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Research on strengthening mechanism of AZ63 cast magnesium alloy based on precipitation phase orientation control

Shihui Ruan, Mingbo Yang, Haijun Wang, Yan Li, Renju Cheng, Yongfeng Zhou and Linxi Jiang

College of Materials Science and Engineering, Chongqing University of Technology, Chongqing, People's Republic of China

ABSTRACT

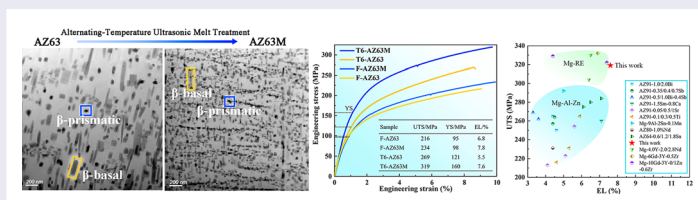
A high strength and toughness AZ63 cast magnesium alloy whose ultimate tensile strength, yield strength and elongation at room temperature respectively reached 319 MPa, 160 MPa and 7.6%, was developed by combining the alternating-temperature ultrasonic melt treatment and T6 heat treatment. The high as-aged tensile properties of the alloy with the alternating-temperature ultrasonic melt treatment are mainly attributed to the remarkable increase of the β -prismatic precipitates with orientation relationships (ORs) of $(\bar{3}\bar{3}0)_\beta // (1\bar{1}00)_\alpha$, $[\bar{1}\bar{1}1]_\beta // [11\bar{2}0]_\alpha$, thereinto the mentioned-increase is possibly related to that the solute-vacancy clusters along $0\bar{1}11$ plane might serve as the nucleation points of the β -prismatic precipitates.

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KEYWORDS

Magnesium alloys; prismatic precipitate; ultrasonic melt treatment; high strength and toughness



1. Introduction

Mg-Al-based cast alloys are the most widely used commercial magnesium alloys due to their high specific strength, low density and low cost, but their applications are limited by their low strength [1,2]. Precipitation hardening is one of the most efficient ways to improve strength, which is strongly related to the geometry factors and the growth orientation relationships (ORs), especially the ORs[3]. It is well known that the β -Mg₁₇Al₁₂ (143 m, a = 1.054 nm) precipitates in Mg-Al alloys have three types of ORs, one is the common Burgers ORs: $(011)_\beta // (0001)_\alpha$, $[\bar{1}\bar{1}1]_\beta // [2\bar{1}\bar{1}0]_\alpha$ for the basal precipitates, which is main growth orientation; the other two are the Crowley ORs: $(10\bar{1}0)_\alpha // (011)_\beta$, $[0001]_\alpha // [11\bar{1}]_\beta$ and Porter ORs: $(10\bar{1}0)_\alpha // (011)_\beta$, $[0001]_\alpha // [51\bar{1}]_\beta$ [3,4]. In Mg-Al-based alloys, the volume fraction of the non-basal β -Mg₁₇Al₁₂ precipitates is much lower than that of the basal β -Mg₁₇Al₁₂ precipitates. Therefore, more attention has been paid to the strengthening effects of the basal β -Mg₁₇Al₁₂ precipitates and then the relational effects of the non-basal

β -Mg₁₇Al₁₂ precipitates are ignored. However, previous investigations about aluminium alloys and rare earth magnesium alloys indicated that the prismatic precipitates had more positive strengthening effects than the basal precipitates [5]. For example, Nie et al.[5] reported that the Orowan's strengthening effect of the prismatic precipitates was at least one order of magnitude higher than that of the basal precipitates under the same size and quantity density conditions. In addition, Liao et al. [6] also found that $\langle a \rangle$ dislocation can easily bypass the β basal precipitates without forming a dislocation loop. It is very necessary to investigate the strengthening effects of the non-basal β -Mg₁₇Al₁₂ precipitates for Mg-Al-based alloys.

