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Effects of aging treatment processes on microstructures and mechanical properties of AZ63 casting magnesium alloy

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Abstract: The effects of three different aging treatment processes, namely single-stage, double-stage, and reverse double-stage aging treatment processes, on the microstructures and mechanical properties of the AZ63 (Mg-6Al-3Zn-0.25Mn) casting magnesium alloy were investigated and compared. The results indicate that the microstructures of all the aged alloys under the three treatment processes are mainly composed of α-Mg, $Mg_{17}Al_{12}$, and Al_4Mn phases, indicating that the double-stage and reverse double-stage aging treatments have no obvious effect on the type of alloy phases. However, as compared with the single-stage and double-stage processes, the reverse double-stage process has a great effect on the quantity of the $Mg_{17}Al_{12}$ phases. After the reverse double-stage aging treatment, which results in a stronger drive for decomposition of the supersaturated solid solution, the number of $Mg_{17}Al_{12}$ phases precipitated in the grains significantly increases. In addition, as compared with the single-stage aged alloy, the tensile properties at room temperature for both the double-stage and reverse double-stage aged alloys are significantly improved. Among them, the reverse double-stage aged alloy achieves the highest tensile strength, yield strength, and elongation of 295 MPa, 167 MPa, and 8.6%, respectively.

Keywords: magnesium alloys; Mg-Al based alloys; AZ63 alloy; heat treatment; aging treatment

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1 Introduction

Magnesium alloys are the lightest structural alloys commonly used in the automotive, aerospace and other industries due to their low density, high specific strength and specific stiffness, good machinability, and electromagnetic shielding performance [1-3]. At present, the widely used magnesium alloys are from the Mg-Al-Zn series casting alloys such as AZ91 alloy, however, due to their relatively low strength and poor ductility as compared with casting aluminum alloys, they cannot meet the requirements for some high-performance components, which further limit their application [4-6]. Therefore, in order to improve the competitiveness of casting magnesium alloys in the light structural material market, it is urgent to develop high-performance casting magnesium alloys with low cost. It has been reported that the AZ63 (Mg-6Al-3Zn-0.25Mn) alloy of AZ (Mg-Al-Zn) system is a potential casting magnesium alloy with low cost [7, 8].

Incesu et al. [9] investigated the effects of aging temperature and time on microstructure and mechanical behavior of AZ63 magnesium alloy, and found that heat treatment can modify the morphology, stability of the precipitates and thus leads to enhanced properties. Aging treatment can change the amount and precipitation position of the $Mg_{17}Al_{12}$ phase, which may affect the mechanical properties of the alloy. In addition, Liu et al. [10] found that aging treatment can increase the continuity of the $Mg_{17}Al_{12}$ phase in AZ63 alloy, and the shape of the $Mg_{17}Al_{12}$ can change from the coarse lamellae to a fine continuous precipitation lamellae, thereby improving the performance of the alloy. At the same time, Wang et al. [11] developed a new AZ63 magnesium alloy containing small amount Y, Nd, Sn and Y additions. Its as-cast tensile strength and elongation were 235 MPa and 8.3%, respectively, however, after being T6-treated, its tensile strength and elongation reached 289 MPa and 11.8%, respectively.

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Despite the achievements mentioned above, the mechanical properties of AZ63 alloy are not satisfactory. Therefore, further enhancement in the properties for this alloy needs to be considered. The common heat treatment processes to improve microstructure and strength include: solid solution treatment, single-stage aging treatment, and double-stage aging treatment [12-14]. However, conventional heat treatment processes have limitations in improving the strength of alloys. Therefore, there is an urgent need to explore new heat treatment processes to improve the strength of alloys. It is found that reverse double-stage aging process is a new process to improve the strength of alloys. Its treatment process includes artificial aging at high temperature firstly and then low temperature [15]. The high temperature treatment can induce recrystallization of the grains in the alloy, so that the grain can be refined; the low temperature treatment can further stabilize and regulate the crystal structure of the alloy [16]. The reverse double-stage aging treatment is often used in Al alloys, but rarely used in Mg alloys [17, 18]. However, Yue et al. [19] found that the reverse double-stage aging can significantly improve the comprehensive mechanical properties of Mg alloys. This study reported on the effect of aging treatment process on the microstructure and mechanical properties of AZ63 alloy, especially compares the effects of three different aging treatment processes, namely single-stage, double-stage, and reverse double-stage aging treatment processes, on the microstructures and mechanical properties of AZ63 alloy.

