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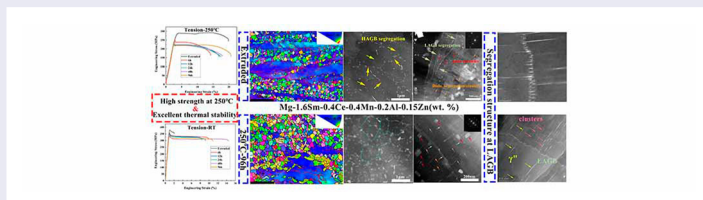
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ABSTRACT

A new Mg–Sm–Ce–Mn-based alloying system with a large miscibility gap was uncovered, and multi-solute co-segregations at high-angular grain boundaries (HAGBs), dislocations and low-angular grain boundaries (LAGBs) are obtained after the proper extrusion process. During following long-term thermal exposure, the solute along HAGBs gradually evolves into nano-precipitates, while the precipitation process along LAGBs is obviously suppressed due to the intrinsically low-energy character, and numerous G. P. zones and γ'' phases are formed in deformed regions, which always keep the limited size and reticular distribution. Consequently, the bimodal-grain structure is thermally stable, and the excellent high-temperature strength can be well maintained.



IMPACT STATEMENT

A new strategy of constructing a stable solute-network to achieve outstanding heat-resistant properties can be realized in low-alloyed Mg wrought alloy, and high-temperature UTS (~ 291 MPa) can even exceed conventional Mg–RE alloys.

ARTICLE HISTORY

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KEYWORDS

Mg wrought alloy; heat resistance; segregation; precipitation; strengthening

1. Introduction

The development of high-strength light metals including aluminum (Al) and magnesium (Mg) alloys has received great attention due to the energy crisis and demand for weight-saving [1–6]. More importantly, several application fields, such as the powertrain components in transportation, require the component exhibiting outstanding heat-resistant properties at the high servicing temperature of 200–300°C. However, it is yet a great challenge to achieve high strength in light metals at elevated temperatures.

The high strength in Al alloys is usually realized by inducing high-density nano-precipitations [7], while the phases are prone to coarsen at temperatures above 200°C.

Consequently, the strength of Al alloys would dramatically deteriorate with temperature increasing. For example, the ultimate tensile strength (UTS) of a typical 7075 alloy at 200 and 300°C would lose 70% and 90% of the value at room temperature (RT). The yield strength (YS) of conventional heat-resistant 2xxx alloys would also decrease by more than 50% during thermal exposure around 250 for 10 h, accounting for the dissolution and coarsening of strengthening phases [8].

For Mg wrought alloys, the quick diffusion of atoms at elevated temperature could also result in the abnormal growth of strengthening phases and grain boundary sliding, which thus lead to the poor high-temperature (HT) strength, usually less than 100 MPa at 250°C, e.g.

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Mg–6Zn–1Mn [9], Mg–6Al–3Sn [10], Mg–3Al–1Zn [11] alloys. Besides, the unstable grain boundary structure could be another important mechanism for the poor thermal stability of Mg alloy. As a result, the normal routine to improve the high-temperature strength in Mg alloy is to add large amounts of rare-earth elements (RE), usually > 10 wt.%, and the high UTS up to ~ 300 MPa at 250°C can be realized [12,13]. However, the developed Mg-RE-based alloys face the limitation of economic effectiveness, and the coarsening of second phases can yet occur during long-term thermal exposure. In contrast, the grain refinement would be more effective in enhancing yield strength as compared with precipitation hardening in Mg alloy at RT [14], and excellent HT strength can be expected if the grain growth and grain boundary sliding at elevated temperatures can be largely suppressed.

Introducing segregation to grain boundaries (GBs) has been proven to be effective in restricting the growth of ultrafine grains in Al and Mg alloys [15,16], while there is still a lack of research on achieving the comprehensive heat-resistant properties, including excellent thermal stability of microstructure and HT strength. In this work, a novel strategy of constructing a solute-network along low-angular grain boundary (LAGB) and dislocations to achieve stable bimodal-grain structure and thus outstanding heat-resistant property in a new Mg–Sm–Ce–Mn-based alloy system has been realized. Pandat software predicted that the large miscibility gap (Ce: 0~1 wt.%, Sm: 0~4 wt.%, at temperature of 530°C) exists in this alloying system, and the composition of Mg–1.6Sm–0.4Ce–0.4Mn–0.2Al–0.15Zn (wt.%) was designed according to the phase diagram shown in Figure 1(a), in which minor Al and Zn atoms are added to promote more pronounced solute segregations.