Previous investigations indicated that through $10\bar{1}2_\beta | \langle 1\bar{1}0\bar{1} \rangle_\beta$ twinning, Burgers ORs might be regulated and the original $(011)_\beta | (0001)_\alpha$ interfacial energies might be changed, and the β -basal precipitates would be transformed into the β -prismatic plates at aging process [7–10]. Liu et al. [11] successfully regulated the precipitate orientation of the β -Mg₁₇Al₁₂ precipitates from the

CONTACT Mingbo Yang ✉ yangmingbo@cqu.edu.cn College of Materials Science and Engineering, Chongqing University of Technology, No. 69, Hongguang Avenue, Banan District, Chongqing 400054, People's Republic of China; Haijun Wang ✉ 2939537998@qq.com College of Materials Science and Engineering, Chongqing University of Technology, No. 69, Hongguang Avenue, Banan District, Chongqing 400054, People's Republic of China

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basal plates to the prismatic plates for the AZ80 alloy by the coupling twinning, aging and detwinning processes (namely TAD), which resulted in an obvious increase of the yield strength. However, up to now, increasing the non-basal β -Mg₁₇Al₁₂ precipitates is difficult for Mg-Al-based cast alloys due to that twinning treatments are usually prepared by plastic deformation. In our ongoing investigations, the authors of this paper first found that the volume fraction of the non-basal β -Mg₁₇Al₁₂ plates in Mg-6Al-3Zn-0.25Mn cast alloy treated by combining the alternating-temperature ultrasonic melt treatment (ATUMT) and T6 heat treatment, might be increased, and then causing an obvious improvement in the tensile properties. This article reports the relevant results, and the special attentions are paid to the strengthening and toughening mechanism.

2. Materials and experiments

The experimental alloys without and with ATUMT, were respectively labelled as AZ63 and AZ63M alloys, which were prepared from commercially pure Mg, Al and Zn (> 99.9 wt%), and Mg-5wt.% Mn master alloy. The experimental alloys were melted in the stainless-steel crucible and protected by a mixed protective atmosphere of CO₂ and SF₆ with a ratio of 99:1. After being held at 720 °C for 20 min, the melt of the AZ63 alloy was stood by mechanical stirring and then poured into a permanent mold (mold temperature: 300 °C) to obtain a casting. For comparison, under the same conditions as the AZ63M alloy, the AZ63M alloy was also melted and cast beside the additional ATUMT. The ATUMT with the frequency of 20.1 kHz was carried out for the AZ63M alloy during the melt temperature range from 720 °C to 650 °C. In addition, the two alloys were solutionized at 400 °C for 12 h and then aged at 180 °C for 0 h to 36 h, respectively.

The actual compositions of the two alloys were determined by an inductively coupled plasma-optical emission spectrometer (ICP-OES) and were given as follows (wt.%): 90.48 Mg, 6.36 Al, 2.93 Zn, 0.22 Mn, 0.0029 Si and 0.0055 Fe for AZ63 alloy and 90.18 Mg, 6.73Al, 3.01Zn, 0.073Mn, 0.0015 Si and 0.0025 Fe for AZ63M alloy. The as-aged microstructures of the AZ63 and AZ63M samples were examined using a scanning electron microscope (SEM, σ IGMA HDTM) and transmission electron microscopy (TEM, JEOL JEM 2100F). Crystal defects in the solutionized AZ63M samples were analyzed using the invisibility rules of the dislocation ($\mathbf{g} \times \mathbf{b} = \mathbf{n}$, $\mathbf{g}$, $\mathbf{b}$ and $\mathbf{n}$ stand for diffracting vector, Burgers vector and integer numbers, respectively). The tensile properties at room temperature (RT) were tested by MTS E45.105 electronic universal testing machine at a constant strain rate of 1 mm/min.

3. Result

From the hardening curves in Figure 1(a), it is found that the peak-aged hardening value of the AZ63 and AZ63M alloys at 180 °C $\times$ 30 h is 86 HV and 102 HV, respectively. Engineering strain–stress curves in Figure 1(b) show that for the AZ63M alloy aged at 180 °C $\times$ 30 h, the ultimate tensile strength, yield strength and elongation at RT respectively reach 319, 160 MPa and 7.6%, which are significantly higher than those for the AZ63 alloy aged at 180 °C $\times$ 30 h. The strain hardening rates (Θ) of the AZ63M alloy are significantly higher than that of the AZ63 alloy (Figure 1(c)). The tensile strength of the AZ63M alloy is significantly higher than that of other reported Mg-Al-based alloys (Figure 1(d)) [12–20]. The above results indicate the AZ63M alloy after ATUMT has a better age-hardening ability than AZ63 alloys.

