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ARTICLE

Effects of Heat Treatment on Microstructure and Mechanical Properties of as-Extruded Mg-9Sn-1.5Y-0.4Zr Magnesium Alloy

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Abstract: The effects of heat treatment on microstructure and mechanical properties of the as-extruded Mg-9Sn-1.5Y-0.4Zr (wt%) alloy were investigated experimentally. The results show that the heat treatment plays an important role on the microstructure and mechanical properties of the as-extruded Mg-9Sn-1.5Y-0.4Zr alloy. The as-extruded alloy is mainly composed of non-uniform Mg_2Sn phase. After the solution treatment at 495 ℃ for 10 h, a majority of Mg_2Sn phases in the alloy dissolve into the matrix. Aging treatment can highly improve the mechanical properties of the Mg-9Sn-1.5Y-0.4Zr alloy. The optimized experimental condition is aging at 250 ℃ for 60 h after solution treatment at 495 ℃ for 10 h. The corresponding mechanical property parameters are: hardness HV of 890 MPa, ultimate tensile strength of 262 MPa, yield strength of 218 MPa, and elongation of 10.4%. Based on the experimental analysis, we find that the precipitation strengthening is the main contributor (~51.76%) to the total yield strength in the aged experimental alloy.

Key words: Mg-9Sn-1.5Y-0.4Zr alloy; heat treatment; microstructure; mechanical property

Magnesium alloys have a great potential for high performance structural applications due to their excellent properties such as low density, high specific strength and superior damping capacity. Therefore, the applications of magnesium alloys are steadily increased in the fields of 3C, automobile and aeronautic industry, etc. Today, the research on the development of lightweight materials for various structural applications in transportation vehicles is driven by the lightweight construction trend to improve energy efficiency [1-3]. However, the lower strength and corrosion resistance of magnesium alloys greatly limit their wide applications such as for key bearing components.

In recent decades, a series of high performance magnesium alloys have been explored[4]. The commercial magnesium alloys with higher strength and heat resistance for industry are mainly based on Mg-RE system. The addition of RE elements refines grains in magnesium alloys and can also improve the mechanical properties both at room and elevated temperature[5-7]. Up to date, the Mg-Mn-RE alloys with excellent corrosion resistance and the Mg-Zn-Zr-RE alloys with well weld ability had been investigated comprehensively. However, the study about Mg-Sn-RE alloys is still not comprehensive yet. From the Mg-Sn phase diagram, it is found that the solubility of Sn in α-Mg solid solution drops sharply from 14.85wt% at the eutectic transformation temperature of 561 ℃ to 0.45wt% at 200 ℃. This provides an important basis for improving the mechanical properties of Mg-Sn alloys through aging [8]. The Mg_2Sn phase with a higher melting temperature at about 770.5 ℃ can be obtained due to the addition of Sn in Mg alloys. As a normal valence compound with high hardness HV of 1190 MPa, it can provide barriers for dislocation slipping and climbing during tensile tests, and thus the mechanical properties both at room and elevated temperature can be improved[9].

In addition, both Y and Zr are the efficient surface active elements for grain refinement. Due to the Y aggregation at the

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solid-liquid interface during solidification, the significant solute redistribution and segregation lead to a constitutional super cooling region in front of the dendrite growth solid-liquid interface. Therefore, the nucleation rate is higher than before which decrease both the grain size and dendrite arm spacing of the experimental alloys. It can reduce the solid-liquid interfacial tension and nucleation energy, the nucleation getting easier for the critical nucleation radius smaller than ever so the mechanical properties of the experimental alloys can be enhanced and also convenient to the subsequent extrusion process. The deformed magnesium alloys always have superior mechanical properties to cast due to refinement of grains, homogenization of microstructure and decrease of casting defects after the plastic processing[10]. However the research on the Mg-Sn-RE system alloys is mainly limited to the as-cast alloys currently [11]. Fang [12] reported that the addition of Y element could improve both the elevate temperature properties and corrosion resistance remarkably. The age hardening characteristics could be also strengthened through the addition of Y element [13].

In the present paper, the effects of heat treatment on the microstructure and mechanical properties of the as-extruded Mg-9Sn-1.5Y-0.4Zr alloy were studied. Based on the study, we expect to provide some basic information for the design of an effective heat treatment technique.

