Effects of heat treatment on microstructure evolution and mechanical properties of Mg-6Zn-1.4Y-0.6Zr alloy

Jing Wang, Xiao-gang Fang, *Shu-sen Wu, Shu-lin Lü

State Key Lab of Materials Processing and Die & Mould Technology, Huazhong University of Science and Technology, Wuhan 430074, China

Abstract: To investigate the effects of solution temperature and the decomposition of I-phase on the microstructure, phase composition and mechanical properties of as-cast Mg-6Zn-1.4Y-0.6Zr alloy, solution treatment at 440 ºC, 460 ºC and 480 ºC and further aging treatment were conducted on the alloy. The results indicate that the net-like intermetallic compounds (mainly I-phase) dissolve into the α-Mg matrix gradually with the increase of solution temperature from 440 ºC to 480 ºC. Besides, the I-phase decomposes completely at 480 ºC, with the formation of fine W-phase (thermal stable phase) and Mg_7Zn_3 phase. In addition, a great number of fine and dispersive Mg-Zn binary phases precipitate in the α-Mg matrix during the aging treatment. Due to the increase of solute atoms and the precipitation of strengthening phases, such as W-phase and Mg-Zn phases, the optimal strength is obtained after solution treatment at 460 ºC for 8 h and aged at 200 ºC for 16 h. The yield strength (YS), ultimate tensile strength (UTS) and elongation are 208 MPa, 257 MPa and 3.8%, respectively. Compared with the as-cast alloy, the increments of YS and UTS are 117% and 58%, respectively, while the decrement of elongation is 46%.

Key words: magnesium alloy; heat treatment; microstructure; mechanical properties

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Magnesium alloys have great potential in electric, aerospace and transport industries due to their low density, high specific strength and stiffness, excellent damping property and good castability [1, 2]. Commercial ZK series magnesium alloys exhibit high strength and good ductility because of the significant refinement in microstructure achieved by Zr addition [3]. However, the inferior elevated temperature strength and poor creep resistance restrict their further applications [4]. Related studies have demonstrated that the addition of rare earth (RE) elements such as yttrium to Mg-Zn-Zr alloy system can obtain outstanding mechanical properties, especially superior creep resistance at both ambient and elevated temperatures [5-7].

The Mg-Zn-Y-Zr alloys exhibit remarkable properties as the result of the unique second phases, such as I-phase (Mg_3YZn_6), W-phase ($Mg_3Y_2Zn_3$) and X-phase ($Mg_{12}YZn$) [8-10]. The I-phase with a unique icosahedral quasicrystal structure has high strength and hardness, low interfacial energy and good thermal stability, is an effective strengthening phase to enhance the mechanical properties [6, 11, 12]. The W-phase has superior thermal stability at elevated temperature, which always distributes along the grain boundary with a continuous network in casting Mg alloys. Generally, only fine and dispersive W-phase can improve the mechanical properties to a certain extent [9].

It has been concluded that valued methods for further improvement for strength of cast Mg-Zn-Y-Zr alloys are thermal deformation (hot extrusion and hot rolling), heat treatment (annealing, solid solution and aging) and the combination of both [13]. Generally, thermal-mechanical processes are relatively complicated and expensive, though these processes bring Mg-Zn-Y-Zr alloys excellent properties. In addition, many castings cannot be thermal-mechanically processed further. Compared with thermal deformation, heat treatment can obtain superior mechanical properties with simple processes and low cost. Hence, precipitation hardening is an attractive way to improve the strength of Mg-Zn-Y-Zr alloys [14-17].

Zn and Y elements exhibit relatively high solubility at elevated temperature and limited solubility at low

*** Shu-sen Wu**

Male, born in 1961, Professor, Ph. D. supervisor. His research interests focuses mainly on the solidification processing of nonferrous alloys through ultrasonic vibration and squeeze casting.

E-mail: ssw636@hust.edu.cn

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temperature. Therefore, they have strong effects on solution and aging strengthening. Ma et al. [18] revealed that only original α-Mg and I-phase exist in the Mg-3Zn-0.6Y-0.6Zr alloy after solution treatment at 425 ºC for 8 h, and the excess Zn atoms enter into the α-Mg matrix entirely. Further aging treatment precipitates a large amount of nanoscale $MgZn_2$ phase, which could enhance the tensile strength greatly. Besides, Geng et al. [19] found that the transformations of I-phase to W- or H-phase occur at 420 ºC in $Mg_{63.5}Zn_{34}Y_{2.5}$ alloy, as a result of slow transformation kinetics and low thermodynamics driving force. Moreover, Feng et al. [15] reported that the precipitation of fine W, β_1' and β_2' phases during aging treatment can improve the mechanical properties of as-cast Mg-3Zn-0.9Y-0.6Nd-0.6Zr alloy significantly.