Figure 2(a,b) show the as-cast microstructures of the AZ63 and AZ63M alloys. The two as-cast alloys are composed of α -Mg, β -Mg₁₇Al₁₂ and Al-Mn phases. A significant change is that the β phase in the as-cast AZ63M alloy exhibits a distinct granular morphology instead of the semi-continuous network β phase. The Al-Mn compounds are identified as Al₈Mn₅ phases by bright-field TEM (BF-TEM) and fast fourier transform (FFT) in Figure 2(c), and it is an agglomerate formed by multiple Al₈Mn₅ particles. After solution treatments, as shown in Figure 2(d,e), the Al₈Mn₅ clusters can remain in the Mg matrix due to the high melting point and good stability, and their distribution in the AZ63M alloy is more dispersed and smaller than that of the AZ63 alloy. These observations indicate that the collapse of the cavitation bubbles in the ATUMT process results in high pressure, which is conducive to the disintegration of the Al₈Mn₅ clusters into some smaller particles. Previous investigations found that the Al₈Mn₅ phases which had a low degree of lattice mismatch with β phases, can serve as a nucleation substrate for β phases [21]. The fine and dispersed Al₈Mn₅ phases would increase the number of nucleation substrates of β phases, further promoting the formation of the granular β phases. In addition, the ATUMT also causes the average grain size of the as-cast AZ63 alloy to decrease from 110 μ m to 90 μ m. Accordingly, the as-cast tensile properties of the AZ63M alloy are slightly improved by the refinements of β phases and grains.

As shown in Figure 3(a,b), the precipitation density in the as-aged AZ63M alloy (180 °C $\times$ 30 h) is higher than that in the as-aged AZ63 alloy. The high-resolution transmission electron microscope (HR-TEM) and FFT in Figure 3(c,d) show that the plate-like precipitates (yellow square in Figure 3(a,b)) are β -basal precipitates with the ORs: (110) $_{\beta}$ // (0002) $_{\alpha}$, $[\bar{1}11]_{\beta}$ // $[11\bar{2}0]_{\alpha}$. The finer block