2 Experimental

The Mg-6Al-3Zn-0.25Mn (wt.%) experimental alloy was prepared by pure Mg, Al, Zn (>99.9wt.%) and Mg-4wt.%Mn master alloy. Pure Zn, Al and Zn alloys were added into the Mg melt in an electrical-resistance furnace under a protective atmosphere of high pure CO_2 and SF_6 at 720 °C. The ratio of CO_2 to SF_6 was 10:1. When the melt temperature increased to 740 °C, Mg-4wt.%Mn master alloy was added to the melt. After being homogenized by artificial stirring at 740 °C and mixed completely, the melt was held at 740 °C for 20 min to refine by C_2Cl_6 additions and then poured into a permanent mould which was coated (h-BN) and preheated to 300 °C to obtain a casting with the size of Φ65 mm×200 mm. The diagram of tensile test is shown in Fig. 1. The actual chemical composition of the test alloy specimen was determined by ICP as follows: 91.31 Mg, 5.68 Al, 2.85 Zn, 0.16 Mn (wt.%).

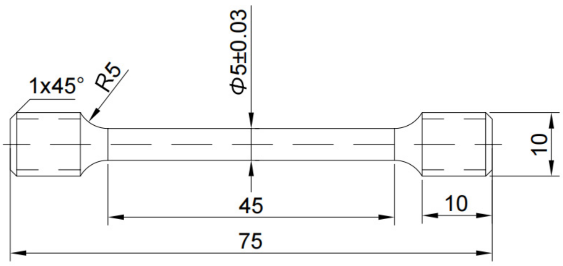

Fig. 1: Diagram of tensile specimen (unit: mm)

In addition, in order to determine the solutionizing temperature required for the experimental alloy, the differential scanning calorimetry (DSC) was carried out using a NETZSCH STA 449C system. Samples processed from the casting, weighing about 10 mg, were heated from 30 to 700 °C at a controlling speed of 10 °C·min^{-1} and kept for 5 min under a flowing argon atmosphere and then cooled down to 100 °C at the same speed in the furnace. Figure 2 shows the DSC differential thermal analysis curve of the as-cast AZ63 alloy. According to the DSC result and the results from the previous study, the optimal solid solution treatment was to hold at 400 °C for 18 h [20]. After solid solution treatment followed by water quenching, the samples were subsequently aged by the single-stage, double-stage and reverse double-stage aging treatment processes (Fig. 3), respectively. The design of the aging process is mainly based on the relevant literatures in the study of AZ system alloys, and the aging process was explored on the basis of the existing heat treatment process [21, 22]. The parameters of the three different aging treatment processes as shown in Fig. 3 are: 180 °C×36 h for the single-stage aging treatment, 180 °C×36 h+220 °C×6 h for the double-stage aging treatment and 220 °C×6 h+180 °C×36 h for the reverse double-stage aging treatment.

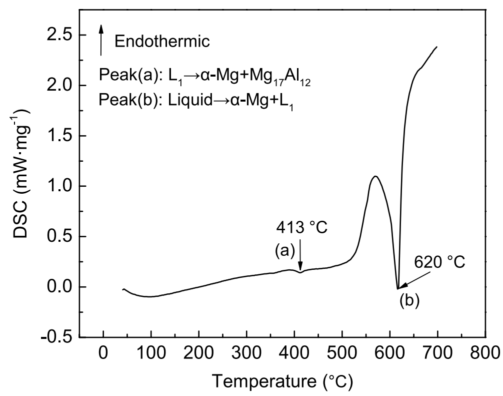

Fig. 2: DSC differential thermal analysis curve of as-cast AZ63 alloy

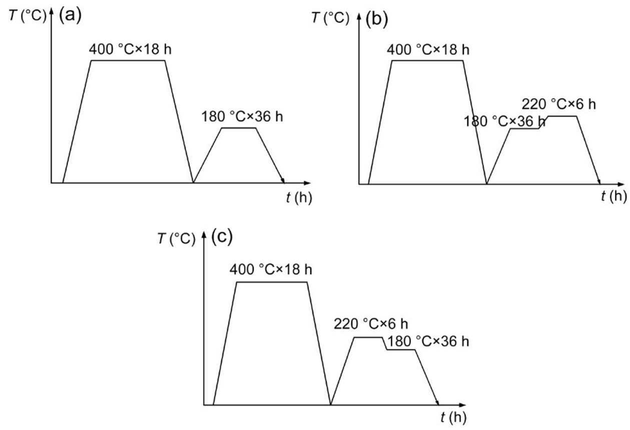

Fig. 3: Flowcharts of three different aging processes: (a) single-stage aged; (b) double-stage aged; (c) reverse double-stage aged

The as-cast samples were etched with 4% nitrate alcohol solution and the heat treatment samples were etched with corrosion solution (10 mL glacial acetic acid+10 mL distilled water+50 mL absolute ethanol+3.5 g picric acid), and then examined and analyzed with a Leica optical microscope, a Zeiss IGMA HDTM field emission scanning electron microscope equipped with energy dispersive spectroscope (EDS) and a JSM-6460LV scanning electron microscope (SEM). At the same time, the phases in the experimental alloys were analyzed by D/Max-1200X type X-ray diffraction (XRD). The tensile properties at room temperature for the as-cast and heat treated experimental alloys were determined with a CMT-5000 tensile tester at a rate of 2 mm·min^{-1}. The ultimate tensile strength (UTS), 0.2% yield strength (YS) and elongation to failure were obtained based on the average of three tests.

