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ARTICLE

Tailoring the Microstructure and Mechanical Properties of AZ91 Alloy by Rheo-rolling and Heat Treatment

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Abstract: The evolution of microstructure and mechanical properties of the continuous rheo-rolled AZ91 magnesium alloy during heat treatment was revealed. Two kinds of $Mg_{17}Al_{12}$ phases precipitated from the supersaturated magnesium matrix were observed: most of them distributed discretely at grain boundary while the rest precipitated in alloy matrix in small size. With higher aging temperature, atom diffusion velocity increased and more precipitates formed. As a result, the hardness and the tensile strength peaked at 16 h, whilst the elongation decreased with the increase of time and temperature. The optimal comprehensive mechanical properties were obtained after solution treatment at 415 °C for 20 h and aging treatment at 220 °C for 16 h. The tailored hardness (HV), tensile strength and elongation of the rheo-rolled alloy after heat treatment reach 990 MPa, 251 MPa and 4.5%, respectively, which are significantly higher than those of the untreated alloy. Compared with traditional processing methods, rheo-rolling followed by heat treatment demonstrates its effectiveness on tailoring and obtaining balanced mechanical properties of AZ91 alloy.

Key words: AZ91 alloy; rheo-rolling; heat treatment; microstructure; mechanical properties

Magnesium alloy is light structural metallic materials, which has been widely applied to consumer electronics, automobiles and biomedical devices due to its excellent performance, such as high specific strength, high specific stiffness, shock absorption and good impact resistance [1-4]. However, magnesium has a hcp crystal lattice, which has only three slip systems. Therefore, deforming of magnesium alloys is difficult at room temperature[5, 6]. Magnesium alloy is usually processed at high temperature and multi-pass annealing is needed, so the production cost is relatively high while the production efficiency is low. In addition, the mechanical properties of magnesium alloy processed by traditional methods need to be improved to meet the requirement of practical application. Rheo-rolling has been proved an innovative low cost processing method, via which homogeneous microstructure and balanced mechanical properties can be obtained. Watari has studied semisolid AZ31B magnesium alloy processed by rheo-rolling, via which alloy sheets with thickness of 2.5 mm were obtained[7]. Guan et al has successfully prepared Mg-Sn-Mn and AZ91 magnesium alloy sheets with improved mechanical properties by rheo-rolling[8, 9].

Heat treatment was reported as an effective method to tailor the microstructure and mechanical properties of magnesium alloys[10, 11]. The incoherent equilibrium $Mg_{17}Al_{12}$ can be precipitated directly from supersaturated magnesium matrix during aging treatment, and the secondary precipitates inside grains were proved to strengthening the matrix effectively[12]. Ju's study showed the effect of second phase strengthening was significant during aging treatment of

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AZ31 alloy and the mechanical properties of the alloy were significantly improved[13]. Our previous study also showed that small-sized $Mg_{17}Al_{12}$ could be precipitated homogeneously from the magnesium matrix during aging process, which played important roles in increasing tensile strength and elongation simultaneously[14]. However, the mechanism of evolution of the microstructure and mechanical properties of rheo-rolled magnesium alloy during heat treatment still needs to be further studied. In order to obtain tailored microstructure and mechanical properties of AZ91 alloy via rheo-rolling and heat treatment, the effect of different heat treatment on the microstructure and mechanical properties of AZ91 alloy prepared by rheo-rolling was studied. Based on the experimental results, the evolution mechanism of the microstructure and mechanical properties was revealed.

1 Experiment

The experimental material was AZ91 magnesium alloy, whose chemical compositions are: 9.0 wt% Al, 1.0 wt% Zn, 0.2 wt% Mn, and balanced Mg. The alloy was heated to 720 °C and held for 30 min by a resistance furnace (HLJ-10-12, Hengli Tech., China) under Ar protective atmosphere. AZ91 alloy sheets were prepared by a self-designed continuous rheo-rolling machine (Fig.1)[15], and the processing parameters were as follows: casting temperature from 670 °C to 690 °C, rolling speed of 0.052 m/s, and vibration frequency of 80 Hz[16]. When the melt touched the sloping plate, the melt was cooled rapidly. Therefore, heterogeneous nucleation formed on the surface of the sloping plate under large undercooling. Under the vibration and shear field, the nucleus ceaselessly fell off from the surface, which led to grain spheroidization and refinement during the rapid solidification.