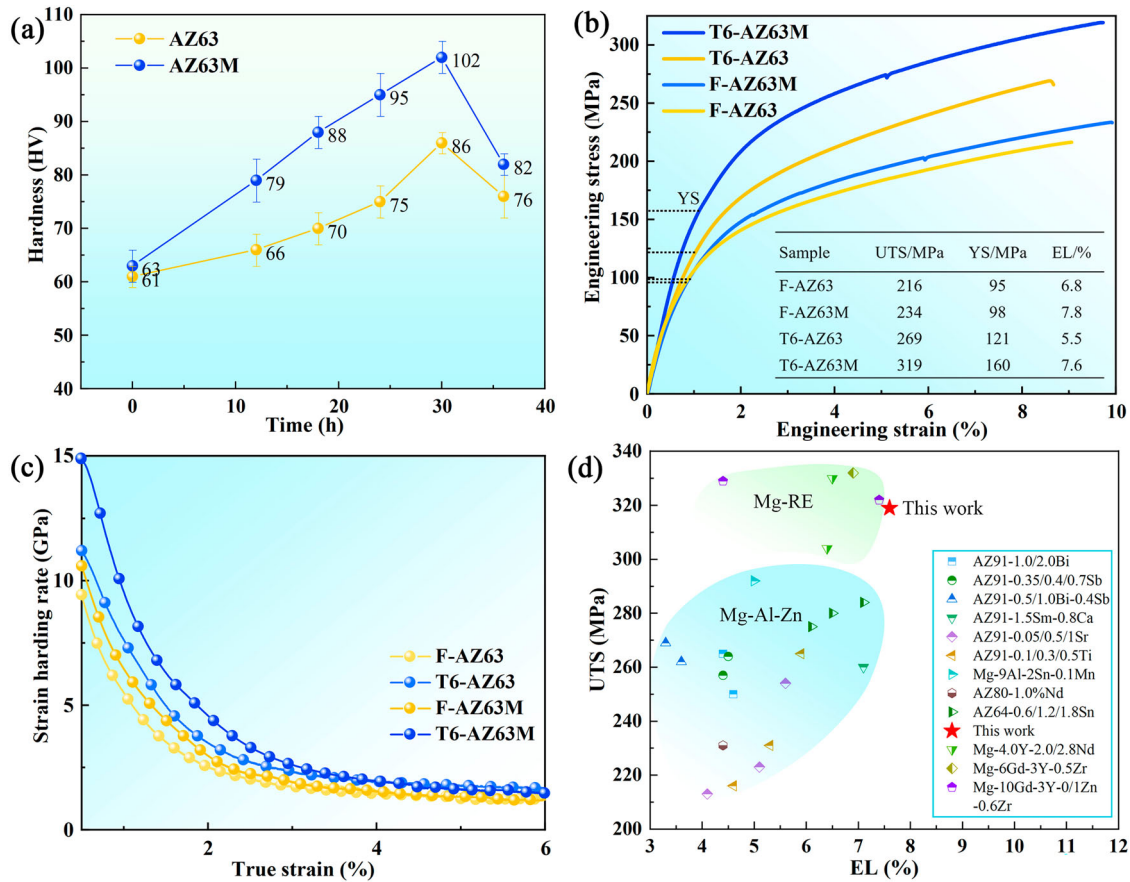


Figure 1. The mechanical properties of the alloys: (a) aging-hardening curves, (b) tensile properties, (c) strain hardening rates, (d) comparison of UTS and EL at RT between this work and the references [12–20].

nano-precipitates (blue square in Figure 3(a,b)) in Figure 3(e,f) are β -prismatic precipitates with the ORs: $(\bar{3}30)_\beta // (1\bar{1}00)_\alpha$, $[111]_\beta // [11\bar{2}0]_\alpha$. Based on the TEM images and Image J Software, the uniform diameter, thickness, and volume fraction of the β -basal precipitates (309 nm, 55 nm, and 0.15) and the β -prismatic precipitates (60 nm, 50 nm, and 0.045) for the as-aged AZ63 alloy are measured, respectively. Similarly, the uniform diameter, thickness, and volume fraction of the β -prismatic precipitates for the as-aged AZ63M alloy is 45 nm, 35 nm, and 0.08, respectively. For the aged-AZ63M alloy, the decrease in the size of the basal phase and the increase in the number of the prismatic suggests that there are changes in the original distribution of solute atoms. As shown in Figure 3(g), some cellular of the coarse discontinuous precipitation (DP) regions (confirmed by FFT as β phase in Figure 3(h)) in the as-aged AZ63 alloys nucleate along one side of the grain boundary and grow into inner grains, and the other sides form continuous precipitation (CP) regions. Some research had proposed that the lamellar DP region was a Burgers ORs precipitation [22]. Oppositely, it is found from Figure 3(i) that for the as-aged AZ63M alloy there are only some DP cellular

with a Z-shaped pattern in the grain boundary, and many CP regions grow on both sides of the grain boundary. These indicate that the solute concentration on both sides of the grain boundary is insufficient to provide further growth for DP cells, so they grow in a CP manner. It is also a more intuitive method that can determine the distribution and segregation phenomenon of solute atoms during the solid solution process through the distribution of precipitates. The growth of the coarse DP zone indicates severe local segregation in the as-aged AZ63 alloy (Figure 3(j)), this uneven distribution of solute concentration leads to a decrease in solute concentration within grains. Compared with the as-aged AZ63 alloy, the segregation of solute atoms in the as-aged AZ63M alloy is not serious (Figure 3(k)), thus increasing the concentration of the solute atoms within grains and applied to the precipitation growth.

In addition, the higher as-aged tensile properties for the AZ63M alloy than those for the AZ63 alloy might be further explained by the following calculation. Previous investigations have indicated that the increment of CRSS ($\Delta\tau$), which reflects the strengthening effects of basal plates and prismatic plates on basal slip, can be predicted