1 Experiment

The alloys were prepared by a conventional casting method with pure Mg and Sn (>99.9wt%), Mg-25wt%Y and Mg-30wt%Zr master alloys as starting materials. To guarantee the purity of the raw materials, the alloys were melted in a graphite crucible under the protective mixed gas atmosphere of CO_2 and 0.2%SF_6 (volume fraction). The refining agent was 55%KCl + 2%CaF_2 + 15%$BaCl_2$ and the burning loss rate was considered in the preparing procedure. The melt was kept being stirred to ensure homogeneity and held at ~750 ℃ for about 30 min. Then, the melt was cast into a preheated (250 ℃) iron mould in order to obtain the as-cast materials. The cavity dimension of the mould was measured as 200 mm ×80 mm ×80 mm. The as-cast ingots were homogenized at 400 ℃ for 10 h and then extruded into rods 20 mm with in diameter at 300 ℃, at the speed of 5 mm/s with the extrusion ratio of 14:1. The type of the specimen for the tensile test was R9 according to GB/T16865-2013. The solid solution treatments of the specimens were performed at 405, 450, 495, 540 ℃ for 10 h. In order to preserve the solid solution treated microstructure, the solid solution treated specimens were quenched into hot water at ~90 ℃. Artificial aging treatments were carried out at 200, 250, and 300 ℃ for 0~120 h in a resistance furnace. Every 12 h, a set of specimens was drawn from the resistance furnace and followed by atmosphere cooling.

The metallographic samples were cut into cylinders (Φ=20 mm, L=15 mm) with an electrical discharge wire from the same position of each specimen. In order to reveal the grain boundaries, the specimens were polished and etched with the reagent of 1.5 g picric acid + 25 mL ethanol + 5 mL acetic acid and 10 mL distilled water. The overall phase structures of the specimens were analyzed by X-ray diffraction (D/Max-2550) with Cu Kα radiation. The microstructures and the morphologies of the specimens were analyzed by optical microscope (OM, MR5000) and scanning electron microscope (SEM, HITACHI S4800) equipped with an energy dispersive spectrometer (EDS). The tensile test was conducted at room temperature by an electronic mechanical universal testing machine (SANS-CMT5105) with a crosshead speed of 0.2 mm/min. 6 specimens of each alloy were employed to obtain a set of mechanical testing data. The hardness test was monitored by Vickers hardness tester (HXD-1000 TM) with a load of 100 g and the holding time was 15 s. The average grain size was evaluated using a linear intercept method.

2 Results and Discussion

2.1 Microstructures

The optical microstructures and SEM images of various Mg-9Sn-1.5Y-0.4Zr alloys are shown in Fig.1 and Fig.2 respectively. Fig.1a and Fig.2a show the OM and SEM images of the as-extruded alloys, respectively. It is clearly found that some particles (white in SEM, black in OM) distribute along the extrusion direction. The EDS analysis (Fig.3) reveals that the white particle (marked as A) is the Mg_2Sn phase, which corresponds to the XRD results (Fig.4a).

Compared Fig.1a~1e it can be found that after solid solution treatment, most of the compounds redistribute homogeneously in the α-Mg matrix. Any significant change is hardly found after solution treated at 405 ℃ for 10 h (compared Fig.1a and 1b) due to the experimental alloys homogenized at 400 ℃ before extruded at 300 ℃. Fig.1e shows the OM image of the experimental alloys after solution treatment at 540 ℃ for 10 h. Besides the larger grain size than Fig.1c and 1d, lots of coarse black granules are found within the grain boundaries, which indicate that the alloy is oxidized seriously. Both the grain sizes of α-Mg in Fig.1c and 1d are small, though slightly increase with the heat treatment temperature build-up but the segregation of the composition of the former is severer. A majority of Mg_2Sn phases in the alloy have been dissolved into the matrix after the solution treatment at 495 ℃ for 10 h. According to the Hall-Petch [14] equation:

$$\sigma = \sigma_0 + kd^{-1/2} \tag{1}$$

The grain refinement is an effective way to enhance the strength of crystalline materials, especially for the magnesium alloys, since the strengthening coefficient k is much larger (k = 280~320 MPa μm$^{1/2}$ in magnesium alloy, for others such as aluminum alloy k = 68 MPa μm$^{1/2}$)[15]. Therefore, the addition of Y and Zr is a promising method to refine grains and to improve