3 Results and discussion

3.1 Microstructures of AZ63 alloys in different states

Figure 4 shows the XRD results of the AZ63 experimental alloys in different states. The microstructures of all the AZ63 experimental alloys are mainly composed of α-Mg, $Mg_{17}Al_{12}$ and Al_4Mn phases. Furthermore, it is found that the peak intensities of the $Mg_{17}Al_{12}$ and Al_4Mn phases in the AZ63 alloys in different states are different. The peak intensities of the $Mg_{17}Al_{12}$ phases are the highest in the alloy aged by the reverse double-stage aging process, followed by the double-stage, and single-stage aging processes, respectively. The peak intensities of the Al_4Mn phases in the alloys in different states are weak due to the low content of Mn atoms. Previous relative investigations indicate that the double-stage aging process has a strong driving force to break down supersaturated solid solution and precipitate more second phases [23]. However, the difference in the peak intensities of these phases indicates that the reverse double-stage aging process can better promote the precipitation of $Mg_{17}Al_{12}$ phases. Figure 5 shows the SEM and EDS images of the as-cast AZ63 alloy. As shown in Fig. 5(a), the microstructure of the as-cast alloy consists of the gray-white α-Mg matrix and intermetallic compound, which is similar to the results of other AZ alloys [24, 25]. Based on the XRD and EDS results, the intermetallic compounds mainly distributed at the grain boundaries in Fig. 5(b) are identified as $Mg_{17}Al_{12}$ phases.

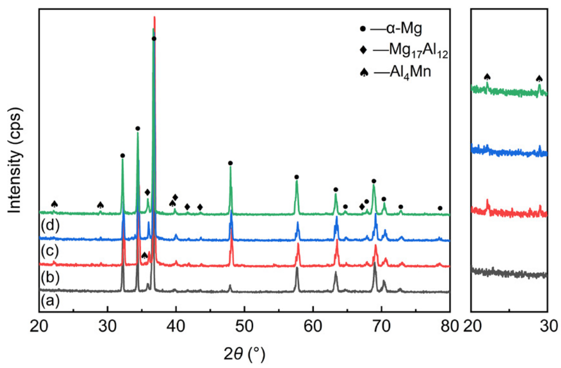

Fig. 4: XRD results of AZ63 alloys in different states: (a) as-cast; (b) single-stage aged; (c) double-stage aged; (d) reverse double-stage aged

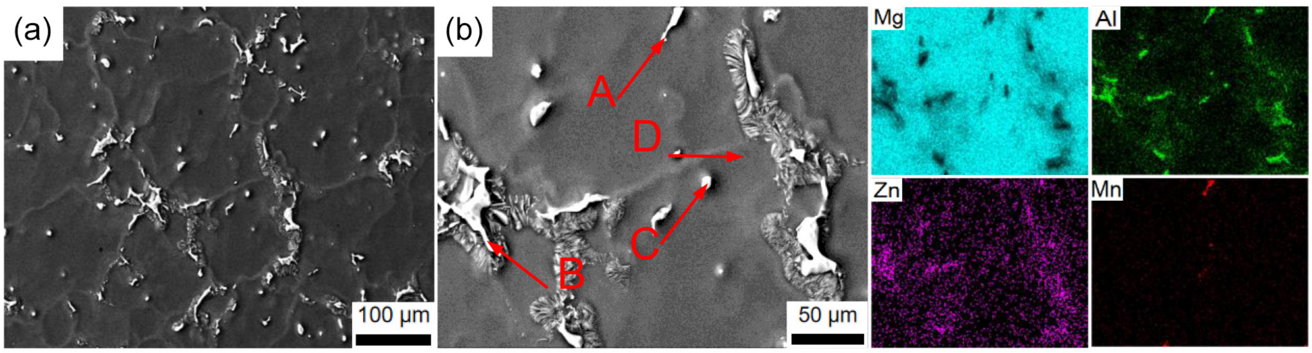

Fig. 5: SEM and EDS images of as-cast AZ63 alloy (a–b)

Table 1: EDS point scanning results of as-cast AZ63 alloy (at.%) in Fig. 1

Position	Mg	Al	Zn	Mn	Total
A	18.86	64.35	0.27	16.52	100
B	76.20	21.63	1.96	0.21	100
C	79.28	15.24	4.52	0.96	100
D	92.97	5.94	1.09	0	100

Figure 6 shows the optical and SEM images of the AZ63 alloys aged by different aging processes. Compared with the as-cast alloy, the grains are obviously refined and the second phases are evenly dispersed after 180 °C×36 h treatment [Figs. 6(a–c)]. The main reason is that the supersaturated solubility of the alloy is higher when it is held at 180 °C for 36 h, which results in a strong drive to decompose the supersaturated solid solution, and the number of precipitated second phases is large and uniformly distributed. At the same time, there is also a large amount of $Mg_{17}Al_{12}$ phases precipitating along grain boundaries on the alloy.

