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ARTICLE

Effects of Heat Treatment on Microstructure and Mechanical Properties of As-extruded Mg-Zn-Y-Zr Alloy

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Abstract: The effects of different heat treatment parameters on the microstructure and mechanical property of as-extruded Mg-4.8Zn-1.2Y-0.4Zr magnesium alloy were described based on the experimental results and analyses. The results indicate that the microstructure of the alloy changes obviously during heat treatment process. After T4 solution treatment, although the alloy grains grow obviously, the plasticity of the alloy is enhanced greatly. This is mainly due to the massive fish-bone W-phase decomposed into fine granular W phase, which reduces the pinning effect of the grain boundary. After T5 heat treatment, besides W-phase, there is a small amount of Mg-Zn phase precipitated in the alloy, which can improve the performance of the alloy. After T6 heat treatment, the W-phase has been almost completely decomposed into Mg-Zn and Mg-Y phase. The dispersively distributed phases greatly improve the strength and plasticity, which increases the ultimate tensile strength and elongation from 311 MPa and 16.89% in as-extruded alloy to 374 MPa and 21.97%, respectively. At last the most suitable scheme for heat treatment of Mg-4.8Zn-1.2Y-0.4Zr magnesium alloy is (500 °C/2 h+200 °C/48 h).

Key words: heat treatment; as-extruded magnesium alloy; microstructure evolution; mechanical properties

With the gradual intensification of world energy, resource crisis and environmental pollution, energy saving especially in vehicles has become an important problem[1,2]. The magnesium alloy has high specific strength and it is the lightest metal among the common metals[3-8]. The magnesium has large reserves, and is cheap [9]. It can be recycled without reducing property[10,11]. But the poor mechanical properties (such as low tensile strength) greatly restrict its practical application [12, 13]. For the engineering applications of Mg alloys, improving the mechanical properties has become a critical challenge. Currently effective methods for further improvement in mechanical properties of as-cast magnesium alloys are plastic processing and heat treatment. The plastic processing (such as extrusion process) can improve the mechanical property by refining the magnesium alloy grains greatly[14,15]. The heat treatment process can change the second phase of the alloy, thus to improve the mechanical property of the alloy[16,17].

In the wrought magnesium alloys, the Mg-Zn-Zr-Y alloy has higher strength, better plasticity, heat treatment and corrosion resistance than others. There are 3 kinds of main ternary equilibrium phases in Mg-Zn-Zr-Y systems: I phase (Mg_3Zn_6Y), W phase ($Mg_3Zn_3Y_2$) and Z phase ($Mg_{12}Yzn$). I phase is octahedron quasicrystal organization, which is distributed along the grain boundary in the as-cast alloy and has adverse effects on the alloy[18,19]. But after hot working process, I phase can increase the mechanical properties of Mg-Zn-Zr-Y alloy because of its quasicrystal structure[20]. W phase is cubic crystal system, which has no positive effects on the strength of the alloy. When the content of W phase in the alloy exceeds 14% (mass fraction), the mechanical property of the alloy declines obviously. Z phase (also known as X phase) is long-period stacking ordered (LPSO) structure. Z phase has the effects of dispersion strengthening, which can greatly improve the mechanical property of the alloy[21,22]. The amount of 3 kinds of ternary equilibrium phases is mainly due to the Y/Zn ratio. In addition, there are hexagonal system H phase-$MgYZn_3$, Mg_7Zn_3, MgZn and so on[23].

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In this work, the effects of heat treatment on microstructure and mechanical properties of as-extruded Mg-Zn-Y-Zr alloy were investigated. We hope to find a suitable heat treatment process to effectively improve the mechanical property of Mg-Zn-Zr-Y alloy.

1 Experiment

The magnesium alloy used in the experiment was as-extruded Mg-4.8%Zn-1.2%Y-0.4%Zr alloy. The alloy had been extruded with the parameter of extrusion temperature 300 ºC and extrusion ratio 25. According to the heat treatment characteristics of magnesium alloy and DSC curve[24-28], the heat treatment scheme was determined.