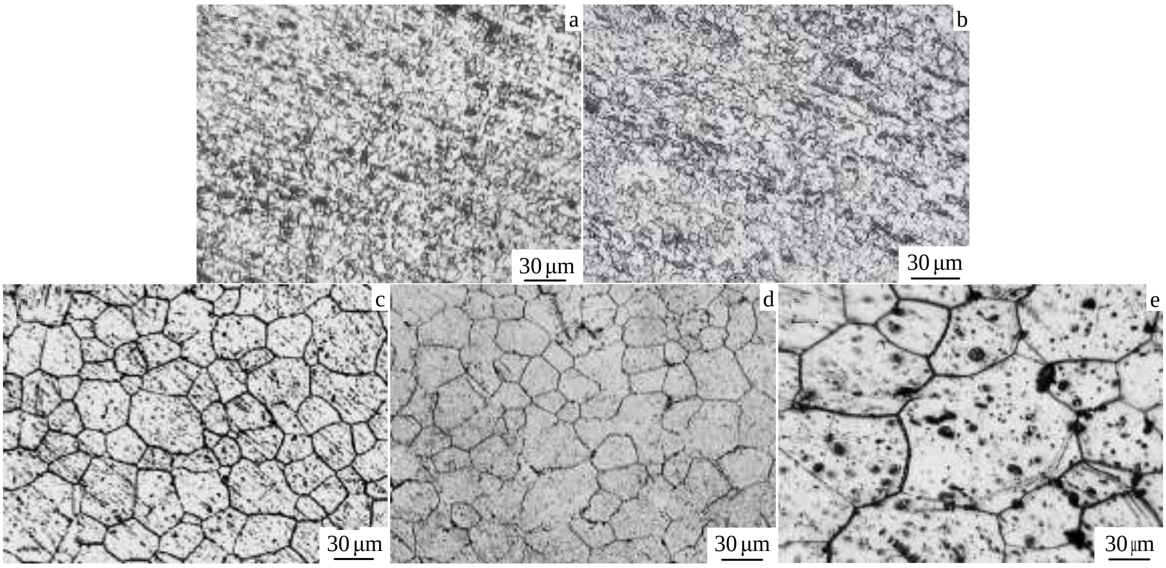

Fig.1 OM images of Mg-9Sn-1.5Y-0.4Zr alloys: (a) as-extruded, (b) solution-treated at 405 ℃ for 10 h, (c) solution-treated at 450 ℃ for 10 h, (d) solution-treated at 495 ℃ for 10 h, and (e) solution-treated at 540 ℃ for 10 h

the mechanical properties of the Mg-Sn based alloys[16]. In summary, the solution treatment at 495 ℃ for 10 h is optimal for the experimental alloys. After this procedure most of the second phase is dissolved in the α-Mg matrix. The influence of solution treatment can be clearly observed in Fig.2b.

The SEM image of experiment alloys after aging at 250 ℃ for 60 h is shown in Fig.2c. The features that the second phase distributing along grain boundaries and the non-uniform phase within grain boundaries transform to fine particles are clearly observed. These second phases at boundaries and in the grain interior can enhance the strength by obstructing the dislocation annihilation and by restricting the slip inside the grains during dislocation creep regime and can also inhibit the grain boundaries sliding during diffusion creep at elevated temperature. Therefore, the ultimate tensile strength (UTS) is enhanced while the elongation is decreased.

From the XRD patterns (Fig.4), it can be confirmed that the second phases are Mg_2Sn, MgY and $Mg_{24}Y_5$. They form as the segregation of Y in α-Mg matrix. It is known that both the MgY and $Mg_{24}Y_5$ phases are thermal stable phase, so the mechanical properties at elevated temperature can been enhanced.

2.2 Mechanical properties

The enhancement in mechanical properties of heat treated Mg-Sn-RE alloys is mainly attributed to three strengthening mechanisms which are grain boundary strengthening, solid solution strengthening, and precipitation strengthening. To completely describe their individual contribution, the three strengthening models aforementioned are employed for the as-extruded and peak aged specimens.

Fig.5 shows the variation of Vickers hardness under different aging conditions. For a given aging temperature, the hardness first increases and then gradually decreases with the increasing of aging time, which indicates that the experimental alloys perform obvious age hardening behavior. The peak hardness HV 890, 832 and 756 MPa are obtained after aging at 250 ℃ for 60 h, 200 ℃ for 84 h, 300 ℃ for 48 h, respectively.