In general, in the double-stage aging process, the first-stage of aging is set at a lower temperature to obtain a great dispersion of precipitates; the second-stage of aging is set at a higher temperature to promote the second phase precipitation from the solid solution, growing to a certain size [26]. It can be seen from Fig. 6(d) that, after the double-stage aging treatment, the microstructure tends to be homogeneous and diffuse. As shown in Figs. 6(e) and (f), a small amount of $Mg_{17}Al_{12}$ phases are precipitated near the grain boundary with discontinuous precipitation, and a large amount of $Mg_{17}Al_{12}$ phases are also precipitated in the crystal with continuous precipitation [27]. The main reason is that an Al-poor region is formed around the $Mg_{17}Al_{12}$ phase, and the Al atoms required for continued growth need to spread for a long time to reach the crystal plane. Therefore, in the growth front of $Mg_{17}Al_{12}$ phases, some regions grow preferentially and extend to the α-Mg matrix. This reduces the diffusion distance of Al atoms to a certain extent, which is conducive to the growth of $Mg_{17}Al_{12}$ phase. Therefore, in terms of grain refinement and microstructure homogenization of the alloy, the double-stage aging process is superior to the single aging process.

It can be seen from Figs. 6(g) and (h) that, a number of lamellar $Mg_{17}Al_{12}$ phases precipitate near the grain boundary, which is distributed in a continuous network on the alloy, and a large number of flocculent $Mg_{17}Al_{12}$ phases are precipitated in the grain. At the same time, the number of second phases increases significantly, and most of them appear as granular and blocky phases diffused on the surface of the alloy after reverse double-stage aging [Fig. 6(i)]. Compared with single-stage and double-stage aging processes, the precipitation effect is more obvious.