The scheme is shown in Table 1. (1) There are two kinds of holding temperatures and three kinds of holding time in the T4 solution treatment which form two sets of scheme. One is that heat preservation time is 2, 3.5, 5 h at 500 ºC, and the other is that heat preservation time is 2, 3.5, 5 h at 470 ºC. After heat preservation, the hot-water quenching has been carried out. (2) The scheme of T5 artificial aging is that heat preservation time is 0~72 h at 200 ºC, the sample is took out every 2 h. (3) The scheme of T6 heat treatment is that the sample is heat preserved for 2 h at 500 ºC, then quenched in hot-water, and at last heat preserved for 48 h at 200 ºC.

The main equipment in the heat treatment experiments contains a resistance furnace, an aging oven and so on.

After the heat treatment was completed, the metallographic samples were cut from the heat treatment sample. The microstructure was observed by OLYPuS-PEM-3 optical microscope and Quanta 200FEG scanning electron microscope (SEM) equipped with Oxford X-ray energy dispersive spectrometer (EDS); the scanning voltage was 20 kV, the scanning electricity was 0.69 NA and the working distance exceeded 5.5 mm. The phase was determined by D/max-rb X-ray diffractometer (XRD) whose scanning speed was 4.8°/min, scanning voltage was 45 kV and scanning area was 2θ=20°~90°. The results of XRD was analyzed by MDI-jade5.0 software. The phase composition analysis was carried out by AXIOS-PW4400 X-ray fluorescence spectrometer. The tensile test specimens were cut from the heat treatment sample, and the tensile properties at room temperature were tested by electronic universal testing machine Instron 5569, and the rate of extension was 1 mm/min. The hardness test was studied at DIGITAL MICROHARDNESS TESTER HVS-1000Z, the loading force was 100 gf and the force retention time was 10 s. The test result was Vickers hardness.

Table 1 Modes and parameters of heat treatment

Heat treatment mode	Preservation temperature and preservation time
T4	500 ºC/2 h, 3.5 h, 5 h 470 ºC/2 h, 3.5 h, 5 h
T5	200 ºC/0~72 h
T6	500 ºC/2 h+200 ºC/48 h

2 Results and Discussion

SEM images of the pre-study of as-extruded Mg-4.8%Zn-1.2%Y-0.4%Zr magnesium alloy are shown in Fig.1, the grain of alloy is thin, and the precipitated phase in the alloy is mainly W phase ($Mg_3Zn_3Y_2$). The morphology of the W phase is massive fish-bone, and the W phase is distributed along the grain boundary.

2.1 T4 solution treatment

When preservation temperature is 500 ºC, once the preservation time exceeds 3 h, the sample has been burnout. It is shown that the alloy has serious overburning. So preservation time of 3.5 h and 5 h at 500 ºC is not suitable for the experiment. After T4 solution treatment, the mechanical property of T4 solution treatment alloy has obviously changed (Table 2). Firstly, compare with as-extruded alloy, the tensile strength of magnesium alloy decreases obviously. Except the alloy at 470 ºC/3.5 h, the plasticity of the rest alloy increases. Secondly, comparison on all the results of solution treatments shows that, the plasticity of alloy with 500 ºC/2 h heat treatment increases most obviously, the elongation of the alloy is nearly 50% higher than that of as-extruded alloy. The solution treatment scheme 500 ºC/2 h is the most suitable for the magnesium alloy of the experiment.

It can be observed from Fig.2 and Fig.3 that, after 500 ºC/2 h solution treatment, the grain of the magnesium alloy has grown significantly. The massive fish-bone second phase has been decomposed into fine granular phase. The