2. Experimental procedure

The as-cast Mg ingots are prepared by melting and homogenized at 530°C for 12 h and then indirectly extruded at 300°C with an extrusion ratio of 23:1 and an extrusion speed of 0.35 mm/s. The as-extruded samples were held at 250°C for 6, 12, 24, 48, and 96 h. Mechanical properties are measured along the extrusion direction (ED) at RT and 250°C with an initial strain rate of 0.001 s^{-1} . Before each HT tensile test, 10 min holding was applied to homogenize the temperature of test samples. Microstructures are characterized using scanning electron microscopy (SEM, JEOL JSM-7001F), electron backscatter diffraction (EBSD), transmission electron microscopy (TEM, JEOL JEM-2100F) and also aberration-corrected scanning transmission electron microscope (STEM, JEM-ARM200F)

under high-angle annular dark-field (HAADF) STEM mode. The misorientation angles between the adjacent grains are used to identify the low-angular grain boundary (LAGB, $2\sim 15^\circ$) and high-angular grain boundary (HAGB > 15°), as indicated by red and black lines, respectively.

3. Results and discussion

The SEM image in Figure 1(b) shows that only limited second phases with micron-meter sizes are precipitated after extrusion of the present Mg sample, indicating that the added Ce and Sm atoms have been mostly dissolved into the Mg matrix during the homogenization process, and the rationality of compositional design above is further confirmed. The RT tensile stress–strain curves of present Mg samples are presented in Figure 1(c), and the YS and UTS of the as-extruded sample are ~ 372 MPa and ~ 379 MPa, while the elongation (EL) is only $\sim 1.2\%$. With heating at 250°C for 6 h, the YS and UTS are declined to ~ 351 MPa and ~ 359 MPa, while the EL is obviously enhanced to $\sim 9\%$. Interestingly, the strength of the present Mg alloy can be maintained after long-term thermal exposure, and the YS and UTS are yet as high as ~ 323 MPa and ~ 331 MPa after heating for 96 h. As shown in Figure 1(e), the thermal stability of the present low-alloyed Mg–Ce–Sm–Mn–Al–Zn alloy is superior to the most reported Mg alloys as well as the 2xxx Al alloys [8,17,18], with a strength–loss ratio less than 12% within 96 h. The HT tensile strength at 250°C is exhibited in Figure 1(d), where the YS and UTS of the as-extruded Mg samples are ~ 275 MPa and ~ 291 MPa, respectively. The HT strength becomes more stable as the duration time exceeds 6 h, and YS and UTS of the 96-h-aged Mg sample can still reach ~ 225 MPa and ~ 237 MPa, revealing excellent thermal stability. Furthermore, the strength and alloying contents of Mg and/or Al alloys are compared in Figure 1(f), which shows that strengths of present Mg alloys with a tiny amount of solute addition are even comparable with those of Mg/Al alloys containing more than 10 wt. % solutes.

EBSD images in Figure 2 show that the as-extruded Mg sample exhibits a bimodal-grain microstructure, which consists of ultrafine dynamic recrystallized (DRXed) grains with random orientation and the un-DRXed regions having a typical fiber texture of $\langle 10\text{--}10 \rangle // \text{ED}$, and the modest texture intensity of ~ 7.55 multiples of the random distribution (mrd.) can be detected. As thermal exposure time reaches 6 and 96 h, as shown in Figure 2(b,c), the bimodal-grain structure is maintained, but with fewer un-DRXed regions, and texture intensity is slightly reduced to 6.39 and 5.53 mrd., indicating the outstanding thermal stability of microstructure. The average