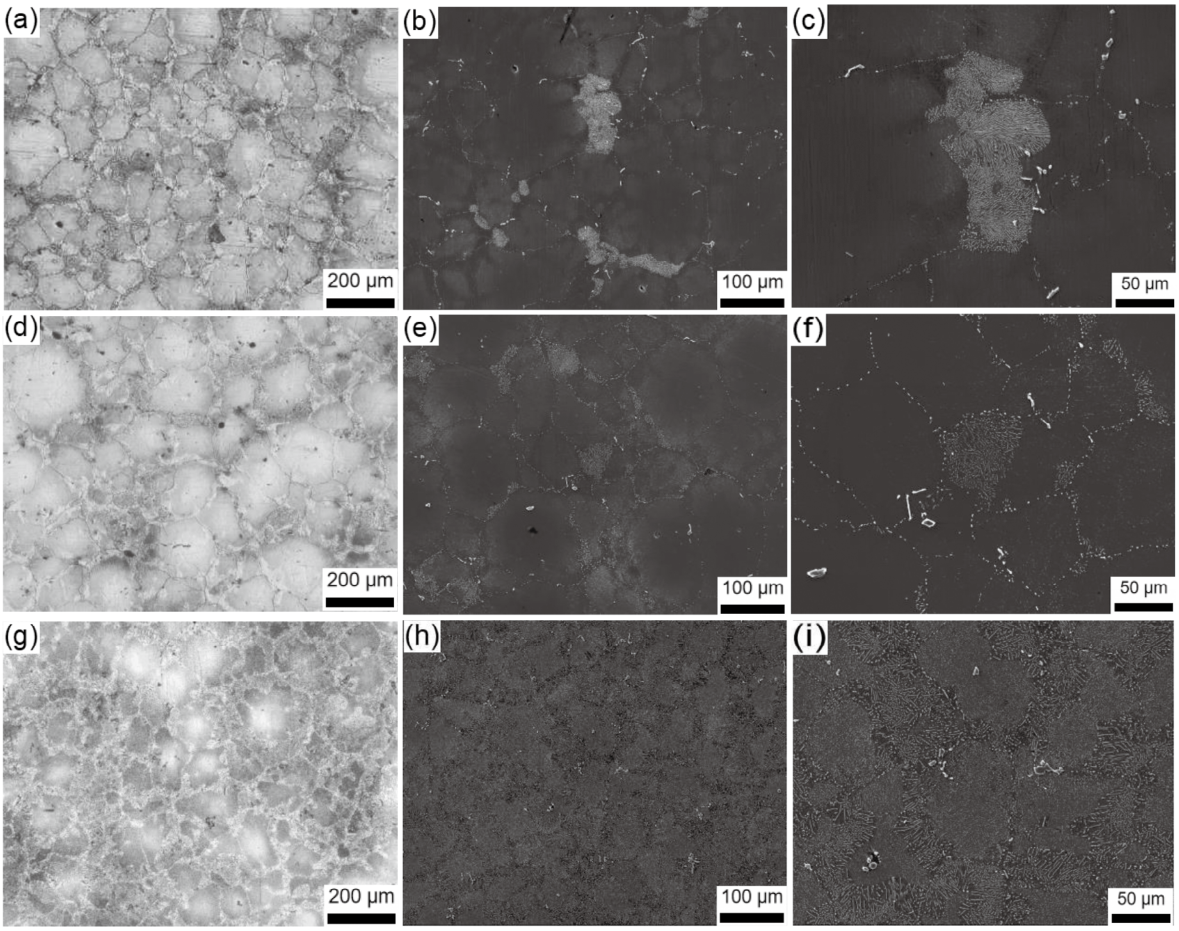

Fig. 6: Optical and SEM images of the AZ63 alloys aged by different aging processes: (a, b, c) single-stage aged; (d, e, f) double-stage aged; (g, h, i) reverse double-stage aged

Figure 7 shows the X-ray mapping results of the AZ63 alloy aged by different aging processes. After single-stage aging treatment, a tine discontinuous precipitation (DP) is distributed along the grain boundaries, as shown in Fig. 7(a). Meanwhile, a small amount of tine continuous precipitation (CP) is precipitated inside the grain. It can be seen from Fig. 7(b) that, the DP area of double-stage aging treatment is less than that of single-stage aging treatment. On the contrary, the area of CP is increasing. These results reconfirm that the predominant occurrence of one of the two types of precipitations influences the other precipitation behavior with respect to nucleation and growth. In other words, the discontinuous and continuous precipitation processes are mutually competitive. The CP area of reverse double-stage aging treatment is bigger than that of single-stage aging and double-stage aging treatments. This is consistent with the model for continuous and discontinuous precipitation proposed by Robson, where continuous precipitates have a pinning