A well-type resistance furnace (TM0910P; Hengtai, China) was used for heat treatment. The samples were quenched in water at 20 °C after solution at 415 °C for 20 h. The aging temperatures were 190, 220 and 250 °C. Samples were taken every 2 h and quenched in water at 20 °C. The samples were polished and etched by the solution of 15 mL HCl + 56 mL C_2H_5OH + 47 mL H_2O. A metallographic microscope (GX51; Olympus, Japan) was used to observe the microstructure, and a scanning electron microscope (SEM, SSX-550; Shimadzu, Japan) equipped with an energy-dispersive (EDS) was used for precipitate identification. An electronic tensile testing machine (CMT5305; MST, China) was used to test the mechanical properties of the alloy sheets, and the tensile speed was set as 3 mm/min. The size of the tensile sample is shown in Fig.2. The hardness value of the alloy was measured by a Vickers hardness tester (450SVD; Wolpert, USA). The applied load was 49 N and the holding time was 15 s. All the tests were carried out in five-duplicates, and the data were expressed as mean ± standard deviation (SD).

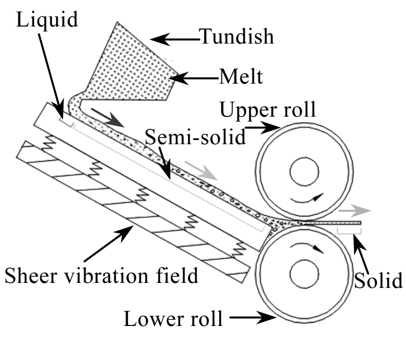

Fig.1 Schematic diagram of semisolid rheo-rolling process

2 Results and Discussion

When the metal melt touched the sloping plate, primary α-Mg matrix formed first on the surface of the sloping plate due to the water-cooling system in the plate. Dynamical non-equilibrium solidification occurred in the melt due to the coupling effects of shearing flow and shearing vibration from the sloping plate. The semisolid slurry was composed of primary α-Mg and melt rich in Al at the end of the sloping plate. The semi-solid alloy slurry was poured into the inlet, and stirred subsequently by the rotating rolls. Under the cooling effect of the rolls, α-Mg crystal formed quickly, and melt formed eutectic structure composed of refined α-Mg grain and $Mg_{17}Al_{12}$ precipitates. The microstructure of AZ91 magnesium alloy processed by rheo-rolling under the optimal process parameters is shown in Fig.3. The microstructure is mainly composed of spherical grain and equiaxed grain with the size of 20 μm to 30 μm, while dendritic grain is relatively fewer. The tensile strength, elongation and hardness (HV) of the processed sheet are 215 MPa, 3.02% and 750 MPa, respectively.