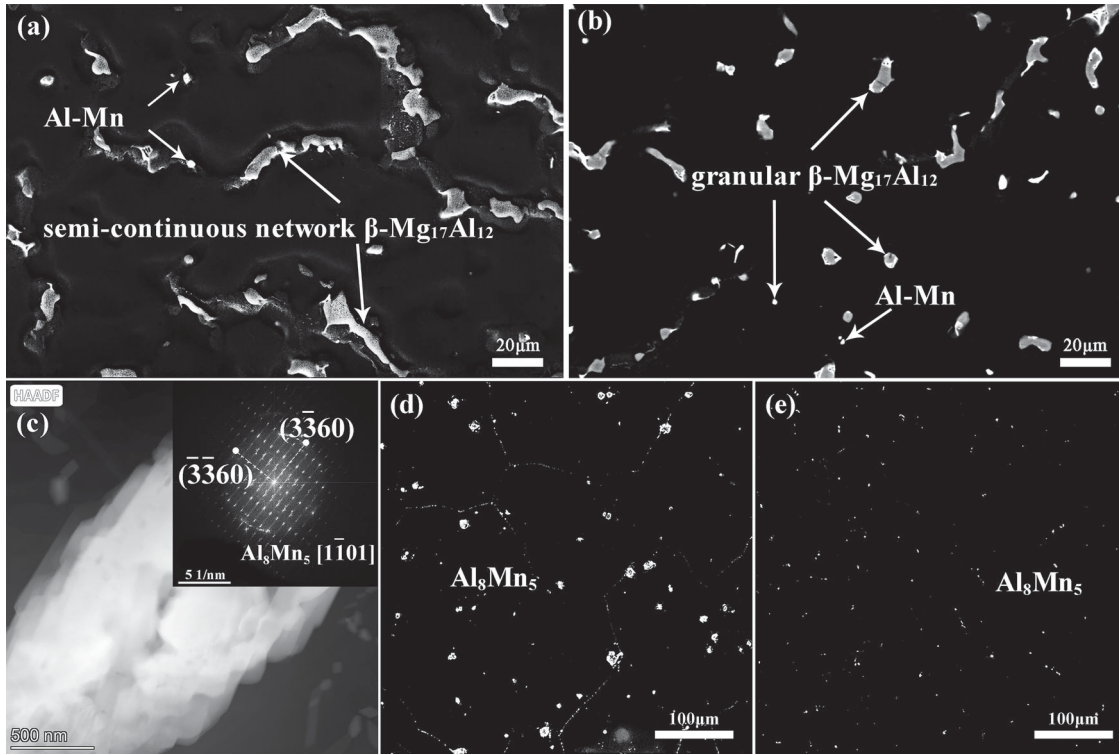


Figure 2. (a, b) SEM images of as-cast microstructure for AZ63 and AZ63M alloy, respectively, (c) BF-TEM and FFT images of Al_8Mn_5 clusters, (d-e) SEM images of solution microstructure for AZ63 and AZ63M alloy, respectively.

by the following relationships, respectively [5]:

$$\Delta\tau = \frac{Gb}{2\pi\sqrt{1-\nu}\left(\frac{0.953}{\sqrt{f}} - 1\right)D} \ln \frac{D}{b} \quad (1)$$

$$\Delta\tau = \frac{Gb}{2\pi\sqrt{1-\nu}\left(0.825\sqrt{\frac{DT}{f}} - 0.393D - 0.886T\right)} \ln \frac{0.886\sqrt{DT}}{b} \quad (2)$$

where D , T , and f are the uniform diameter, thickness, and volume fraction of the precipitates, respectively. G and ν are the shear modulus and the Poisson's ratio of the Mg matrix, they respectively are 16.5 GPa and 0.35 for Mg alloys. The magnitude of b is approximated to be the core radius of the dislocations. Accordingly, the $\Delta\tau$ of 16 and 36 MPa for the basal slip, respectively produced by basal and prismatic precipitates in the as-aged AZ63 alloy, and the $\Delta\tau$ of 65 MPa for the basal slip produced by the prismatic precipitates in the as-aged AZ63M alloy, are calculated according to the Equations 1 and 2. As far as the age hardening of the AZ63 alloy is concerned, the prismatic precipitation is more effective in inhibiting the basal slip than the basal precipitation. Thus, the higher density number of the β -prismatic precipitates is the main cause of the further increase in the YS and Θ of the AZ63M alloys after ageing. Similarly, as

shown in Figure 1(d), the strength of the as-aged AZ63M alloy mainly reinforced by the β -prismatic precipitates is significantly higher than that of Mg-Al alloys reinforced by the basal precipitates and is close to the strength of Mg-RE alloys reinforced by the β' -prismatic precipitates (Mg-RE).