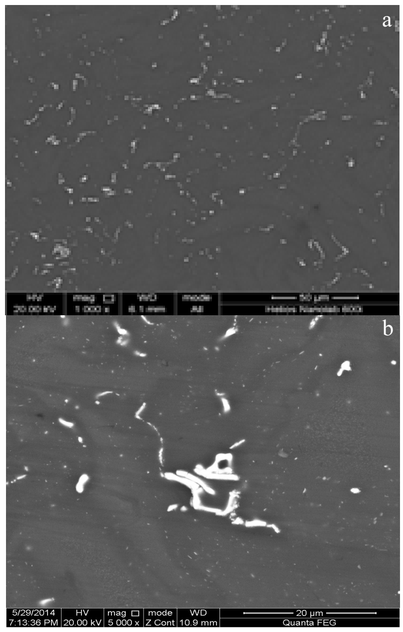

Fig.1 Microstructures of the as-extruded alloy

Table 2 Mechanical property of alloy after solution treatment

Number	Preservation temperature / °C	Preservation time/h	Elongation, δ/%	Yield strength, σ_s/MPa	Tensile strength, σ_b/MPa
1	500	2	27.21	170.13	283.78
2	470	2	18.08	186.00	280.42
3	470	3.5	14.68	178.67	279.63
4	470	5	17.91	175.67	279.23
5	As-extruded	0	16.89	234.34	311.04

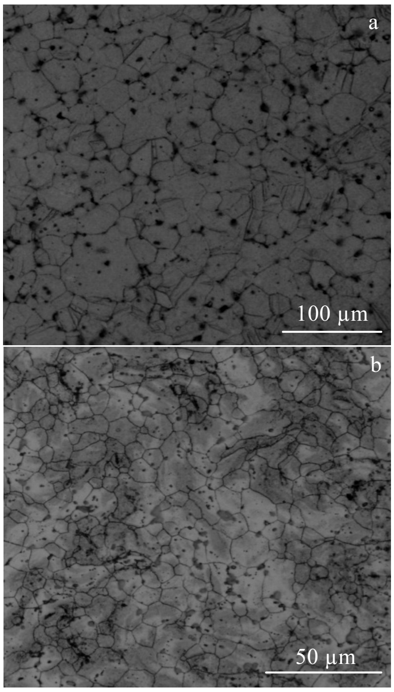

Fig.2 Microstructures of the alloy solutionized at different parameters: (a) 500 °C/2 h and (b) 470 °C/5 h

second phase is mainly distributed in grains and triangular grain boundary. The number of precipitated phase in the grain boundary decreases, and the grain boundary becomes thin. There are a spot of lamellar-type precipitated phase which is bases on grain boundary in the grain. After 470 °C/5 h solution treatment, the grain of the magnesium alloy is slightly smaller than that of 500 °C/2 h solution treated alloy, and the size of grain is uneven. The second phase distributed along crystal boundary decreases significantly. The granular decomposed second phase is distributed uniformly in the grain [19].

According to Fig.4 and Fig.5, the variation of second phase of alloy is studied. After solution treatment, the main residual phase is W ($Mg_3Zn_3Y_2$) phase (Fig.5), which is distributed along the grain boundary. After 500 °C/2 h heat treatment, the massive fish-bone W-phase has been decomposed into fine granular phase. After 470 °C/5 h,

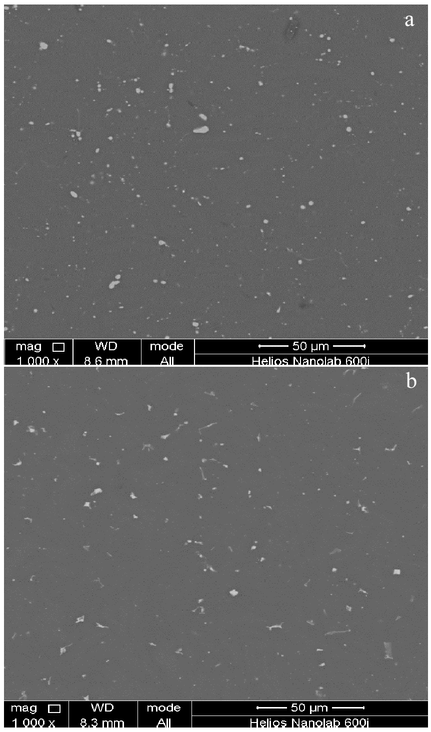

Fig.3 SEM images of the alloy solutionized at different parameters: (a) 500 °C/2 h and (b) 470 °C/5 h

there is part of massive fish-bone W-phase residual. According to phase diagram, the phase in B of Fig.4a is Mg_2Zn_3 phase, and there is some Zr segregated.