At present, most studies about heat treatment of Mg-Zn-Y-Zr alloys focus on the reservation of I-phase [16, 18], and the corresponding solution temperatures are near the transformation temperature of I-phase. However, the effects of the decomposition of I-phase at higher solution temperature on the microstructure, phase constituent and mechanical properties are rarely studied. Meanwhile, the majority of investigations pays much attention to the soaking time [20], with few studies about the variations in holding temperature. Therefore, this work aims to study the above problems.

In the present study, solution treatment at 440 ºC, 460 ºC and 480 ºC for 8 h were conducted on the as-cast Mg-6Zn-1.4Y-0.6Zr alloy, and then aging treated under the same conditions. The effects of solution temperature and the decomposition of I-phase on the microstructure, phase constituent and mechanical properties were studied. The relationships between different precipitates and mechanical properties were also discussed.

1 Experimental procedures

An alloy with nominal composition Mg-6Zn-1.4Y-0.6Zr (wt.%) was prepared in an electric resistance furnace in a mild steel crucible at 780 ºC under mixed atmosphere of SF_6 and N_2 with the volume ratio of 1:99. The raw materials were pure Mg and Zn, Mg-30wt.% Y and Mg-30wt.% Zr master alloys. After all the materials were melted, the melt was stirred for homogenization, and then refined for 10 min with Ar gas bubbling and held for 30 min at 700 ºC. The alloy ingots (30 mm in diameter, 100 mm in length) were cast in a permanent mold preheated to 200 ºC and cooled in air. The samples for heat treatment were cut from the ingots by electric spark linear cutting machine with a diameter of 9 mm. The specimens were solution treated under atmosphere of Ar at 440 ºC, 460 ºC and 480 ºC for 8 h followed by quenching in hot water. Afterwards, isothermal aging treatment at 200 ºC for 16 h was conducted on the solution treated samples.

Microstructures of the specimens were characterized by an optical microscope (OM, Axiovert 200MAT) and a scanning electron microscope (SEM, Nova NanoSEM 450) equipped with an energy dispersive spectrometer (EDS). Specimens for OM and SEM observations were mechanically polished and then etched in a solution of 4 vol.% HNO_3 and 96 vol.% ethanol. The phase analysis of samples were determined by the X-ray diffraction (XRD, X' Pert PRO X) using Cu Kα radiation with a scanning angle from 20° to 90°, and a scanning rate of $10°\cdot min^{-1}$. Cylindrical specimens with gauge diameter of 5 mm and gauge length of 30 mm were prepared for tensile tests. The room temperature tensile tests were performed on a SHIMASZU AG-IC100KN universal testing machine to assess the ultimate tensile strength (UTS), yield strength (YS) and elongation to failure.

2 Results and discussion

2.1 Microstructure of as-cast Mg-6Zn-1.4Y-0.6Zr alloy

Figure 1(a) shows the microstructure of the as-cast Mg-6Zn-1.4Y-0.6Zr alloy cast at 625 ºC, which is under the liquidus temperature (about 630 ºC). Similar to rheo-squeeze casting Mg-RE-Zn-Y alloy in reference [21], the microstructure is composed of two different sized grains, which are defined as α_1-Mg (the larger grains, rose-like dendrites) and α_2-Mg (the smaller grains, typical equiaxed dendrites) respectively. The α_1-Mg grains form primarily during the cooling process from 700 ºC to 625 ºC in the holding furnace preheated to 625 ºC, while the α_2-Mg grains nucleate and grow in the mold cavity after pouring. Besides, the net-like intermetallic compounds distributed continuously along the grain boundaries of α-Mg.