4. Discussion

As mentioned earlier, improving the volume fraction of β -prismatic precipitates in Mg-Al alloys is a key way to enhance the mechanical properties. It is well known that the solute segregation at the atomic scale is the first step in precipitation nucleation processes, and this will determine the subsequent growth orientation of precipitation. Aging-hardening curves indicate that nucleation sites of the β -prismatic precipitates in the AZ63M alloy occur in early aging, and its quantity is higher than that in the AZ63 alloy. In addition, it is inferred that the increase in nucleation sites of the β -prismatic plates was caused by other factors instead of the elastic strain energy or the interfacial energy due to no significant changes in alloy composition and growth size of the β -prismatic plates. Recently, Peng et al. [23] proposed that solute-vacancy binding energy in Mg-Al alloys dominates the solute segregation rather than the stress when the size of solute-vacancy clusters exceeds the critical value, and the

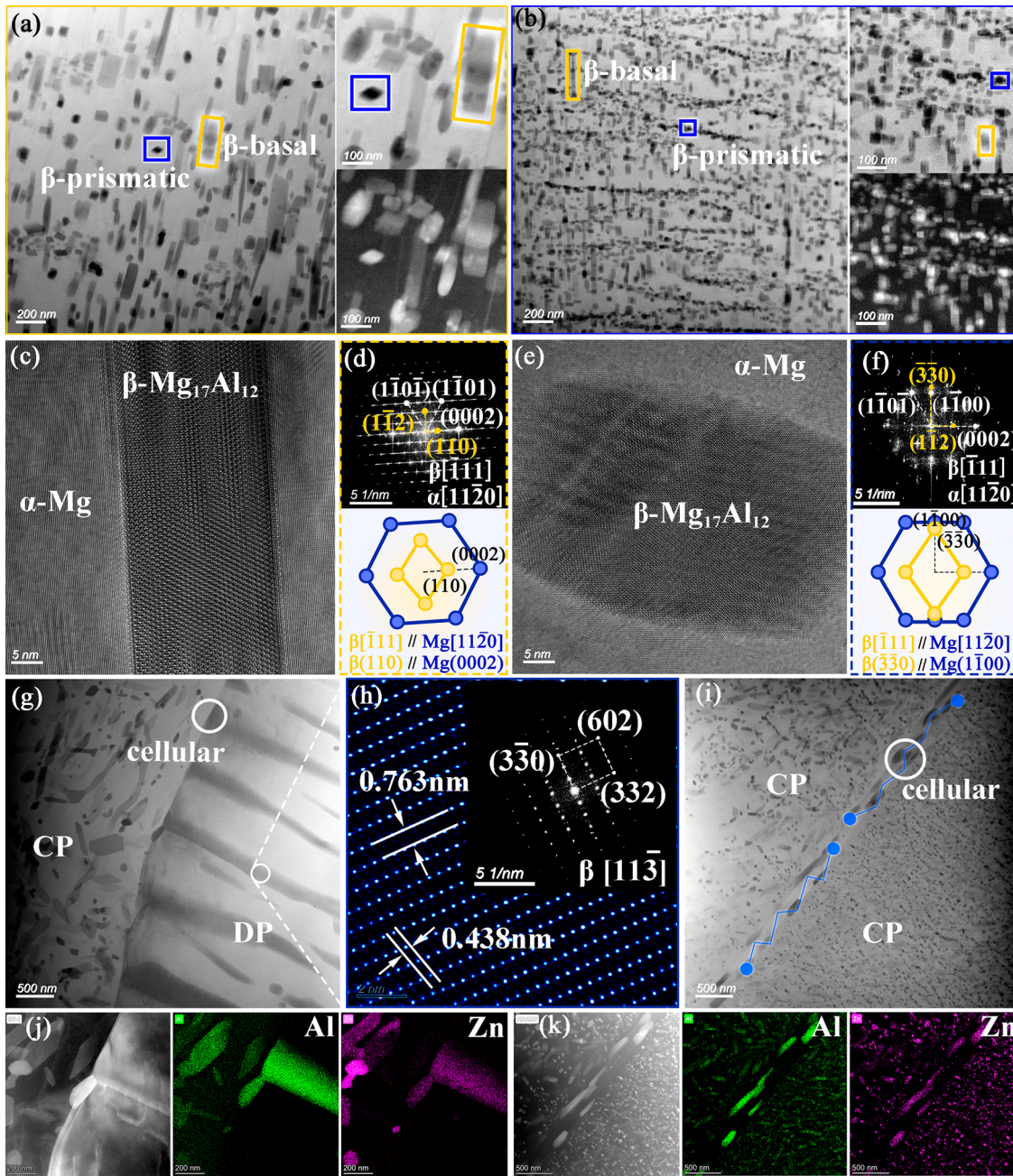


Figure 3. TEM images of the aged alloy ($z = [11\bar{2}0]$): (a, b) BF-TEM of nano-precipitation in AZ63 and AZ63M alloys, respectively, (c, d) HR-TEM, FFT and diagram between basal precipitation and matrix, (e, f) HR-TEM, FFT and diagram between prismatic precipitation and matrix, (g) BF-TEM of grain boundary precipitates in as-aged AZ63 alloy (h) HR-TEM and FFT of DP regions, (i) BF-TEM micrographs of grain boundary precipitates in as-aged AZ63M alloys, (j, k) EDS mappings of grain boundary precipitates in as-aged AZ63 and AZ63M alloy, respectively.