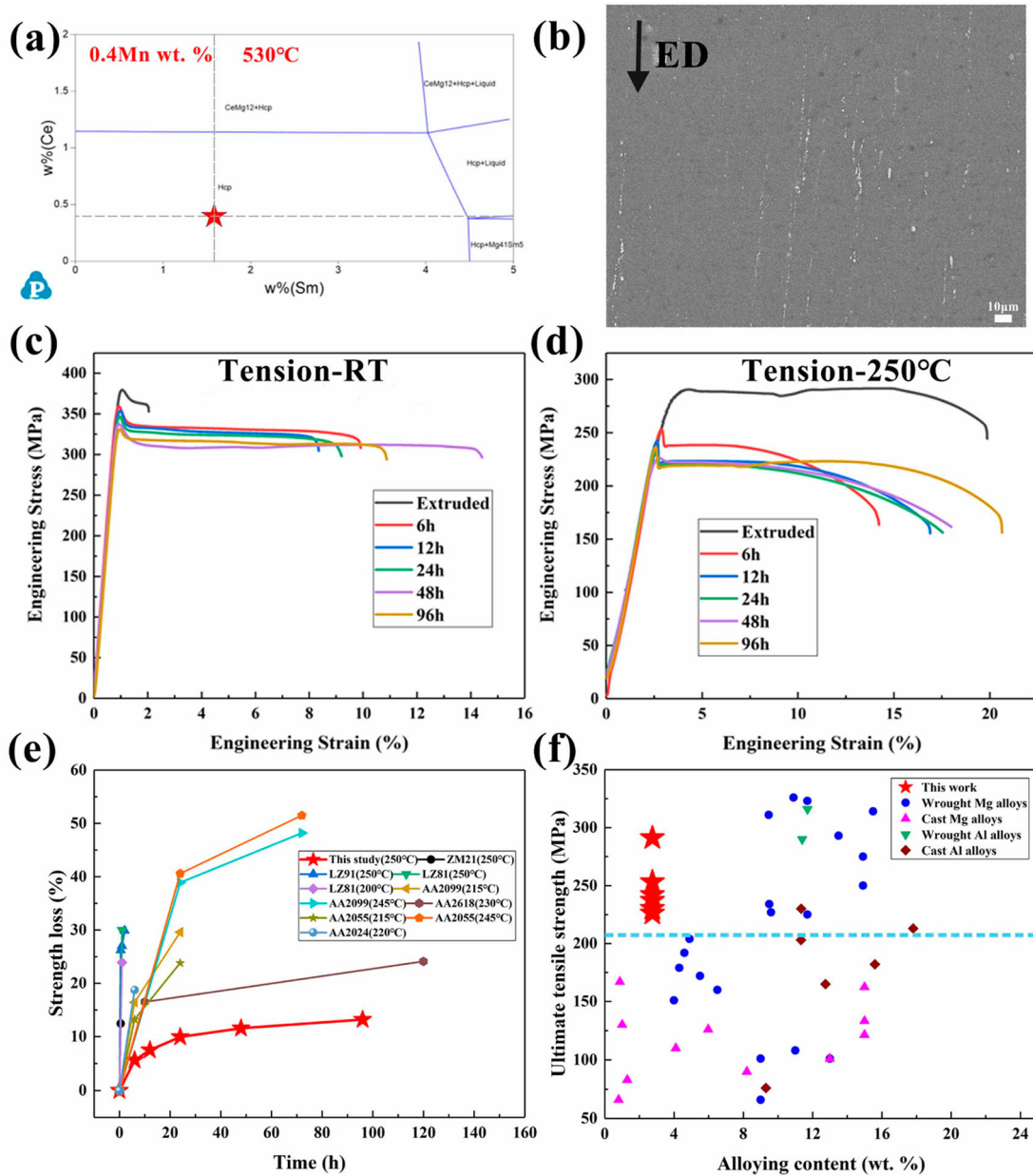


Figure 1. (a) Calculated isothermal diagram of Mg-xSm-yCe-0.4Mn wt.% alloy system; (b) the SEM image of the as-extruded Mg-1.6Sm-0.4Ce-0.4Mn-0.2Al-0.15Zn sample; (c) and (d) RT and HT tensile curves of present Mg alloys after different thermal exposure time at 250°C, respectively; (e) comparison of RT strength loss of various Mg alloys and Al alloys during thermal exposure at 200–250°C [8, 17, 18]; and (f) comparison of UTS and alloying content of various Mg and Al alloys at 250°C [9,10,12,13,19,20,21].

DRXed grain size only exhibits certain growth, from $\sim 0.83 \mu\text{m}$ in the as-extruded sample to $\sim 1.08 \mu\text{m}$ in the 96-h-aged sample. Geometrically necessary dislocations (GNDs) density can be evaluated from kernel average misorientation (KAM) map (Figure 2(d–f)), based on the formula of $\rho_{\text{GND}} = 2\Theta/x_b$ [22], where x is the unit length which is equal to twice of step size (μ) used in EBSD scanning; b is the magnitude of Burgers vector ($|b| = 0.32 \text{ nm}$); and Θ is evaluated from the local misorientation profile in the KAM map of the EBSD data. The

calculation results suggest that GNDs density decreases slightly from $\sim 3.7 \times 10^{14}/\text{m}^2$ in the as-extruded sample to $\sim 3.2 \times 10^{14}/\text{m}^2$ after exposure for 6 h (Figure 2(e,h)), and there is even no evidence of change after further thermally holding for 96 h, with the number density of $\sim 2.9 \times 10^{14}/\text{m}^2$ (Figure 2(f,i)).

Figure 3(a–c) display typical HAADF-STEM images of as-extruded Mg samples, and the size of DRXed grains ranges 0.7–1.0 μm . Bright contrast can be found at the HAGBs in DRXed regions, Figure 3(a), indicating