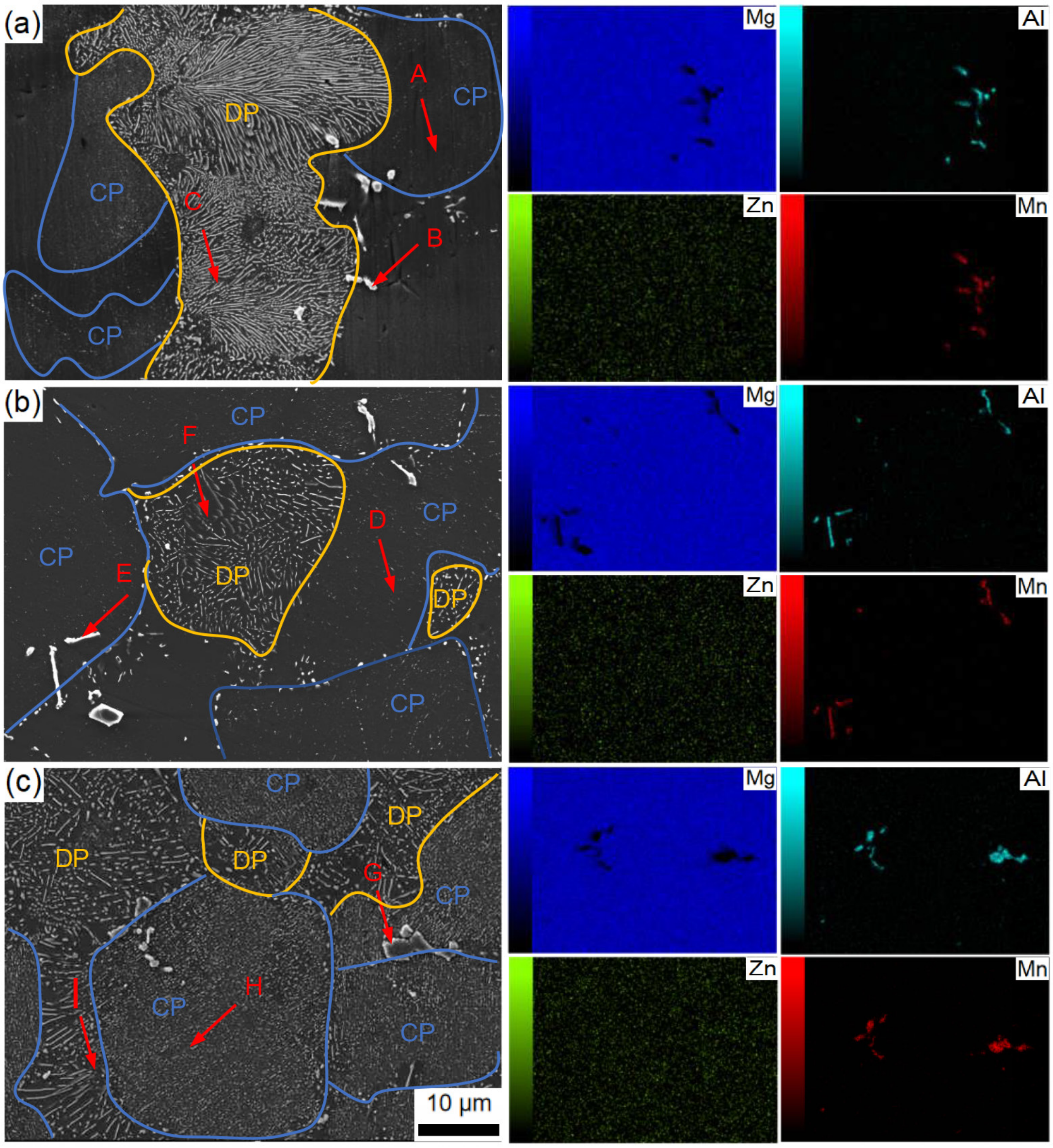

Fig. 7: X-ray mapping results of the AZ63 alloy aged by different aging processes: (a) single-stage aged; (b) double-stage aged; (c) reverse double-stage aged

effect on the migrating reaction front, which is necessary for the growth of discontinuous precipitates [28]. At the same time, after reverse double-stage aging treatment, a small amount of block compounds appear in the microstructure. According to the EDS surface scanning results, after three different treatment processes, Zn element is evenly distributed in the grain boundary and within the grain, while Al and Mn elements gather in the second phases. Apparently, Al atoms are more clustered due to its high content. The EDS point scanning results of the AZ63 alloy aged by different aging processes are list in Table 2. Combined with the EDS test results, the granular and stripy second phases in Figs. 7(a) and (b) are identified as Al_4Mn phases. Meanwhile, it can be inferred that blocky compounds are Al_4Mn phases in Fig. 7(c). In addition, the XRD results may be also further confirmed by the microstructures for the AZ63 experimental alloys in different states. Obviously, the reverse double-stage aging process has positive effect on the microstructure of the AZ63 magnesium alloys.

Table 2: EDS point scanning results of the AZ63 alloy aged by different aging processes (at.%) in Fig. 7

Position	Mg	Al	Zn	Mn	Total
A	93.46	5.29	1.25	0	100
B	45.11	42.93	0.32	11.64	100
C	88.88	9.64	1.48	0	100
D	91.85	6.96	1.11	0.08	100
E	16.92	67.25	0.12	15.71	100
F	89.58	8.47	0.93	1.02	100
G	19.19	63.82	0.23	16.76	100
H	93.79	5.13	1.08	0	100
I	92.41	6.06	1.53	0	100

3.2 Tensile properties of AZ63 alloys in different states

Tensile properties of the AZ63 alloys aged by different aging processes are shown in Fig. 8. It can be observed from Fig. 8 that the aging treatment significantly improves the tensile properties of the as-cast AZ63 alloy. Compared with the as-cast alloy, after the single-stage aging treatment, the ultimate tensile strength and yield strength are increased by 47.6% and 16.3%. Compared with the single-stage aged alloy, after the double-stage aging treatment, the ultimate tensile strength, yield strength and elongation are increased by 12.4%, 4.7% and 32.1%. The main reason is that, on the one hand, the presence of fine and homogeneous phases distributed along the grain boundaries can effectively hinder the slip and diffusion of crystals, improving the mechanical properties of alloys. Compared Fig. 7(a) with Fig. 7(b), the alloy by double-stage aging treatment has finer precipitates with a higher number density than the alloy treated by single-stage aging treatment. Moreover, a greater number of the short rod-like precipitates are observed in the alloy by double-stage aging treatment. According to the Orowan mechanism, the stress for dislocation bypassed the second phase is in direct proportion to the radius of the particle [29]. Refinement of precipitates is an important way to achieve strength improvement for precipitation-hardening magnesium alloys. Therefore, the double-stage aged sample exhibits higher strength and better plasticity than the single-stage aged sample since the finer precipitates are involved. Meanwhile, a significant number of precipitates contributes to the high strength according to the study by Xu et al. [30]. Apparently, after the double-stage aging treatment, more $Mg_{17}Al_{12}$ phases are easier to have more significant pinning effect on the dislocation. On the other hand, the fine second phase is diffusely distributed along grain boundaries, resulting in increased dislocation density, so that the stress is evenly distributed and further improves the strength [28]. Compared with the single-stage aged alloy, after the reverse double-stage aging treatment, the ultimate tensile strength, yield strength and elongation are increased by 17.5%, 11.3% and 53.6%, respectively. The main reason is that the formation of continuous precipitates within the grain has a more positive effect on the strength of alloys. Zhao et al. [31] found that AZ80 alloys attained the highest strength after aging treatment, which was mainly due to the fact that the formed CP was precipitation strengthened. Meanwhile, Kim et al. [32] found that at higher aging temperatures near the dissolution temperature, volume diffusion dominates the diffusion mechanism, resulting in occurrence of only continuous precipitation inside the α-Mg grains, which results in a significant increase in the mechanical properties of the alloy. Therefore, continuous precipitation of $Mg_{17}Al_{12}$ phases in the grain with good nailing effect hinders dislocation movement and further improves alloy properties [33]. Moreover, after reverse double-stage aging treatment, compared with long strip Al_4Mn phase, blocky Al_4Mn phase has less effect on the cleavage of matrix, which directly indicates that the mechanical properties of the alloy are significantly improved. Therefore, double-stage and reverse double-stage aging treatments have a significant effect on the microstructure and mechanical properties of the AZ80 alloy.