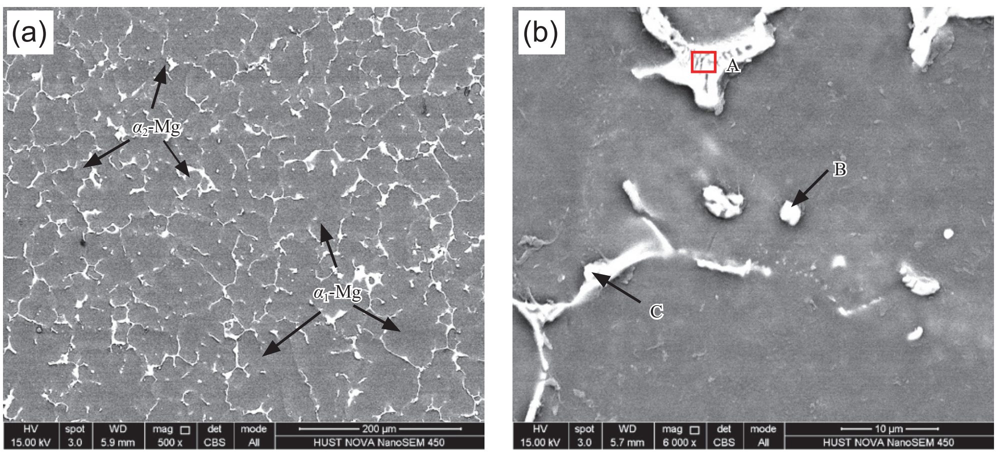

Fig. 1: Microstructures of as-cast Mg-6Zn-1.4Y-0.6Zr alloy: (a) low magnification; (b) high magnification

Figure 1(b) shows the intermetallic compounds in high magnification of Fig. 1(a). The lamellar eutectic structures and strip-like phases can be clearly observed around position A and C, respectively. Moreover, some granular phases also can be found, such as position B. The corresponding EDS results are listed in Table 1. The energy spectrum of positions A, B and C reveals that the compositions of the intermetallic compounds contain the elements of Mg, Zn, and Y. It indicates that the majority of Zn and Y atoms will concentrate around the grain boundary and react with Mg atoms to form Mg-Zn-Y ternary phases, except for a small amount of Zn atoms that dissolve into the α-Mg matrix. The Zn/Y atom ratio of positions A and C are both approximate to 6.0, thus the lamellar eutectic structures and strip-like phases can be identified as the I-phase (Mg_3YZn_6). These are consistent with the results of XRD in Fig. 3(a), i.e. the as-cast Mg-6Zn-1.4Y-0.6Zr alloy mainly consists of α-Mg matrix, α-Zr, I-phase and W-phase. The diffraction peaks of W-phase are fewer and lower because of its rare amount. In addition, position B was detected to be yttrium-rich phase (65.32at.% Mg, 2.61at.% Zn, 32.08at.% Y). Alok Singh et al. [16] reported that the elemental yttrium phase could be detected (~97at.% Y) in the solution treated Mg-Zn-Y alloy.

Table 1: EDS results of positions A to F in Fig. 1(b) and Fig. 4(b)

Positions	Element (at.%)				
	Mg	Zn	Y	Zr	Zn/Y ratio
A	70.67	24.96	4.37	-	5.71
B	65.32	2.61	32.08	-	0.08
C	68.48	26.41	5.11	-	5.17
D	66.98	33.02	-	-	-
E	36.82	43.65	-	19.53	-
F	97.64	2.36	-	-	-

2.2 Microstructure evolution of solution-treated alloys

Figure 2 shows the microstructure evolution of the solution-treated Mg-6Zn-1.4Y-0.6Zr alloy with the increase of solution temperature from 440 ºC to 480 ºC for 8 h. Metallographic observation reveals that the majority of the original net-like intermetallic compounds have been dissolved into the matrix at 440 ºC. The residuals distributed intermittently along the grain boundaries in Fig. 2(b) are supposed to be the un-dissolved I-phase and the thermal stable W-phase. This can be confirmed from the XRD analysis in Fig. 3(b). Meanwhile, a small amount of particles are formed in the coarsened grains. After solution treatment at 460 ºC for 8 h, in Fig. 2(c), the smaller grains and numerous fine and dispersive particles can be clearly observed. Moreover, the massive lamellar eutectic structures and stripe-like phases (i.e. I-phase) virtually disappeared. When the solution temperature rises to 480 ºC, the grains in certain regions become coarse due to the grain mergence [Fig. 2(d)]. Besides, the heterogeneous distribution of refined and concentrated particles can be seen clearly.