solute-vacancy clusters will be as the nucleation sites of precipitates. Therefore, it is further inferred that the size of the solute-vacancy clusters was significantly improved after solute homogenization, and then further forming the nucleation sites of the β -prismatic precipitates. However, the detection of vacancies remains challenging until now, and existing research mainly focuses on deformable alloys because of that the higher density vacancies are

easy to detect and collect by positron annihilation spectroscopy (PAS) [24–26]. Recently, based on the effects that vacancy clusters can form Frank dislocation loops after collapsing[27], Chen et al. [28] indirectly characterised the actual distribution of vacancies of casting aluminium alloy through Frank dislocation loops in double-beam TEM conditions ($g = 101$). In order to further verify our supposition, the as-solutionized microstructure

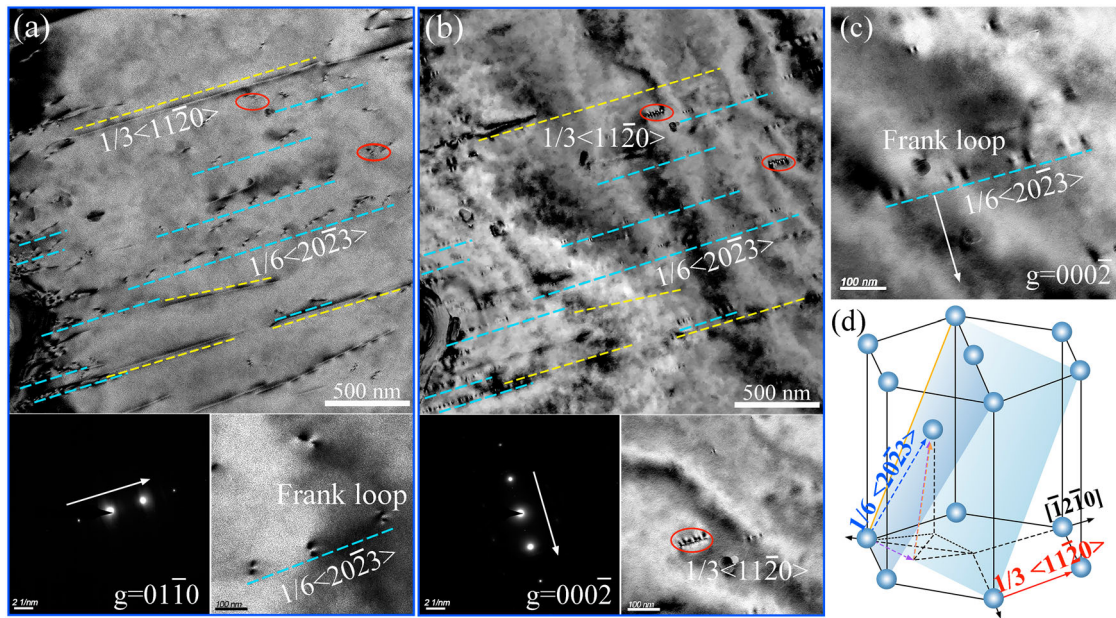


Figure 4. The Two-beam BF-TEM of the solid solution AZ63M alloy, $z = [2\bar{1}\bar{1}0]$: (a) $g = [0110]$, strip-like defects (yellow dashed line) and loop-like defects (blue dashed line) are visible; $g = [01\bar{1}0]$, strip-like defects (yellow dashed line), loop-like defects (blue dashed line) and rod-like defects (red loops) are visible; (c) Partial enlargement of Figure b, loop-like defects present in an orderly arrangement; (d) Schematic diagram showing different dislocation Burgers vectors in Mg lattice.