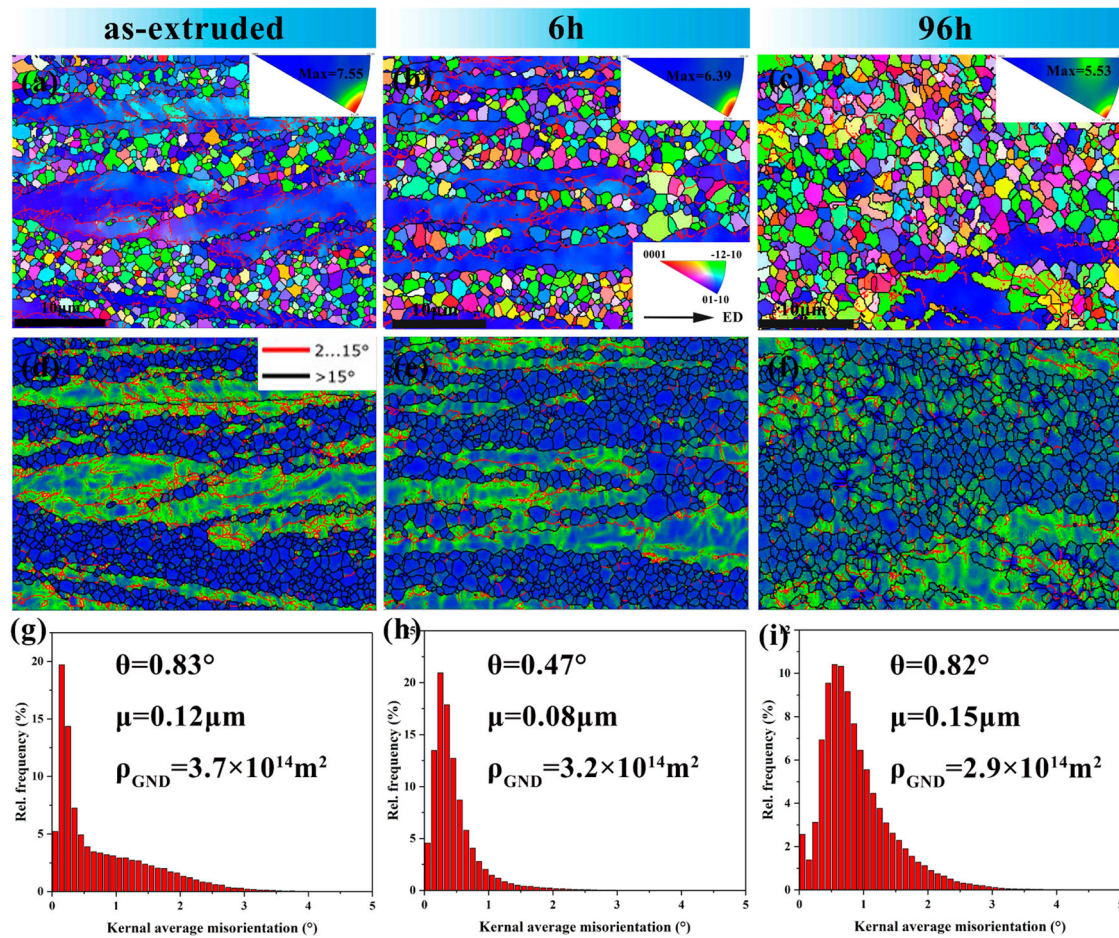


Figure 2. (a–c) Orientation distribution maps and inverse pole figures; (d–f) KAM maps; and (g–i) distribution of KAM value for (a, d, g) as-extruded sample, (b, e, h) thermal exposure for 6 h, and (c, f, i) 96 h.

the obvious solute segregation and the dynamic precipitations, both of which would contribute to restrict the migration of HAGBs. Importantly, solute segregation also occurs along both the LAGBs and residual dislocations in un-DRXed regions, and corresponding EDS mapping confirms the multi-solute co-segregation of Sm, Zn, Ce, Mn, and Al at LAGBs (Figure 3(b,c)). For 6h-aged Mg sample, the solute segregation along HAGBs is largely weakened, and some solutes have evolved to be nano-precipitations, and the DRXed grain size is yet less than $1 \mu\text{m}$ (Figure 3(d)). In contrast, the solute segregation networks along LAGBs and dislocations are well remained, accompanied by some nanoparticles (Figure 3(e,f)). With prolonging the exposure time to 48 h, Figure 3(g–i), numerous second phases become forming both along HAGBs and inside DRXed grain, and grain size has increased to $\sim 1 \mu\text{m}$. A complex substructure is formed within un-DRXed region, and solute segregations along LAGB/dislocation are dominant, (Figure 3(h,i)). After exposure for 96 h (Figures 3(j–l)), the size of nano-precipitation does not

increase much, and DRXed grain size is also less than $\sim 1.2 \mu\text{m}$. Along the LAGBs, the solute segregation can be easily detected, and some plate-like nanophases are gradually formed, along with the bulk nanoparticles and high-density residual dislocations.