The results show that the obvious increase in elongation of magnesium alloy can be mainly attributed to the decomposition of W phase. The decomposition of the second phase reduces the pinning effect of the grain boundary. On the other hand, the fine size and distribution of granular W-phase can effectively eliminate the stress concentration which forms during the tensile testing, and prevents the microcrack initiation around the interface between the W-phase and the Mg matrix[18]. The reason of strength decline is that the solution heat treatment eliminates the strain hardening. In addition, the grain growth also decreases the strength of the alloy.

2.2 Aging treatment

The aging treatment temperature is 200 °C. The sample was taken out to measure the micro-hardness every 2 h (from 0 to 72 h). The hardness curve of alloy was studied to find the aging peak. The hardness value of alloy variation with preservation time is shown in Fig.6.

According to the age hardening curve of alloy (Fig.6), the hardness value of alloy didn't change obviously on the whole. It shows that the influence of aging treatment on the alloy is not obvious. The main reason is that there is less supersaturated solid solution in the as-extruded alloy, so the

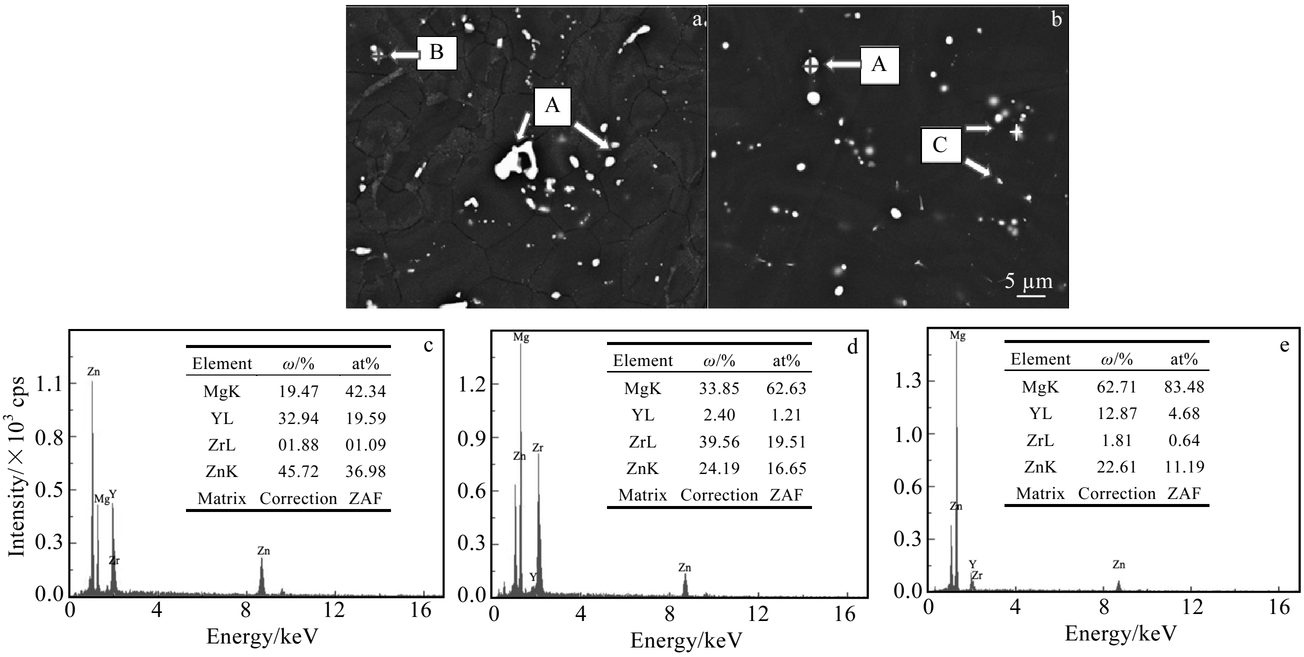

Fig.4 SEM images (a, b) and EDS results (c~e) of alloy after solid solution treatment: (a) 470 ºC/5 h; (b) 500 ºC/2 h; (c) point A in Fig.4b; (d) point B in Fig.4a; (e) point C in Fig.4b