The XRD patterns reveal the variation of phase constituent of the solution-treated samples with different solution temperatures, as shown in Fig. 3. It can be concluded that the solution temperature has remarkable impact on the phase constituent of Mg-6Zn-1.4Y-0.6Zr alloys. With the increase of solution temperature, the diffraction peaks of W-phase (the melting point is about 510 ºC [22]) always exist, while the peaks of I-phase decrease gradually. For the alloy solution treated at 480 ºC for 8 h, no peaks corresponding to I-phase can be detected within the sensitivity limit of XRD, which means that I-phase may have decomposed into the matrix completely. Geng et al. [19] reported that I-phase is thermodynamically unstable at 420 ºC. Therefore, I-phase would decompose into elements of Mg, Zn and Y in the process of solution treatment above 420 ºC. Meanwhile, the diffusion of the Y atom is much slower than that of Zn and Mg atoms due to its larger atomic radius. Thus, plenty of Y atoms can promote the transformations of I-phase to W-phase in consideration of slow transformation kinetics and low thermodynamic driving force. In addition, the peaks of the Mg_7Zn_3 phase appear after solution treatment. The formation of W-phase only takes away some of the Zn atoms, the excess Zn atoms will precipitate again in the form of Mg_7Zn_3 phase in the matrix.

2.3 Microstructure and mechanical properties of aged alloys

Figure 4 gives the SEM images of the alloy after solution at 460 ºC and isothermal aging treatment (aged at 200 ºC for 16 h). It can be seen that the microstructure of aged alloy in Fig. 4(a) changes little, compared with the corresponding solution treated alloy in Fig. 2(c). Figure 4(b), high magnification of a region in Fig. 4(a), shows that many granular and rod-like phases precipitate at grain boundaries and inside grains. Meanwhile, a great deal of fine and dispersive particles can be observed clearly in the matrix, as shown in Fig. 4(c). With the result of EDS analyses shown in Table 1, the compositions of interior grain (region F) are mainly element Mg and a small amount of element Zn. According to relative investigations [13-15, 22], these dispersive particles ought to be nanoscale Mg-Zn binary precipitates during low temperature aging treatment,

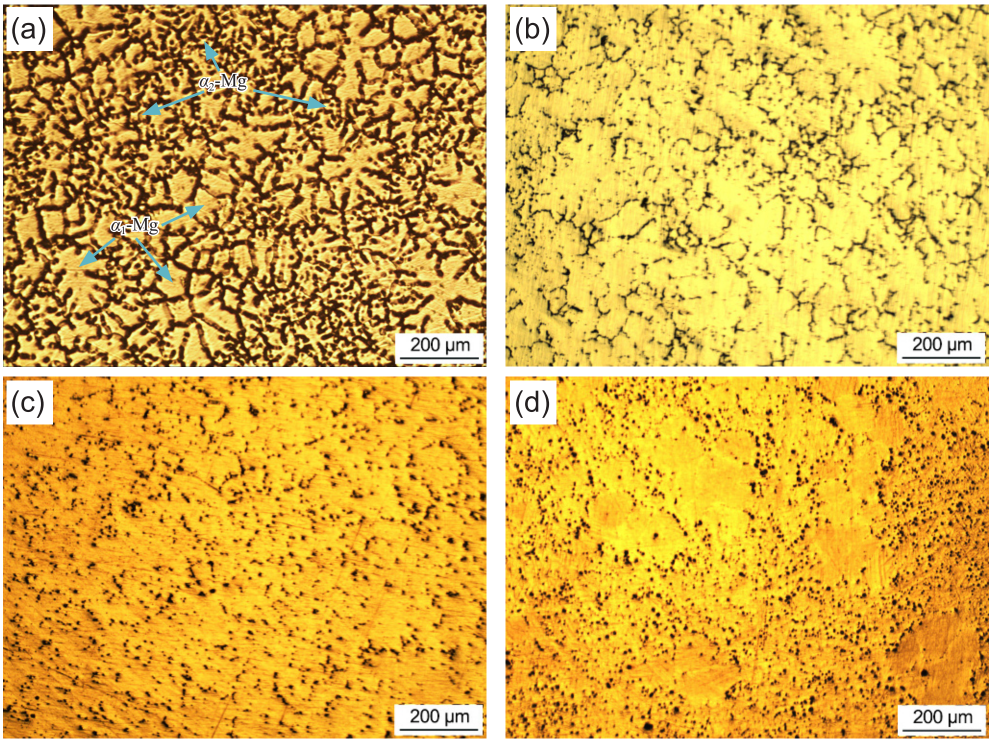

Fig. 2: Optical images of Mg-6Zn-1.4Y-0.6Zr alloy under different solution treatment conditions: (a) as-cast; (b) 440 ºC, 8 h; (c) 460 ºC, 8 h; (d) 480 ºC, 8 h