To further reveal the mechanism for a highly stable bimodal-grain structure, an enlarged image for a 96-h-aged sample nearby the LAGB region is shown in Figure 4(a), with an incident beam of $B = < 10^{-10} >$. Corresponding EDS mapping in Figure 4(d) confirms that considerable solute Sm, Zn, and Mn atoms are enriched at the LAGBs. Besides, some plate-like nanophases are formed either at the LAGB or at grain interiors (Figure 4(a,d)), which would be further illustrated in the following section. Some other LAGBs are composed of serrated lines, which should be the residual dislocations and are decorated with solute segregations (Figure 4(b,c)). According to the morphology, the long-straight line defects lying on the basal plane belong to the dissociated $< c + a >$ dislocations [23], and they are connected by the mixed/screw typed $< c + a >$ ones.

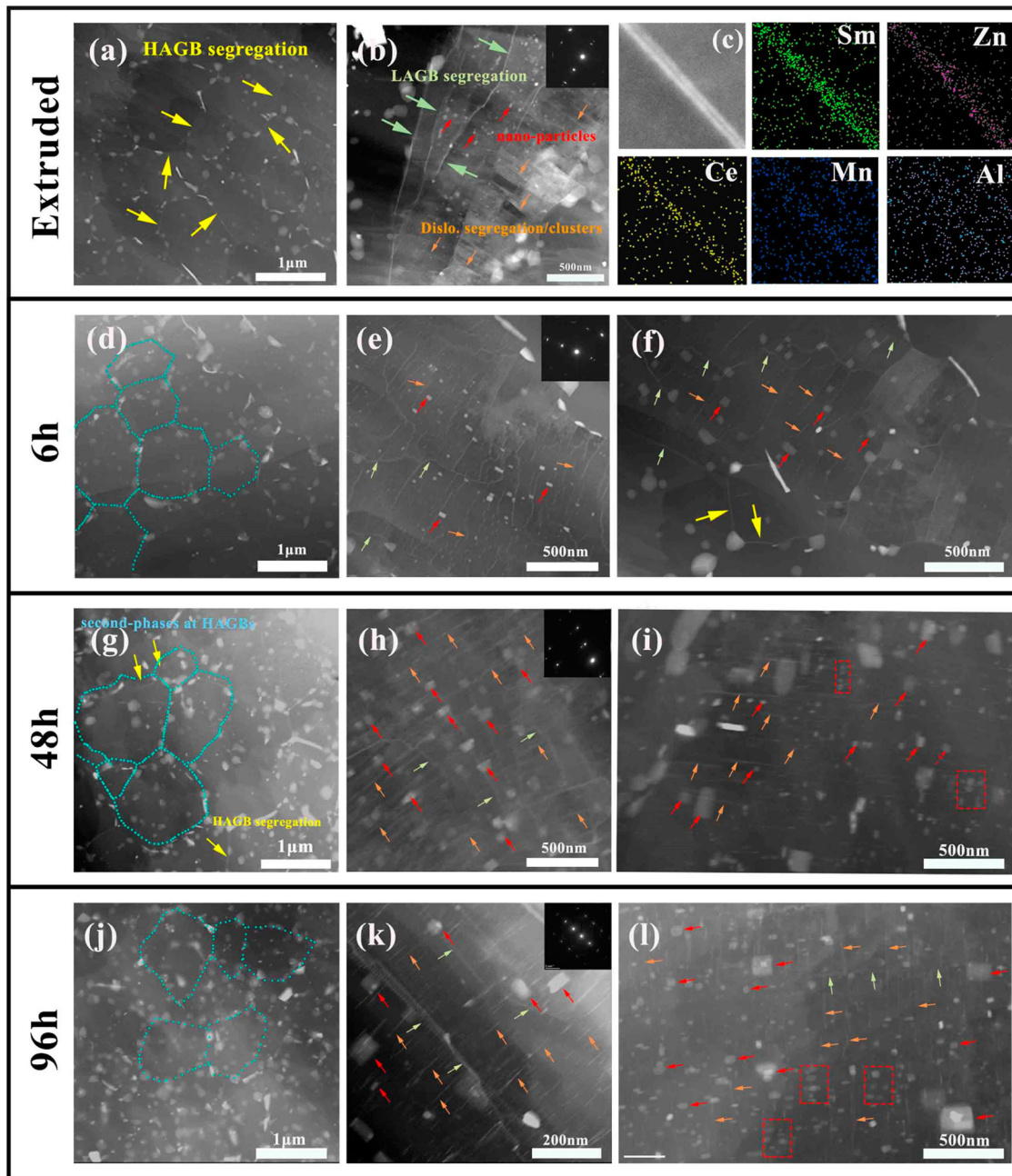


Figure 3. HAADF-STEM images of (a–c) as-extruded, and (d–f) 6 h, (g–i) 48- and 96-h heated Mg samples. (a, d, g, j) DRXed region with segregation at HAGBs (yellow arrows) and second phases, (b, e, f, h, i, k, l) un-DRXed regions with segregation at LAGBs (green arrows) and dislocations, linear clusters (orange arrows) and nanoparticles (red arrows), and (c) EDS mapping at LAGBs.