It is well known that the size, shape and distribution of the Mg_2Sn phase are different with different aging time and

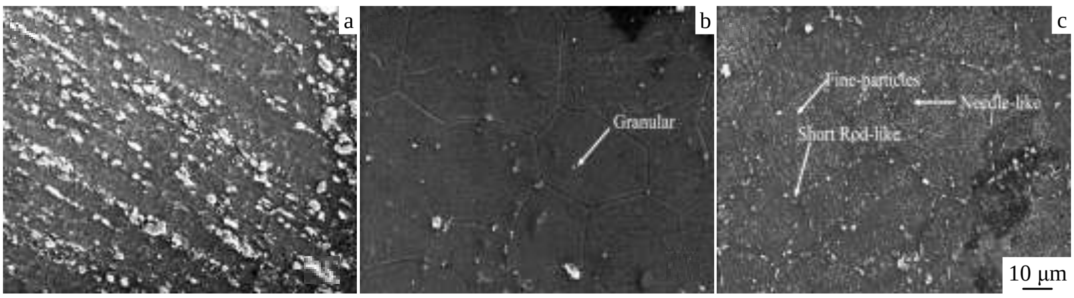

Fig.2 SEM images of Mg-9Sn-1.5Y-0.4Zr alloys: (a) as-extruded, (b) solution-treated at 495 ℃ for 10 h, and (c) aged at 250 ℃ for 60 h

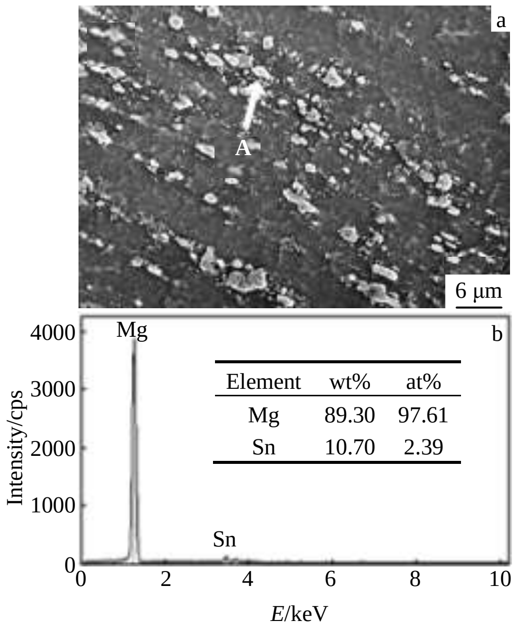

Fig.3 SEM image (a) and EDS analyzing result (b) of as-extruded Mg-9Sn-1.5Y-0.4Zr alloy

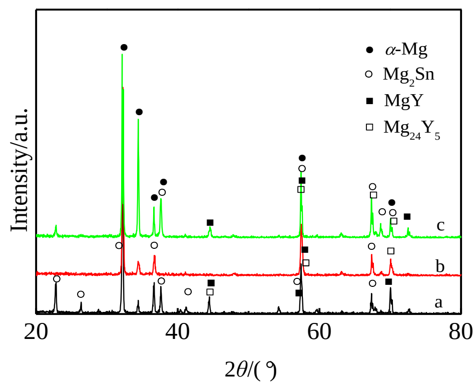

Fig.4 XRD patterns of Mg-9Sn-1.5Y-0.4Zr alloys: (a) as-extruded, (b) solution-treated at 495 ℃ for 10 h, and (c) aged at 250 ℃ for 60 h

temperatures. With the temperature increasing, the precipitate speeds up and the distribution becomes inhomogeneous and even coarse network. At last, the coarse network structure will be the crack source of the experimental alloys. So the mechanical properties will be deteriorated.

Fig.6 shows the tensile properties of the aging treated experimental alloy at 250 ℃. With the time increasing, the yield strength (YS) decreases at first to 24 h and then increases up to 72 h and dropped sharply after that. The elongation decreases continuously with the increasing aging time. Both the peak YS (218 MPa) and the peak hardness value (890 MPa) of the experimental alloys are obtained at 60 h. The peak YS of the aged alloy is obviously higher than that of as-extruded alloy, suggesting a strong aging hardening behavior of the experimental alloys. As shown in Fig.2c after aging at 250 ℃ for 60 h lots of short rod-like phases and refined particles distribute along the grain boundaries and within the grains. According to the study of Liu et al[8] the fine precipitations at the grain boundaries and within the grains are possibly related to the continuous non-uniform and uniform precipitation of Mg_2Sn phase during the aging treatment, respectively. It can be confirmed that the particles within the boundaries and the short rod-like phases are Mg_2Sn phases and the needle-like compounds are MgY, $Mg_{24}Y_5$ phases. Furthermore, Mg_2Sn, MgY and $Mg_{24}Y_5$ phases in the artificial aging treatment alloy are consistent with the XRD patterns (Fig.4). Based on the above analysis, the hardness change of the experimental alloys is the result of the Sn precipitated in the matrix again. The formation of Mg_2Sn phase increases the hardness first and then decreases it. As the aging time goes, the Mg_2Sn phase starts to grow and gather, and the behavior of over aging is the main reason of the hardness decrease. During the solid solution treatment, a majority of Mg_2Sn phases in the alloy dissolve in the α-Mg matrix and precipitate again during the aging treatment procedure. The heat treatment improves the structure and distribution characteristics of the secondary phase in the experimental alloys and thus the mechanical properties can be enhanced.