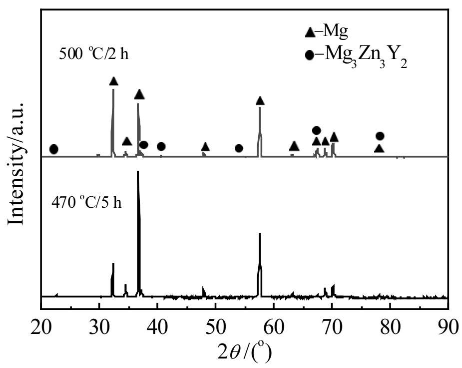

Fig.5 XRD patterns of alloy after solid solution treatment

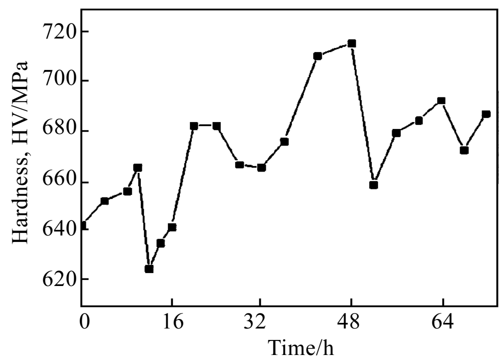

Fig.6 Hardness values of alloy with preservation time at 200 ºC

phase which precipitated during aging treatment process is also less. When the preservation temperature is 200 ºC, the max hardness is 715.9 MPa. The preservation time corresponding to the hardness peak at 200 ºC is 48 h. 200 ºC/48 h aging parameter is more suitable for the aging treatment.

Fig.7 is the microstructures of the alloy aged at 200 ºC/48 h. The grain boundary of structure becomes thin after heat preservation for 48 h at 200 ºC. The coarse second phase distributed along the grain boundary decreases, meanwhile the granular precipitated phase in the grain increases.

Fig.8 is the SEM and EDS results of the alloy after T5 treatment. After 200 ºC/48 h treatment, the number of precipitated phase declines. Besides W phase, there is some granular Mg-Zn phase. Combined with the results of XRD (as shown in Fig.9), the binary phase is $MgZn_2$ phase. By this token, in the process of aging Zn atom precipitated out from grain boundary, and formed new binary phase with Mg atom, meanwhile the massive fish-bone remained W-phase of alloy decomposed into fine granular phase.

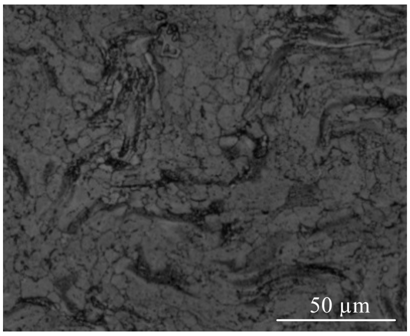

Fig.7 Microstructures of the alloy aged at 200 °C/48 h

The mechanical property of magnesium alloy with different aging treatment parameters is shown in Table 3. After comparing the results of different aging schemes, the most suitable ageing process has been confirmed, i.e. preservation temperature 200 °C and preservation time 48 h.

Conclusively, after aging treatment, the grain size has increased the least among the 3 kinds of heat treatment processes. The aging treatment effectively improves the plasticity of alloy, due to the decomposition of parts of W phase. The decomposition of the coarse fish-bone second phase reduces the pinning effect to the grain boundary. But the W phase is not completely decomposed, so the elongation is less than that of the solid solution alloy. On the other hand, the variation of W phase which is distributed along the grain boundary leads to the decrease in strength of the alloy, but after aging treatment, the granular phase is newly formed in the grain. The dispersive distribution granular phase in the grain can enhance the strength of the alloy. On the whole, the aging treatment can only finitely improve the strength of the alloy.