Figure 9 shows SEM images of tensile fractographs for the different AZ63 alloys tested at room temperature. As can be seen from Fig. 9, a number of cleavage planes and steps are present, and some minute tearing ridges can also be found in the tensile fracture surfaces, indicating that all the tensile fracture surfaces are characterized by a mixture of cleavage and quasi-cleavage fractures. Especially after the reverse double-stage aging treatment, a number of compounds exist in the alloy fracture, and the second phase is diffusely distributed near the tearing ridges, which can well prevent the dislocation motion, thus greatly improving the ultimate tensile strength and yield strength of the alloy. Obviously, the morphology of the AZ63 alloys tensile fracture coincides with the difference in tensile properties. It is well known that most Mg-Al series casting alloys fractured by transgranular fracture, intergranular fracture, and mixed transgranular/intergranular fracture [34]. In order to further study the fracture characteristic, the optical images of longitudinal sections for the different AZ63 alloys failed in tensile test at room temperature are shown in Fig. 10, It is observed that the fracture mode of AZ63 is changed from typical intergranular fracture to a mixture of transgranular and intergranular fracture through different aging processes. After the single-stage aging treatment, a number of large zigzag tooth appears on the alloy profile [Fig. 10(b)]. It can be seen from Figs. 10(c) and (d) that, after the double-stage and reverse double-stage aging treatments, a number of small and relatively smooth zigzag tooth appears on the alloy profile. Especially after the reverse double-stage aging treatment, the microstructure is dense and bright and without defects, and its strength reaches the highest among them.

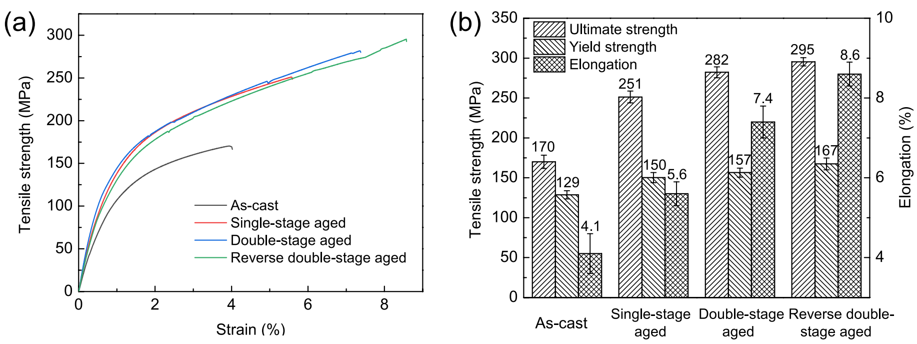

Fig. 8: Tensile properties of the AZ63 alloys aged by different aging processes: (a–b)