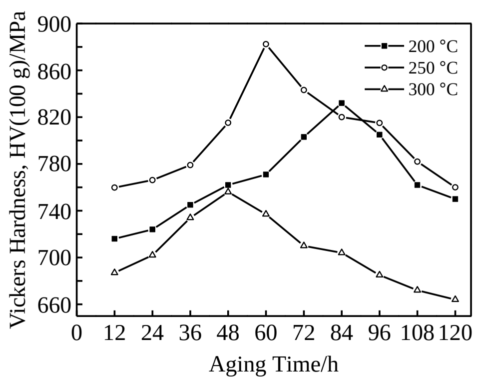

Fig.5 Aging hardening curves of Mg-9Sn-1.5Y-0.4Zr alloys under different aging conditions

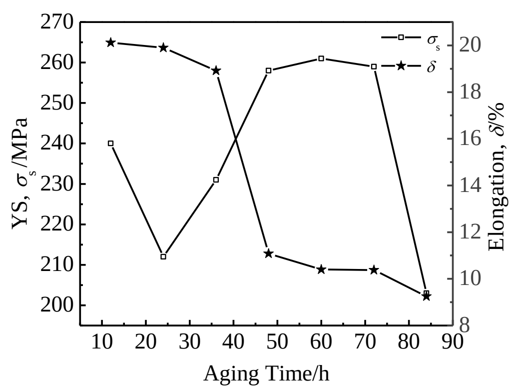

Fig.6 Tensile properties of Mg-9Sn-1.5Y-0.4Zr alloys aged at 250 ℃

Grain boundary strengthening σ_{gb} and solid solution strengthening σ_{ss} should be responsible for the yield strength of the as-extruded alloy. The equation is expressed as:

$$\sigma_y = \sigma_{Mg} + \sigma_{gb} + \sigma_{ss} \quad (1)$$

σ_{Mg} = 21 MPa[17] is the yield strength of pure Mg matrix. σ_{gb} can be calculated through the Hall-Petch equation:

$$\sigma_{gb} = \sigma_0 + kd^{-1/2} \quad (2)$$

where, σ_0 (24 MPa for Mg-Sn alloys) is the intrinsic resistance of the lattice to the dislocation motion, k (88~157 MPa for Mg-Sn alloy, the median value 122.5 MPa is taken in this paper) is a constant parameter and d (8 μm) is the average grain size of the as-extruded experimental alloy. Then σ_{gb} is calculated to be ~67.31 MPa which represents the contribution of grain refinement to the yield strength of the as-extruded experimental alloy.

With respect to the solid solution strengthening in multicomponent alloys, the model proposed by Gypen and Deruyttere is widely accepted for predicting strength using the following form[18,19]:

$$\sigma_{ss} = \left(\sum_i K_i^{1/n} C_i\right)^n \quad (3)$$

where, n is a constant with a value of 2/3 or 1/2. K_i and C_i are the strengthening and concentration constants for solute i, respectively. In the present study, only a small amount of Zr elements were introduced in the alloy as grain refiner during the casting procedure. The effect of solid solution Zr atom on the strength of the alloy is so little that they are not considered in the evaluation since the Zr element can not form intermediate secondary phase during casting, therefore the strengthening effects of Zr element can be ignored. Considering Sn and Y are the main solid solution strengthening elements in the experimental alloys, their combinative contributions to the yield strength can be described as:

$$\sigma_{ss} = \left(K_{Sn}^{1/n} C_{Sn} + K_Y^{1/n} C_Y\right)^n \quad (4)$$

where, K_{Gd} (1168 MPa at%$^{-2/3}$) and K_Y (1249 MPa at%$^{-2/3}$) are the strengthening constants in Mg-Gd and Mg-Y binary alloy[20]. In this paper, taking $K_{Sn}= K_{Gd}$ instead of K_{Sn} for the study on Mg-Sn alloy has not been clear yet and the similar characteristic between Sn and Gd. The assumption that no interaction exists between Sn and Y is applied and n takes a value of 2/3. Lastly, the total YS of the as-extruded alloy can be expressed as:

$$\sigma_y = \sigma_{Mg} + \sigma_0 + kd^{-1/2} + \left(K_{Sn}^{1/n} C_{Sn} + K_Y^{1/n} C_Y\right)^n \quad (5)$$

Taking C_{Sn}=1.98at%, C_Y = 0.44 at% and constants of σ_{Mg}, σ_0, n and k into Eq.(5): σ_y=187.28 MPa. This value is somewhat larger than the experimental results (173.95 MPa).