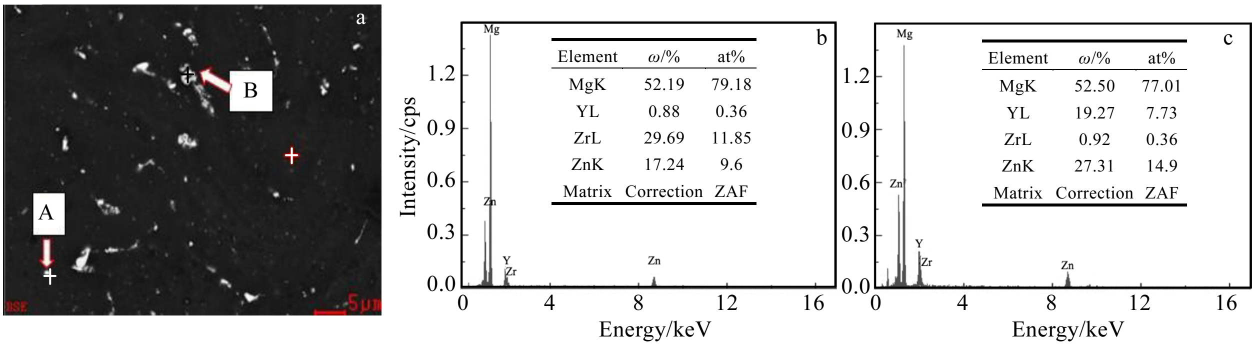

Fig.8 SEM image (a) and EDS results (b, c) of alloy aged at 200 °C/48 h: (b) point A in Fig.8a and (c) point B in Fig.8a

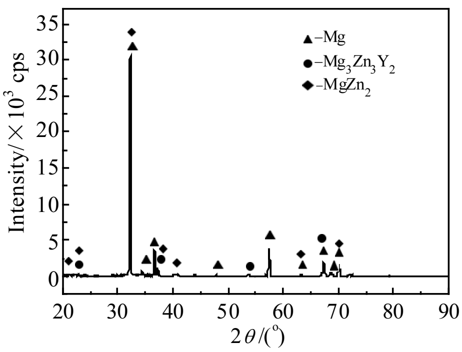

Fig.9 XRD pattern of alloy aged at 200 °C/48 h

Table 3 Mechanical property of alloy after artificial aging

Serial number	Solution temperature/°C	Solution time/h	Elongation, δ/%	Yield strength, σ_s/MPa	Tensile strength, σ_b/MPa
1	200	12	18.53	236.68	309.93
2	200	24	24.15	251.65	319.85
3	200	48	25.36	256.89	325.64

2.3 T6 heat treatment

Fig.10 is the microstructure of the alloy after T6 treatment. The black granular precipitated phase can be observed obviously. The major granule is distributed along the grain boundary.

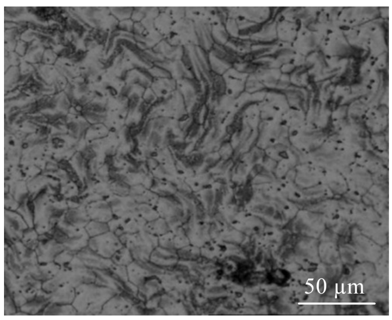

Fig.10 Microstructure of alloy after T6 treatment

Fig.11 is the EDS results of alloy after T6 treatment. After T6 heat treatment, the massive fish-bone W-phase of as-extruded alloy was decomposed into fine granular phase. The larger phase with higher brightness is basically distributed along the grain boundary, and the granulum with lower brightness is dispersively distributed within the grain. The results of EDS show that there are elements Mg, Zn, Zr and Y at point A. The solid solubility of Zr in Mg is very low, so it can be determined that the Zr element in point A is formed by segregation. It is Mg-Zn binary phase at point B. The point C is the granulum with lower brightness. Due to the test precision of the instrument, the composition of C is affected by Mg substrate, and the practical composition is Mg-Y binary phase. Combined with the results of XRD (Fig.12) it is found that the phase of A and C is Mg_2Y and the phase of B is $MgZn_2$.