Similar dislocation constructions have also been reported in the Mg–Ca- and Mg–Ce- based alloys [22,24]. The enlarged TEM image in Figure 4(c) demonstrates that four layered segregations are formed along the long-straight lines, which correspond to the faulted atomic structure in dissociated $\langle c+a \rangle$ dislocations and are also similar to structural units (SUs) of LPSO phases in Mg–Al–RE alloys [25].

Figure 5 shows the typical HAADF-STEM images for the 96-h-aged sample, and numerous plate-like

nanophases are distributed nearby LAGBs. Enlarged TEM images in Figure 5(b,c) show that the phases involve the γ'' phases with a height of 5 atomic layers, and the transition region of G.P. zones, which are spanned 3–6 atomic layers and exhibit the h.c.p. structure. That is, the γ'' phases should have been transformed from G.P. zones. EDS mapping shows the plate-like nanophase is enriched with multi-elements of Sm, Mn, Zn, Al, and Ce (Figure 5(d)). Figure 5(e,f) displays the STEM/EDS mapping for the bulk nanophases in the 96-h-aged sample,

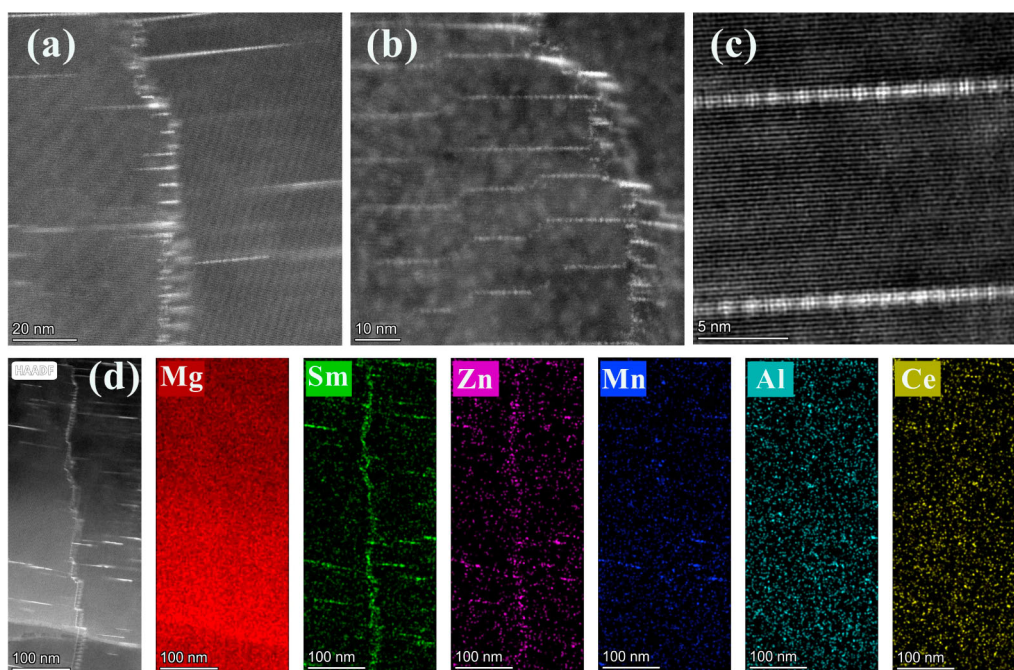


Figure 4. HAADF-STEM images at LAGBs in 9-6h-exposed sample: (a) high-density ultrashort linear clusters around LAGB, (b) long-straight dislocations involved segregation, (c) multiple SUs component, and (d) EDS mapping of the elemental distributions at LAGBs.

which are enriched with Ce and Sm atoms, as well as several nano-Mn particles.

As described earlier, a low-alloyed multicomponent heat-resistant Mg alloy with excellent thermal stability has been achieved through constructing co-segregation networks at linear/planar defects. During the initial 6 h heating, dislocation density decreases slightly from $\sim 3.7 \times 10^{14}/\text{m}^2$ to $\sim 3.2 \times 10^{14}/\text{m}^2$, which corresponds to the static recovery. In the following heating process, the segregation along both LAGBs and dislocation lines is thermally stable, which can be well maintained even after long-term thermal exposure to 96 h. Previous works have shown that the nano-precipitations along grain boundaries strongly depend on the type of GB [26,27]. For example, the β -phase precipitation along LAGB in Al–Mg alloy can be largely suppressed due to the low interfacial energy and low diffusion coefficient characters [26]. In the present Mg alloy, a similar precipitation behavior has been captured, and the introduction of a high fraction of LAGB can effectively evade the formation of nano-precipitations, and the solute atoms always exist as stable networks along the dislocation walls. In contrast, the solute precipitation along HAGB is relatively quick, and certain amounts of nanophases have been formed in the 6-h-aged sample, and abundant nanophase both along GB and in grain interiors are also generated in the 96-h-aged sample (Figure 3). It is well known that the excess energy of the HAGB is much higher than that of LAGB [28]. Consequently, HAGBs are easier to act