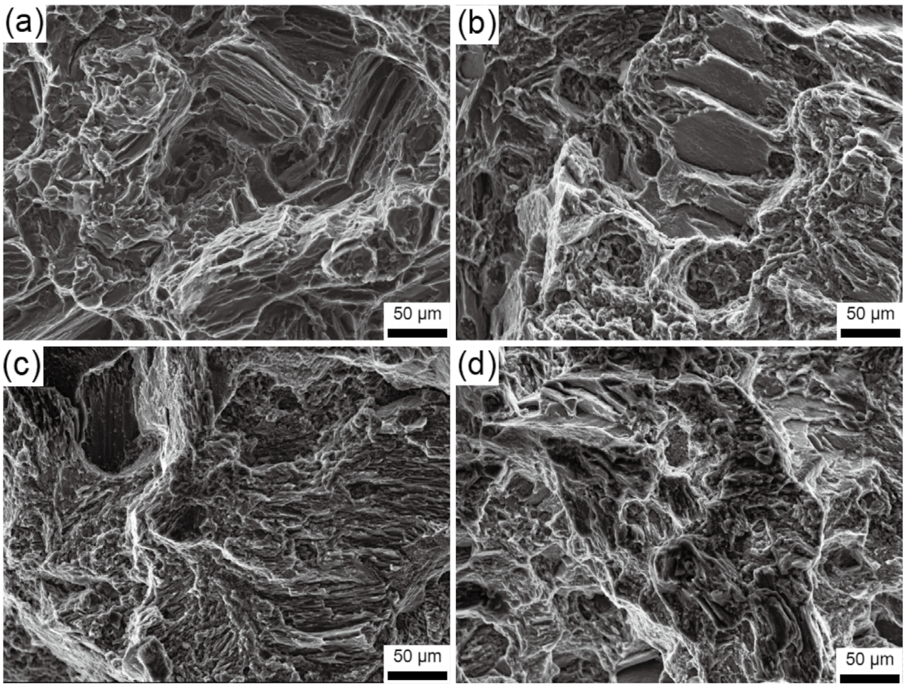

Fig. 9: SEM images of tensile fractographs for the different AZ63 alloys tested at room temperature: (a) as-cast; (b) single-stage aged; (c) double-stage aged; (d) reverse double-stage aged

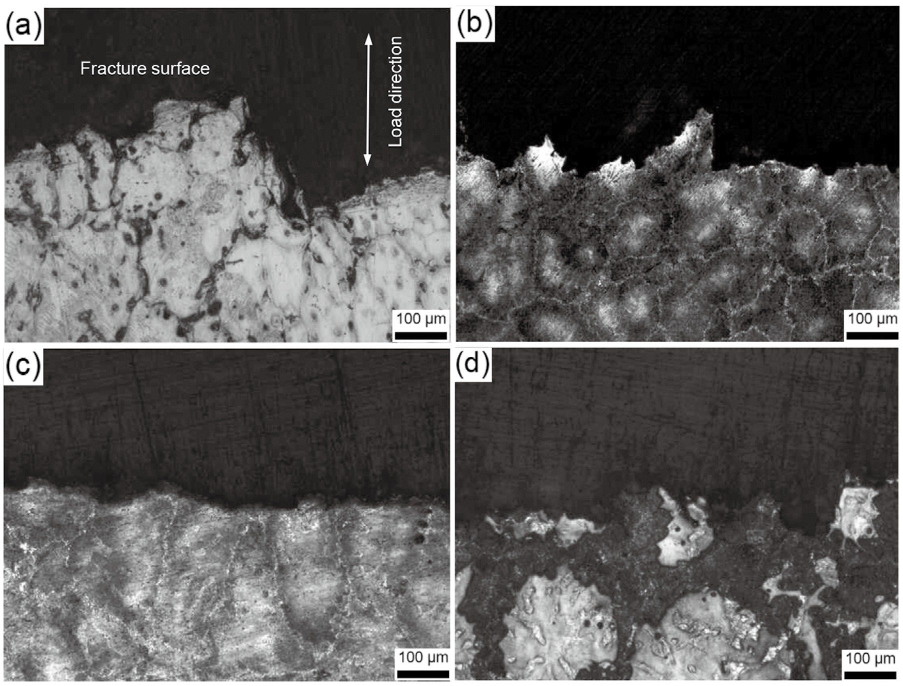

Fig. 10: Optical images of longitudinal sections for the different AZ63 alloys failed in tensile test at room temperature: (a) as-cast; (b) single-stage aged; (c) double-stage aged; (d) reverse double-stage aged

4 Conclusions

(1) The microstructures of the AZ63 alloys aged under the three aging processes are mainly composed of α-Mg, $Mg_{17}Al_{12}$ and Al_4Mn phases. After single-stage aging and double-stage aging treatments, the grains are significantly refined and a large number of $Mg_{17}Al_{12}$ phases precipitate from the vicinity of grain boundaries. Compared with the single-stage and double-stage aging treatment processes, the reverse double-stage aging treatment process has a great effect on the quantity of the $Mg_{17}Al_{12}$ phases. More $Mg_{17}Al_{12}$ phases are precipitated, especially within the grain. Meanwhile, after reverse double-stage aging treatment, a small amount of massive Al_4Mn phases appear in the microstructure.

(2) The tensile properties at room temperature for the AZ63 experimental alloy can be improved by single-stage, double-stage, or reverse double-stage aging treatment processes. After the single-stage aging treatment, ultimate tensile strength,

yield strength, and elongation obtained are 251 MPa, 150 MPa, and 5.6%, respectively. Compared with the single-stage aged alloy, after the double-stage aging treatment, the ultimate tensile strength, yield strength, and elongation are increased by 12.4%, 4.7% and 32.1%, respectively. In particular, after reverse double-stage aging treatment, the ultimate tensile strength, yield strength, and elongation of the alloy reach the highest, which are 295 MPa, 167 MPa, and 8.6%, respectively.

Acknowledgements

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Conflict of interest

The authors declare that they have no conflict of interest.

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