After T6 heat treatment, the tensile strength and elongation of magnesium alloy have been improved obviously. At last the tensile strength and elongation of magnesium alloy are 374.1 MPa and 21.97%, respectively. The increase in mechanical property is mainly due to the variation of the precipitates. It is known that the shape, size and distribution of the precipitates have greatly affected the strength of the magnesium alloys. On the other hand, the precipitates can

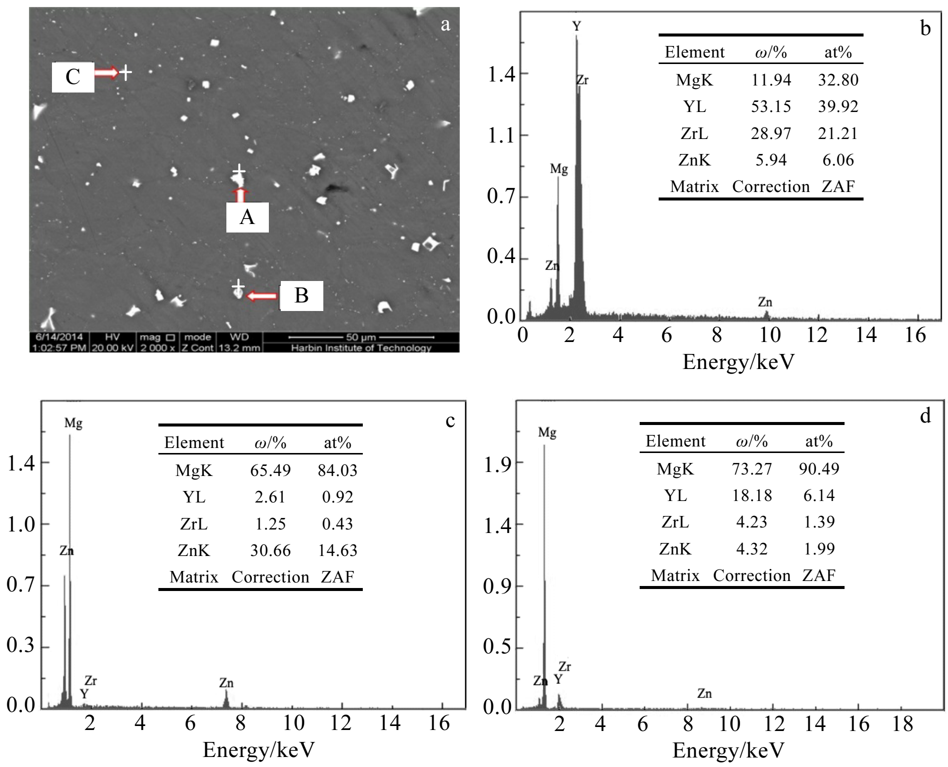

Fig.11 SEM image (a) and EDS results (b~d) of alloy after T6 treatment: (b) point A in Fig.11a; (c) point B in Fig.11a; (d) point c in Fig.11a

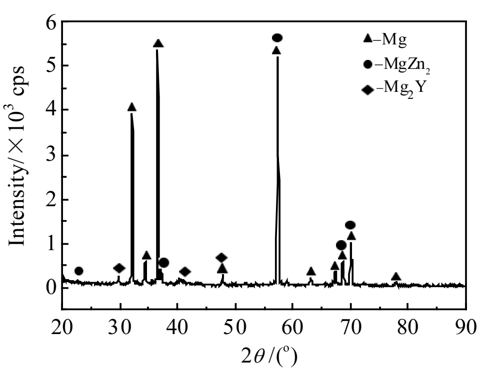

Fig.12 XRD pattern of alloy after T6 treatment

block the movement of dislocations, twins or slip, and then decrease the elongation of alloy. After the T6 heat treatment, the massive fish-bone W-phase of as-extruded alloy has been decomposed into fine granular Mg_2Y or $MgZn_2$ phase. The decomposition of massive phase weakens the pinning effect of the grain boundary, which greatly improves the plasticity of the alloy. But with the decomposition of W phase at the grain boundary, parts of elements Zn and Y dissolve into the grain and form dispersed granular precipitated phase. These dispersed granular precipitated phase block the movement of dislocations, and then reduce the elongation of the alloy. So the elongation of T6 alloy is lower than that of the other two alloys. Simultaneously, for the mechanical property of alloy, the reduction of W phase can improve the strength of alloy and the dispersed granular precipitated phases also greatly increase the mechanical property. On the whole, these conditions lead to the high strength and decent plasticity of the T6 heat treatment alloy.