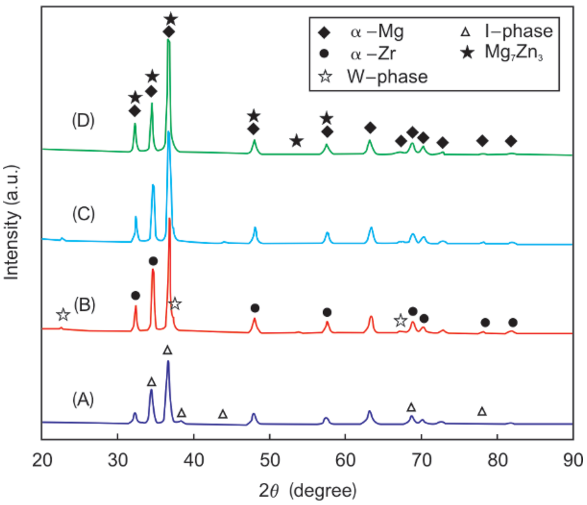

Fig. 3: XRD patterns for different solution treated samples: (a) as-cast; (b) 440 ºC, 8 h; (c) 460 ºC, 8 h; (d) 480 ºC, 8 h

such as $MgZn_2$, Mg_2Zn_{11}, MgZn′ (β_1′) and β_2′ phases. However, further study is required concerning the precipitates in the aged Mg-6Zn-1.4Y-0.6Zr alloys. The atom ratio of Mg and Y in the granular phases (position D) is close to 7/3, suggesting that these granular phases may probably be Mg_7Zn_3 phase. In addition, position E was detected to be the Zr-containing phase (36.82at.% Mg, 43.65at.% Zn, 19.63at.% Zr).

The variations of tensile properties of aged samples with different solution treated temperatures are shown in Fig. 5. The results indicate that the yield strength (YS) and ultimate tensile strength (UTS) of the alloy initially increases and then decreases with an increase in solution temperature. After solution treatment at 460 ºC for 8 h and aging at 200 ºC for 16 h, the alloy exhibits the highest YS (208 MPa) and UTS (257 MPa). Compared with the corresponding values of the as-cast Mg-6Zn-1.4Y-0.6Zr alloy, the increments of YS and UTS are 117% and 58%, respectively.

