T5 and T6 treatments increases from 165 MPa to 269 MPa and to 218.3 MPa, increased by 63.1% and 32.1%, respectively.

3) The precipitation strengthening derived from the MgSnY and Mg_2Sn phases plays the most important role in improving the yield strength of Mg-6Zn-0.5Y-2Sn alloys with heat treatment.

4) T5 treatment is more effective in improving comprehensive tensile properties of Mg-6Zn-0.5Y-2Sn alloys than T6 treatment.

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热处理对超声挤压态 Mg-6Zn-0.5Y-2Sn 合金微观组织和拉伸性能的影响

薛寒松[1,2]，陈扬扬[1]，黄 杰[1]，李信宇[1]，饶劲松[1]，潘复生[1,2]
(1. 重庆大学 材料科学与工程学院，重庆 400044)
(2. 重庆大学 国家镁合金材料工程技术研究中心，重庆 400044)

摘 要：研究了热处理对超声挤压态 Mg-6Zn-0.5Y-2Sn 合金微观组织和拉伸性能的影响。结果表明，经过时效处理（T5）和固溶+时效处理（T6），合金的晶粒尺寸变得更加均匀，MgSnY 相和 Mg_2Sn 相分布的更加弥散，且数量有所增加。而且，T5 和 T6 处理均提高了合金的屈服强度，其中 T5 处理的提高作用十分明显。经过 T5 处理之后，合金的屈服强度从 165 MPa 增加到了 269 MPa，增长率达到 63.1%。MgSnY 相和 Mg_2Sn 相在合金基体中产生的析出强化作用，是提高热处理态合金屈服强度的主要原因。此外，T5 处理比 T6 处理更加有效地提升了合金的综合力学性能。

关键词：Mg-6Zn-0.5Y-2Sn 合金；超声处理；热处理；屈服强度；拉伸性能

作者简介：薛寒松，男，1968 年生，博士，副教授，重庆大学材料科学与工程学院，重庆 400044，电话：023-65111212，E-mail: hsxue@sohu.com