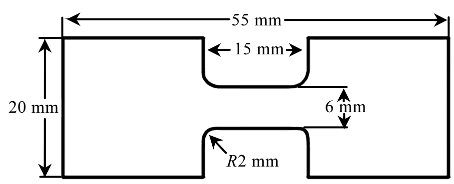

Fig.2 Scheme of the tensile test sample

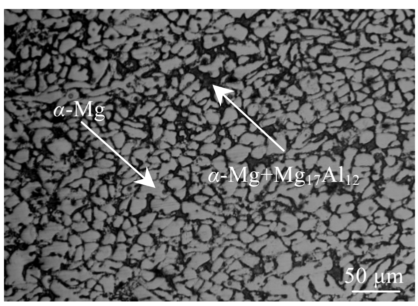

Fig.3 Optical microstructure of semisolid rheo-rolled AZ91 alloy

$Mg_{17}Al_{12}$ distributed in the grain boundary could be decomposed and dissolved into α-Mg matrix during solution treatment due to the high solubility (10.5 wt%) of Al in Mg at 415 °C. During the aging treatment at 220~250 °C, the supersaturated Al atoms in the matrix precipitated in the form of intermetallic compounds. Fig.4 and Fig.5 show the distribution of precipitates in different regions. The coarse and discontinuous precipitate (DP) in Fig.4 is inhomogeneously distributed at the grain boundary, while the much smaller continuous precipitate (CP) in flake shape in Fig.5 is uniformly distributed inside grain. EDS analysis of the precipitate at point A in Fig.4b and point B in Fig.5b show that the precipitates are composed of Al and Mg in a ratio of 17:12, which demonstrates the presence of $Mg_{17}Al_{12}$.

The microstructures of the alloy aged under different temperature and time are shown in Fig.6, Fig.7 and Fig.8. When the aging temperature is 190 °C, $Mg_{17}Al_{12}$ doesn't precipitate in the initial stage due to the low atom diffusion velocity at low aging temperature (Fig.6). When the aging time reaches 8 h, discrete $Mg_{17}Al_{12}$ DP starts to form at the grain boundary, and the grains are obviously coarsened. When the aging time is continuously prolonged to 16 h, small-sized $Mg_{17}Al_{12}$ CP is observed inside grain in a small amount. After 20 h aging, a lot of small-sized $Mg_{17}Al_{12}$ CPs are found to uniformly distribute inside grain. With the increase of the density of precipitates, the aggregation and growth of the precipitate is not observed. The phase transition driving force and diffusion velocity increase when aging temperature is raised to 220 °C. With increasing the aging time, the density of small-sized $Mg_{17}Al_{12}$ CPs also keeps increasing.

The growth of $Mg_{17}Al_{12}$ CP was observed when aging time reached 16 h (Fig.7). The density and growth of $Mg_{17}Al_{12}$ precipitates become more significant when aging temperature is raised to 250 °C, $Mg_{17}Al_{12}$ phase precipitates rapidly in less than 4 h (Fig.8). Due to the fast growth of $Mg_{17}Al_{12}$ precipitates at higher temperature, their distribution becomes inhomogeneous when aging time is extended to 16 h at 250 °C.

The relation of Vickers hardness, tensile strength, and elongation with aging time at different aging temperatures are illustrated in Fig.9. After solution treatment, the hardness (HV) value of AZ91 alloy decreases from 750 to 710 MPa owing to dissolving of primary $Mg_{17}Al_{12}$ phase. In the initial stage of aging treatment, the hardness value increases significantly. The hardness peaks at 20, 16 and 8 h when the aging temperature is 190, 220 and 250 °C, respectively. The peak hardness (HV) of 992 MPa is reached at 220 °C

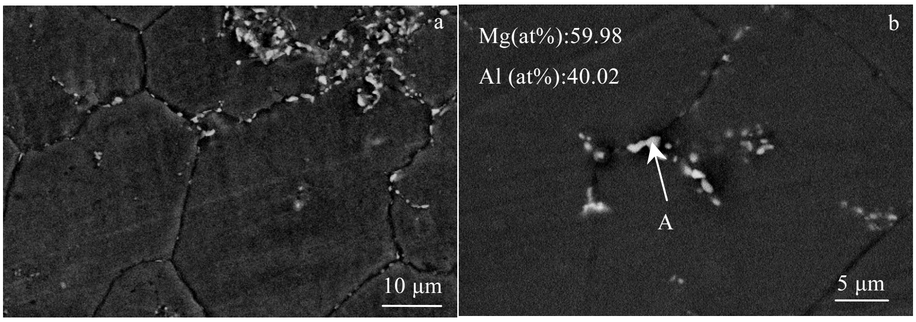

Fig.4 SEM images and EDS analysis of DP at grain boundary after aging treatment: (a) lower magnification; (b) higher magnification and EDS result of point A in Fig.4b