For the as-cast Mg-6Zn-1.4Y-0.6Zr alloy, the bulky net-

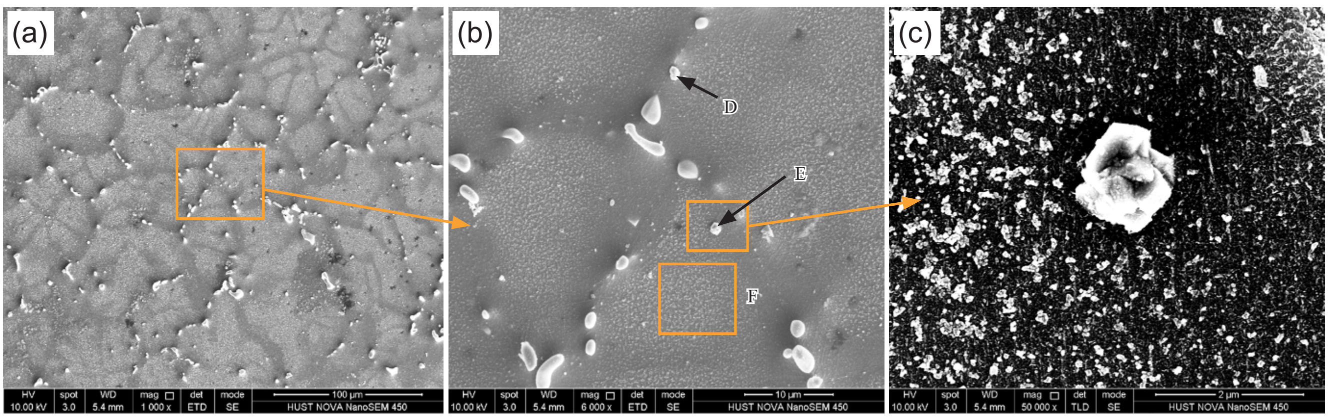

Fig. 4: SEM images of aged alloy after solution treatment at 460 ºC

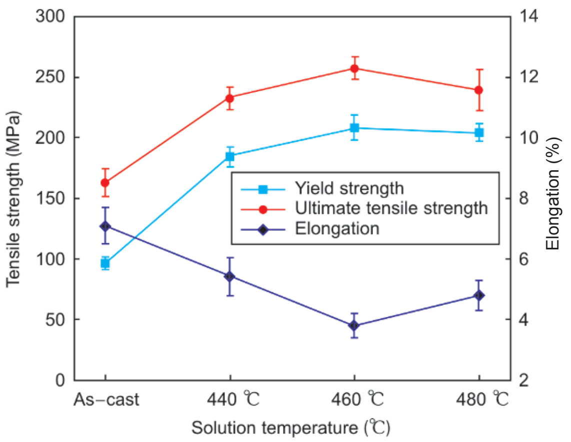

Fig. 5: Tensile properties of aged samples with different solution temperatures

like intermetallic compounds (mainly I-phase) distributed continuously along the grain boundaries. Hence, the alloy shows low YS (96 MPa) and UTS (163 MPa), and higher elongation (7.1%), although the I-phase is an effective strengthening phase for the Mg-Zn-Y-Zr alloy. From the results above, the improvement of the tensile strength in the heat-treated Mg-6Zn-1.4Y-0.6Zr alloy can be mainly attributed to the solution strengthening and precipitation strengthening.

After solution treatment, the majority of intermetallic compounds dissolve into the α-Mg matrix. Solute atoms increase with the increase of solution temperature, which results in effective solution strengthening. Meanwhile, the I-phase decomposed gradually with the formation of thermally stable W-phase. Generally, W-phase is not considered as an effective strengthening phase because of its limited structural symmetry and weak adhesive force with α-Mg matrix [14]. However, related studies [5, 9] indicated that the fine size and diffusive distribution of W-phase particles can benefit the mechanical properties in the process of extrusion. In this study, fine W-phase particles distribute along the grain boundaries after heat treatment. These particles can act as strong pinning points for grain boundaries and dislocations, and blocking the movement of dislocations to strengthen the Mg-6Zn-1.4Y-0.6Zr alloy. Moreover, a great number of nanoscale Mg-Zn binary particles precipitate in the matrix during the subsequent aging treatment, which brings strong precipitation strengthening. Combined effects of the above factors enhance the tensile strength of the heat-treated Mg-6Zn-1.4Y-0.6Zr alloys significantly. For the alloy solution treated at 480 ºC, coarse grains are the main reason for the slight decrease of the mechanical properties.

For all the heat-treated alloys, obvious reduction in elongation can be observed as shown in Fig. 5, which is lower than that of the as-cast alloy. It illustrates that the elongation shows a contrary tendency with the YS and UTS. The results are mainly determined by the precipitates' blocking to the movement of slip and dislocations.

3 Conclusions

The effects of solution and aging treatment on the microstructure evolution and mechanical properties of the Mg-6Zn-1.4Y-0.6Zr alloy have been investigated. The results are summarized as follows:

(1) With the increase of solution temperature from 440 ºC to 480 ºC, the net-like intermetallic compounds (mainly I-phase) dissolve into the α-Mg matrix gradually. The I-phase decomposes completely at 480 ºC, with the formation of fine W-phase (thermal stable phase) and Mg_7Zn_3 phase. In addition, a great number of fine and dispersive Mg-Zn binary phases precipitate in the α-Mg matrix during the aging treatment.

(2) Due to the increase of solute atoms and the precipitation of fine and dispersive strengthening phases, such as W-phase and Mg-Zn phases, the optimal strength is obtained after being solution treated at 460 ºC for 8 h and aged at 200 ºC for 16 h. The yield strength (YS), ultimate tensile strength (UTS) and elongation are 208 MPa, 257 MPa and 3.8%, respectively. Compared with the as-cast alloy, the increments of YS and UTS are 117% and 58% respectively, while the decrement of elongation is 46%.

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