of the AZ63M alloy is exhibited by double-beam TEM ($z = [2\bar{1}\bar{1}0]$). According to the extinction rule of dislocations and research on the relevant literature [10,29], there are three types of dislocations in the solutionized AZ63M alloy. Strip-like dislocation (yellow dashed line in Figure 4(a)) can be confirmed as $\langle a \rangle$ perfect dislocations with $\mathbf{b} = 1/3 \langle 11\bar{2}0 \rangle$ that slip on basal plane 0001; a few rod-like dislocations (red loops in Figure 4(b)) can be confirmed as $\langle a \rangle$ perfect dislocations with $\mathbf{b} = 1/3 \langle 11\bar{2}0 \rangle$ that slip on prism plane $10\bar{1}0$; dislocation loops aligned in rows (blue dashed line in Figure 4(a,b)) can be confirmed as Frank partial dislocations with $\mathbf{b} = 1/6 \langle 20\bar{2}3 \rangle$ that slip on pyramidal plane $0\bar{1}11$. Excess point defect (vacancy/ interstitial) promoted $\langle c+a \rangle$ dislocation dissociation and formed new loops. The formation of the Frank loops indicates that there are vacancy clusters in the pyramidal plane $0\bar{1}11_{\text{Mg}}$. Vacancy clusters can capture partial solute atoms to form solute-vacancy clusters in $0\bar{1}11_{\text{Mg}}$. In addition, the β -prismatic precipitates in as-aged AZ63M alloy also are aligned in rows. Based on the above analysis, it is thought that the solute-vacancy clusters would be the nucleation sites of the β -prismatic precipitates, however, this needs to be further confirmed in future works.

In addition, in the solute-vacancy interactions, solute atoms can accelerate the diffusion of mono-vacancy and suppress the diffusion of vacancy clusters; vacancy

clusters can absorb more solute atoms to form solute-vacancy clusters. The distribution of precipitates is a more effective means of determining solute concentration. For the as-aged AZ63M alloy, the incomplete growth of the DP region indicates the transfer of solute atoms previously enriched at partial grain boundaries to the interior of the grains; the increase of solute concentration will accelerate the diffusion of vacancies along the pyramidal or prismatic plane to form vacancy clusters (indirect characterisation of Frank loops in Figure 4); vacancy clusters capture partial solute atoms to form solute-vacancy clusters; solute-vacancy clusters will be as the nucleation sites of β -prismatic precipitates. For the cast Mg alloys, the concentration of solute atoms plays a crucial role in the formation of solute-vacancy clusters. Simply controlling the mass fraction of elements cannot truly increase the average concentration of solute atoms in the matrix due to the severe element segregation phenomenon. On the contrary, it will only cause serious solute loss and significant growth of DP under segregation conditions. Based on the above analysis, it is thought that solute homogenisation could never be achieved through heat treatment because it is difficult to achieve a balance between solute diffusion and grain boundary growth. Therefore, optimising the process to achieve dispersion distribution of the second phase rich in solute elements in Mg-Al-based alloys can greatly solve this problem.

5. Summary

A high strength and toughness AZ63 cast magnesium alloy with the ultimate tensile strength, yield strength and elongation at room temperature, 319 MPa, 160 MPa and 7.6%, has been developed by combining the alternating-temperature ultrasonic melt treatment and T6 heat treatment. The high as-aged tensile properties of the AZ63 alloy are mainly attributed to the remarkable increase of the β -prismatic precipitates with orientation relationships (ORs) of $(\bar{3}30)_{\beta} // (1\bar{1}00)_{\alpha}$, $[\bar{1}11]_{\beta} // [11\bar{2}0]_{\alpha}$, thereinto the mentioned-increase is possibly related to that the solute-vacancy clusters along $0\bar{1}11$ plane might serve as the nucleation points of the β -prismatic precipitates. Obviously, the ATUMT might be used to increase the density number of the β -prismatic precipitates and then improve the mechanical properties for Mg-Al-based cast alloys.

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Data availability statement

The data that support the findings of this study are available from the authors upon reasonable request.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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