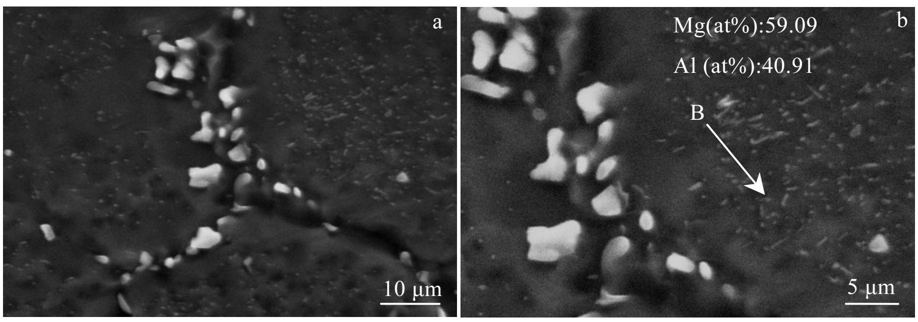

Fig.5 SEM images and EDS analysis of CP at grain boundary after aging treatment: (a) lower magnification; (b) higher magnification and EDS result of point B in Fig.5b

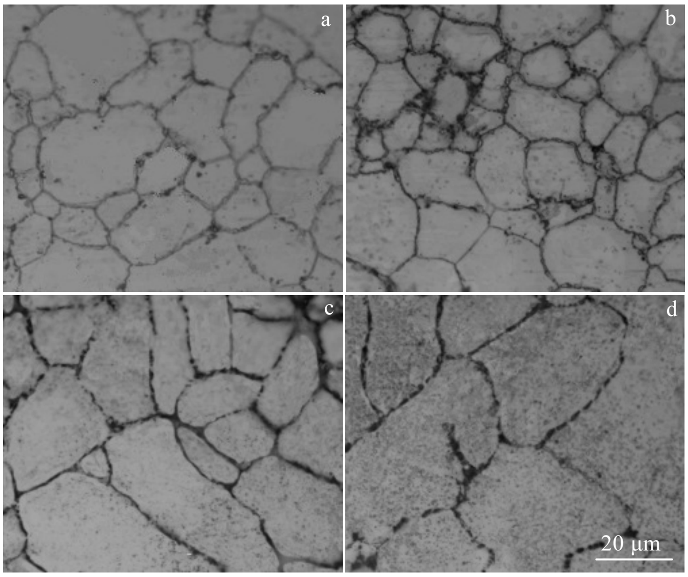

Fig.6 Microstructures of the alloy aged for different time at 190 °C: (a) 4 h, (b) 8 h, (c) 16 h, and (d) 20 h

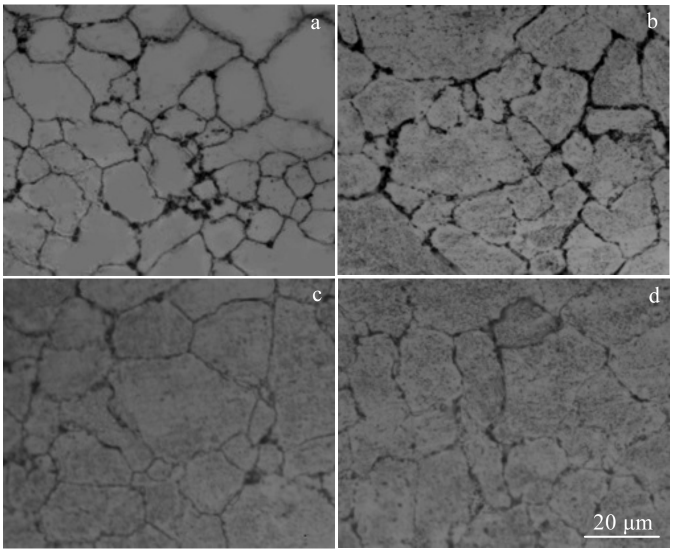

Fig.7 Microstructures of the alloy aged for different time at 220 °C: (a) 4 h, (b) 8 h, (c) 16 h, and (d) 20 h