Table 4 is the mechanical property comparison of alloys under different heat treatments. After heat treatment process, the mechanical property of Mg-4.8Zn-1.2Y- 0.4Zr magnesium alloy changes regularly. Compared with as-extruded alloy, after T4 solution treatment, the strength dramatically declines, but the plasticity improves most obviously and the elongation reaches the maximum. After T5 artificial aging, both the strength and plasticity have been enhanced, but the increment in the strength is smaller than that of the alloy after T6 heat treatment. After T6 heat treatment, both the strength and plasticity have been greatly improved, and the strength has reached the maximum. The elongation of T6 alloy is slightly lower than that of T4 alloy, but conclusively, the mechanical performance of T6 alloy is the most excellent on the whole. Compared with as-extruded alloy, the tensile strength and elongation of T6 treatment alloy increased by 60 MPa and 5%, with the amplification of 20% and 30%, respectively. At last after comparing the comprehensive performance of alloys with different heat treatments, the most suitable scheme for Mg-4.8Zn-1.2Y-0.4Zr magnesium alloy is (500 ºC/2 h+200 ºC/ 48 h).

Table 4 Mechanical property of alloys under different heat treatments

Heat treatment	Elongation, δ/%	Yield strength, σ_s/MPa	Tensile strength, σ_b/MPa
T4	27.21	170.13	283.78
T5	25.36	256.89	325.64
T6	21.97	283.37	374.10
As-extruded	16.89	234.34	311.04

3 Conclusions

1) After 500 ºC/2 h solution treatment, the alloy grain has grown obviously, and the massive fish-bone W-phase has been decomposed into fine granular W phase, which is beneficial to plasticity of alloy. But there are limited decomposed atomics formed solid solution distributed in the magnesium substrate. The effect of solid solution strengthening is not obvious.

2) After T5 artificial aging, a part of W phase has been decomposed. Besides W-phase, there is a small amount of Mg-Zn phase precipitated in the alloy; it can improve the performance of the alloy. After comparing different aging schemes, the most suitable ageing process is 200 ºC/48 h.

3) After T6 heat treatment, the W-phase has been almost completely decomposed into Mg-Zn and Mg-Y phase. There are vast dispersively distributed phase formed in the grain. The dispersively distributed phase greatly improves the strength and plasticity.

4) At last after comparing the comprehensive performance of the alloys with different heat treatment, the most suitable scheme for heat treatment of Mg-4.8Zn-1.2Y-0.4Zr magnesium alloy is (500 ºC/2 h+200 ºC/48 h).

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热处理工艺对挤压态 Mg-Zn-Y-Zr 镁合金组织性能的影响

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摘　要：通过对实验结果进行分析，来探究热处理工艺对挤压态 Mg-4.8Zn-1.2Y-0.4Zr 镁合金的组织性能的影响规律。研究表明在热处理过程中，挤压态镁合金中的组织发生了明显的变化，T4 固溶处理后，虽然合金的晶粒明显长大，但是合金的塑性仍然有显著提升，这主要是因为 W 相由粗大的鱼骨状分解为细小的颗粒状降低了晶界的阻塞作用；T5 热处理后合金中除 W 相外，还有少量的 Mg-Zn 相析出，提高了合金的性能。T6 热处理后合金中 W 相几乎全部分解，析出了 Mg-Zn 和 Mg-Y 等新相，这些弥散分布的相大大提高了合金的强度和塑性。T6 热处理后合金的极限抗拉强度和延伸率分别从挤压态的 311 MPa、16.89%提高到 374 MPa、21.97%。最终得到最适合 Mg-4.8Zn-1.2Y-0.4Zr 合金的热处理方式（500 ℃/2 h+200 ℃/48 h）。

关键词：热处理；挤压镁合金；组织演变；力学性能

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