According to the above analysis, the precipitation strengthening σ_{ppt} can be obtained by subtracting the above evaluated results of σ_{Mg}, σ_{gb}' (d=22 μm) and σ_{ss}' (C_{Sn}=0.01at%) from the total YS of the aged experimental alloy. In the aged experimental alloy σ_{ppt} reaches to a high level of ~112.83 MPa because a number of atoms in the matrix are increasingly depleted by the formation of strengthening precipitates. The YS of peak aged alloy is much higher than that of the as-extruded alloy indicating a strong strengthening effect of precipitation strengthening (~51.76%) in the aged experimental alloy. This can be ascribed to the precipitation of much tiny strengthening phases in the peak aged alloy which can effectively prevent the gliding of dislocation and the deformation of grain boundaries during the tensile test.

Table 1 Tensile properties of the as-extruded and peak-aged Mg-9Sn-1.5Y-0.4Zr alloys

Alloys state	UTS/MPa	YS/MPa	Elongation/%
As-extruded	294	173.95	21.2
Peak-aged	262	218	10.4

In the peak aged experimental alloy, the combined effects of grain boundary strengthening and precipitation strengthening are the main strengthening methods for the excellent performance. Tensile properties of the as-extruded and the peak-aged Mg-9Sn-1.5Y-0.4Zr alloys are listed in Table 1. The optimum mechanical properties of peak-aged Mg-9Sn-1.5Y-0.4Zr alloys reach UTS = 262 MPa, YS = 218 MPa, and elongation = 10.4%

In summary, the influence of other element addition and different heat treatments on magnesium alloys mechanical properties is more obvious than that of one strengthening mechanisms. In order to explore the optimum heat treatment, it is an important task to deeply research the strengthening mechanism of heat treatment for Mg-Sn-RE alloy in the future.

3 Conclusions

1) The Mg-9Sn-1.5Y-0.4Zr alloy has an obvious aging hardening behavior. The Mg_2Sn, MgY and $Mg_{24}Y_5$ phases precipitated along the grain boundaries and within the grains after artificial aging treatment can enhance the hardness remarkably. The maximum hardness HV value in this study is 890 MPa with aging treatment at 250 ℃ for 60 h.

2) The mechanical properties of as-extruded Mg-9Sn-1.5Y-0.4Zr alloy can be highly improved by the heat treatment. After aging treatment the yield ratio is greatly increased while the elongation was decreased in some degree. Based on the experimental results, the optimum mechanical properties are UTS=262 MPa, YS=218 MPa, elongation=10.4% while the corresponding heat treatment technology is: solution treatment at 495 ℃ for 10 h and then aging at 250 ℃ for 60 h.

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热处理对挤压态 Mg-9Sn-1.5Y-0.4Zr 镁合金显微组织与力学性能的影响

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摘　要：对热处理的挤压态 Mg-9Sn-1.5Y-0.4Zr 镁合金显微组织与力学性能的影响进行了实验性探究。结果显示热处理对挤压态 Mg-9Sn-1.5Y-0.4Zr 镁合金显微组织与力学性能具有显著影响。挤压态合金主要由非均匀分布的 Mg_2Sn 相组成。经过 495 ℃，10 h 固溶处理之后，大部分 Mg_2Sn 相溶入到基体中。时效处理能大幅改善 Mg-9Sn-1.5Y-0.4Zr 合金的力学性能，最佳时效工艺为：在 250 ℃条件下时效 60 h。实验最终力学性能参数为：维氏硬度 HV 890 MPa，极限抗拉强度 262 MPa，屈服强度 218 MPa，延伸率 10.4%。基于实验结果分析，可发现对于经时效处理的挤压态 Mg-9Sn-1.5Y-0.4Zr 合金，沉淀强化是主要的强化因素（~51.76%）。

关键词：Mg-9Sn-1.5Y-0.4Zr 合金；热处理；显微组织；力学性能

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