after 16 h aging. In under-aging and over-aging condition, the harness improving effect is weaker than that in peak-aging condition. Due to higher temperature, the alloy entered the period of over-aging after 8 h at 250 °C because of the aggregation and fast growth of the secondary precipitates[17]. The morphology of precipitates performs significant influence on tensile strength during aging treatment (Fig.9b). Tensile strength of the alloy rapidly increases in direct proportion to the amount of small-sized $Mg_{17}Al_{12}$ CP in alloy matrix. The tensile strength increases slowly after aging for 4~12 h at 190 °C, because the coarse and discrete secondary phase is the chief form of precipitation which could not effectively strengthen the matrix. Besides, the coarse and fine grain interfaces shown in Fig.3 induces dislocation pile-up and leads to the back-stress strengthening[18]. This strengthening effect could also be promoted by the interfaces between small-sized precipitates and the matrix, via which the peak tensile strength of 251 MPa was obtained after aging for 16 h at 220 °C. When aging time exceeds 16 h, over-aging induced by aggregation and fast growth of secondary precipitates lead to significant loss of tensile strength. When aging temperature is 250 °C, the peak tensile strength is reached rapidly within 8 h as a result of the simultaneous precipitation of discrete $Mg_{17}Al_{12}$ DP and small-sized $Mg_{17}Al_{12}$ CP. However, the higher aging temperature leads to faster aggregation and growth of the secondary precipitates, which causes an early over-aging condition when the aging time exceeds 8 h.

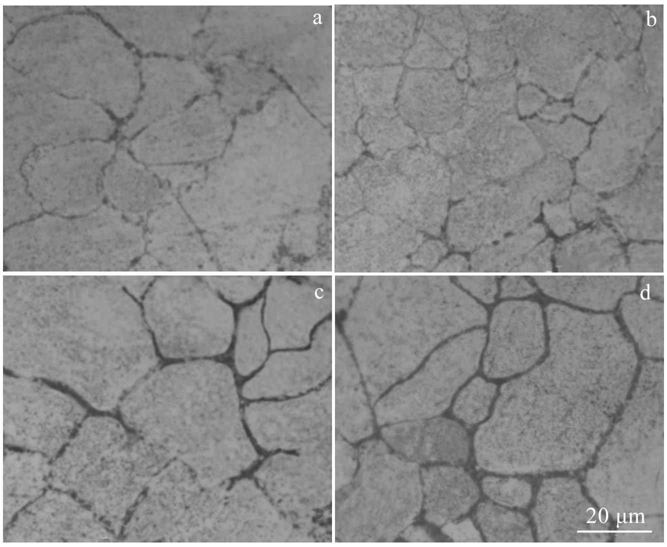

Fig.8 Microstructures of the alloy aged for different time at 250 °C: (a) 2 h, (b) 4 h, (c) 8 h, and (d) 12 h

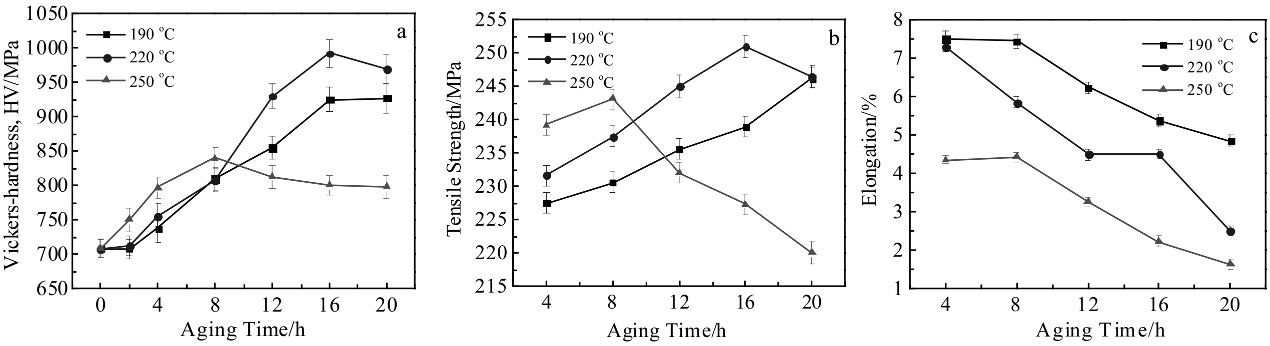

Fig.9 Effect of aging time and temperature on mechanical properties of the heat treated alloy: (a) Vickers hardness, (b) tensile strength, and (c) elongation