as nucleation sites for irregularly shaped second phases. Despite that, the size of nano-precipitation is always stable, less than 200 nm, which should be correlated to the low diffusion coefficient of added solute atoms at 250°C, Ce: $4.3 \times 10^{-17} \text{ m}^2/\text{s}$, Sm: $2.4 \times 10^{-17} \text{ m}^2/\text{s}$, Mn: $6.0 \times 10^{-20} \text{ m}^2/\text{s}$, and Mg: $8.0 \times 10^{-18} \text{ m}^2/\text{s}$ [29]. The stable nano-precipitations can pin the migration of HAGBs, and the average grain size of DRXed grains in 96-h-aged sample is always less than 1.2 μm .

Besides, as compared with single-type solute addition, the multi-solute co-segregation at linear/planar defects would be more stable in total interfacial energy, further improving the thermal stability. For example, the atomic size of Ce (0.182 nm) and Sm (0.180 nm) is larger than that of the Mg matrix (0.160 nm), while the size of Al (0.143 nm), Mn (0.132 nm), and Zn (0.139 nm) atoms are less than Mg. As a result, the large elastic strain can be more readily relaxed when the solute atoms above are added simultaneously. The enrichment of Ce, Sm, Mn, Al, and Zn atoms along the LAGBs of the as-extruded Mg sample also provides direct evidence (Figure 3). Finally, the solute segregation along defects can stabilize the deformation substructure, since the mobility of LAGBs and dislocations can be largely suppressed. In this sense, considerable residual dislocations can be retained in the 96-h-aged sample, and also more atoms have diffused into the residual dislocations adjacent to the LAGBs (Figure 4). Combined with the low-energy character of LAGBs, the static recrystallization behavior of the present