Different from the significant improvement of hardness and tensile strength, aging always have negative effect in elongation at different temperatures due to the formation of secondary precipitates (Fig.9c). After solution treatment, the elongation is obviously improved compared with that of untreated alloy due to dissolution of eutectic structure at grain boundary. During aging period, the elongation decreases because of the increase in brittleness brought by precipitation of DP. Duly's research proved DP's volume and morphology is more important to the alloy's elongation whereas CP mainly determines the alloy's hardness and strength[19]. Therefore, lower aging temperature at 190 °C leads to much smaller loss in elongation than at 250 °C because less DP precipitates at the early stage of aging. Hence, the optimal heat treatment parameters of continuous rheo-rolled AZ91 alloy are obtained as follows: 415 °C solution for 20 h and 220 °C aging for 16 h. The mechanical properties of the alloy process under the optimal parameters are: hardness (HV) of 990 MPa, tensile strength of 251 MPa, and elongation of 4.5%, which are significant higher than those of the untreated alloy. In the as-cast state, AZ91 alloys tensile strength are usually between 70 MPa and 120 MPa and their elongation are 2% to 5%[20, 21]. The difference in the strength and elongation can be caused by casting temperature, cooling rate, impurity level and solidification methods. Compared with traditional processing methods, rheo-rolling followed by heat treatment demonstrates its effectiveness on tailoring and obtaining balanced mechanical properties of AZ91 alloy.

3 Conclusions

1) The continuous rheo-rolled AZ91 alloy matrix is mainly composed of spherical grain and equiaxed grain with the size of 20 to 30 μm. Two kinds of secondary precipitates are identified after aging treatment, namely the coarse and discontinuous precipitate (DP) at the grain boundary and the small-sized continuous precipitate (CP) inside grain.

2) DP's volume and morphology is more important to the alloy's elongation while CP mainly determines the alloy's hardness and strength. Lower aging temperature at 190 °C leads to significant increase in Vickers hardness and tensile strength whilst much smaller loss in elongation than 250 °C, because less DP precipitates at the early stage of aging.

3) The optimal heat treatment parameters of continuous rheo-rolled AZ91 alloy are obtained as follows: 415 °C solution for 20 h and 220 °C aging for 16 h. The mechanical properties of the alloy processed under the optimal parameters are: hardness (HV) of 990 MPa, tensile strength of 251 MPa, and elongation of 4.5%, which are significant higher than those of the untreated alloy. Compared with traditional processing methods, rheo-rolling followed by heat treatment demonstrates its effectiveness on tailoring and obtaining balanced mechanical properties of AZ91 alloy.

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流变轧制 **AZ91** 合金的热处理和组织性能调控

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摘　要：对连续流变轧制 AZ91 合金在热处理过程中的组织和力学性能演化进行了研究。热处理后 2 种析出相在基体中出现：一种是晶界处的非连续析出相，另一种是从过饱和基体中析出的小尺寸连续析出相。随着时效温度的升高，原子扩散速度也随之提高，导致更多的析出相生成和长大。合金的维氏硬度和拉伸强度峰值在 16 h 时效后出现，而合金的延伸率随着时效时间的延长和时效温度的提高呈下降趋势。经过对实验结果的分析，适合提升合金综合力学性能的热处理制度为 415 ℃固溶 20 h 加 220 ℃时效 16 h。经热处理后得到的维氏硬度、拉伸强度和延伸率分别为 990 MPa，251 MPa 和 4.5%，各项性能均显著优于流变轧制态合金。相对于传统成型手段，流变轧制加热处理方法成型的 AZ91 合金展现了优异且均衡的综合力学性能。

关键词：AZ91合金；流变轧制；热处理；微观组织；力学性能

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