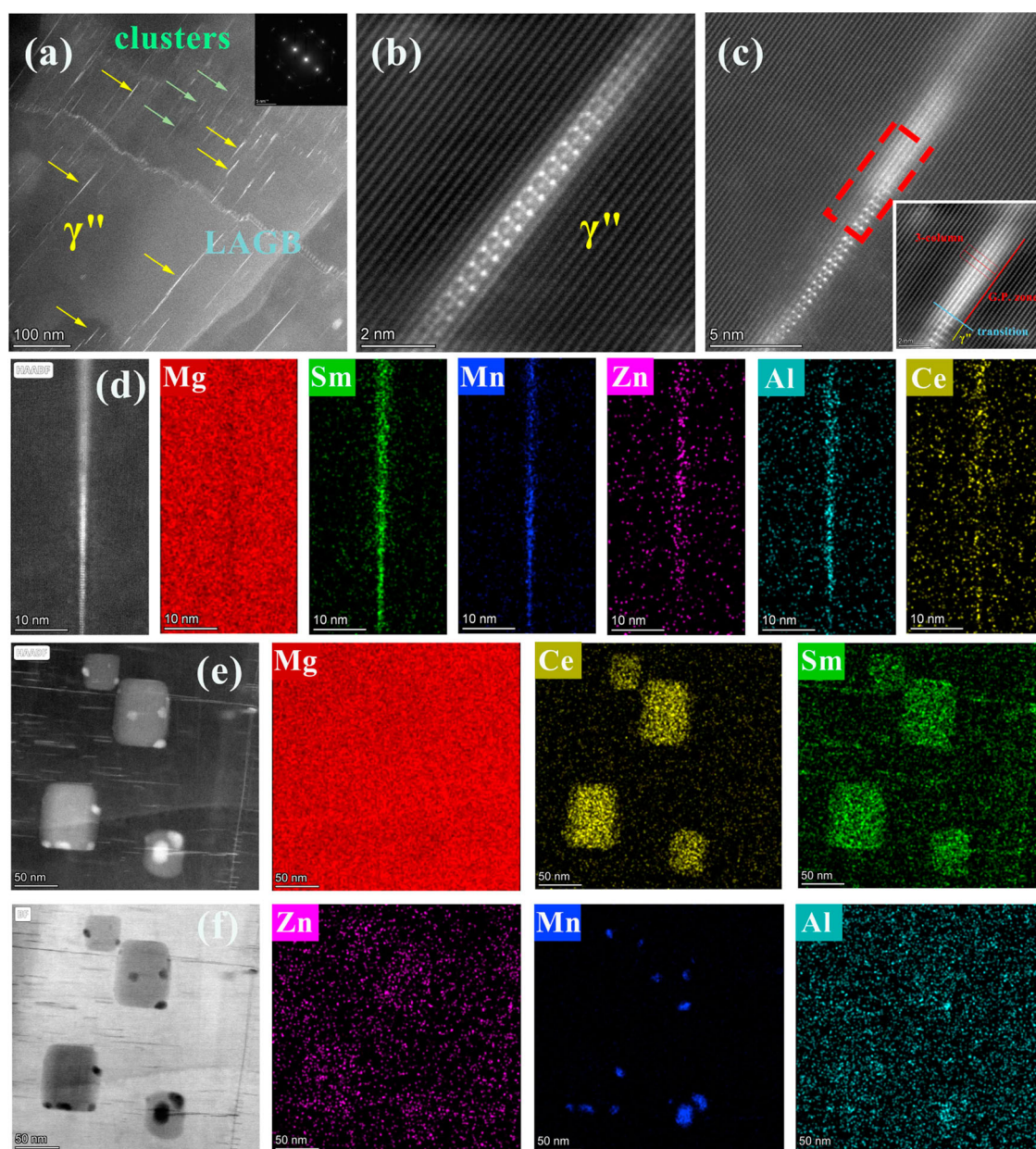


Figure 5. HAADF-STEM images of (a) the region around LAGB, (b) γ'' phase, and (c) transition of G.P. zone and γ'' . Corresponding EDS mapping for (d) linear clusters containing G.P. zone and γ'' phase and (e, f) bulk nanophases.

Mg alloy is significantly retarded, and the bimodal-grain structure can remain.

For the present Mg alloys, fine DRXed grains and sub-grain lamellae together contribute to the high strength of ~ 372 MPa at RT, by following the Hall–Petch relationship. The relatively high dislocation density leads to the poor EL of $\sim 1.2\%$ in the as-extrude sample, which is regained after the following short-time heating, $\sim 9\%$ in the 6h-aged sample. Dislocation evolution could be the possible reason. For the as-extruded sample, the moving dislocations are prone to evolve into sessile ones before reaching LAGBs during the RT tensile test, because of the high density of residual dislocations,

which leads to low ductility. However, there will be fewer immobile-type dislocations after thermal exposure, which is thus beneficial for ductility. Moreover, the stable finely DRXed grains/sub-grain structure and unchanged fiber texture together contribute to the high strength of as-aged Mg samples at RT. At high temperatures, on the other hand, the construction of solute segregation along GBs/dislocations in wrought Mg alloy could be one of the most effective routes to improve HT strength due to the strong dragging effect on GB sliding. As a result, the high YS of ~ 275 MPa at 250°C is obtained in as-extruded Mg samples. Moreover, the tensile curves at HT also exhibit a yield drop phenomenon instead of work

hardening, which further demonstrates the suppression of GB sliding. The critical resolved shear stress for non-basal dislocation slip can be significantly decreased with increasing temperature, and the ductility at HT can be thus much enhanced.

After short-time aging, the YS of the 6-h-aged sample at 250°C is decreased to ~ 225 MPa, and the weakened segregation along HAGBs at this stage could be one important reason since some HAGB sliding becomes possible under this circumstance. Otherwise, the precipitations of the Mg-RE phase within DRXed regions introduce incoherent interfaces, which would be detrimental to ductility. Consequently, the elongation of 6-h-aged is decreased, and a balance between GB sliding and incoherent interphase should exist in controlling the total elongations of present Mg alloys at HT. During the following heating as long as 96 h, the Mg sample exhibits a thermally stable microstructure and LAGBs are always pinned by solute atoms. Consequently, the LAGB sliding behaviors are largely suppressed, and the high YS of ~ 220 MPa in the 96-h-aged sample at 250°C can be obtained. Besides, the thermally stable plate-like nanophase (G.P. zone, γ'') and SUs at dislocations can also act as effective obstacles to impede dislocation movement at high temperatures, which can compensate for the strength loss from partial dislocations recovery and solute precipitation at HAGBs. Consequently, the multi-solute segregation networks, including segregation at GBs and dislocations, and nanoparticles/clusters could collectively contribute to improving the strength of present Mg alloy at high temperatures.

4. Conclusions

In summary, a low-alloyed multicomponent heat-resistant Mg alloy can be achieved through a traditional extrusion process, with outstanding thermal stability and strength at 250°C, superior to common heat-resistant Al alloys and costly high-RE Mg alloys. The excellent HT strength of the as-extruded sample is mainly attributed to the segregation networks at HAGBs, LAGBs, and dislocations, which could significantly increase the resistance to dislocation slip and GB sliding. During long-term thermal exposure, more second phases could precipitate at the HAGBs, while the segregation at dislocations and dislocation-typed LAGBs could be retained, resulting in stable mechanical properties at both RT and HT. This study provides a profound insight into constructing a multi-solute co-segregation network at dislocation and low-energy GBs, which is important for the application of heat-resistant Mg alloys under long-term thermal exposure.

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Disclosure statement

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

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