

Phase Spheroidization and Solid-Solution Strengthening of Extruded Mg-Si-Zn-Ca-Y Alloy Induced by Low-Temperature Annealing

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Abstract

Microstructural evolution and mechanical properties of as-extruded Mg-2.5Si-4Zn-0.7Ca-1Y alloys were characterized under diverse heat treatment conditions. An optimal regime of 200 °C for 12 h emerged, delivering a peak Vickers hardness of ~100 HV0.2. After heat treatment, substantial dissolution of Zn into the α -Mg matrix occurred and Ca/Y-rich intermetallics underwent spheroidization. The microstructural changes facilitated the improvement of ultimate tensile strength (UTS) at 200 °C from ~160 MPa to ~200 MPa, while the elongation decreased from ~18% to ~6%. The enhanced high-temperature strength derives from Zn solid-solution strengthening and spheroidization of the Ca/Y-rich network phase, with a possible additional contribution from Y redistribution near the Mg₂Si-containing regions.

Keywords: Mg-Si-Zn-Ca-Y alloy; extrusion; heat treatment; microstructure; mechanical properties

1. Introduction

Magnesium alloys are suitable for lightweight structural applications due to their low density and high specific stiffness [1]. But common Mg-Al series alloys exhibit softening at elevated temperatures [2]. Development of heat-resistant alternatives has attracted increasing interest. Mg-Zn and Mg-RE alloys enhance high-temperature performance through stable phases like Ca₂Mg₆Zn₃ or long-period stacking ordered (LPSO) [3–5]. Significant cost and density increases occur with these methods.

Low-cost heat-resistant Mg-Si alloys are promising, but coarse as-cast Mg₂Si limits performance [6]. While alloying [7] and severe plastic deformation techniques such as friction stir processing [8] have been shown to refine Mg₂Si, hot extrusion remains the most industrially relevant route but introduces new issues. During extrusion, Mg₂Si fragments into small pieces with sharp shapes and the low-modulus MgZn phase deforms into continuous bands, severely cleaving the matrix and deteriorating properties [9,10]. These Mg₂Si particles can cut the matrix and such continuous MgZn bands serve as preferential crack propagation paths under tensile loading, which can compromise the alloy's ductility. Our previous studies have progressively optimized the composition of this alloy system: 0.7 wt.% Ca was identified as the optimal content under both friction stir processing [8] and hot extrusion [10] routes for eutectic-network refinement, and 1 wt.% Y was subsequently established as the optimal rare-earth addition on top of the 0.7Ca baseline in the extruded state [11]. Building on this fully optimized Mg-2.5Si-4Zn-0.7Ca-1Y composition,



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the present work shifts the focus from composition design to post-extrusion processing, and for the first time systematically investigates the effect of low-temperature annealing on the microstructural evolution and elevated-temperature performance of this alloy—a processing stage that has not been addressed in our previous work or in the broader Mg–Si–Zn literature. Furthermore, our previous research [10] also demonstrated that although extrusion effectively fragments the coarse Mg₂Si phase, certain Mg₂Si particles with sharp edges still persist in the as-extruded matrix, acting as stress concentrators that initiate microcracks. Therefore, a subsequent heat treatment is considered necessary to further spheroidize these sharp-edged Mg₂Si particles and dissolve the detrimental banded MgZn phase for Zn solid-solution strengthening. However, conventional high-temperature solid-solution treatments often induce severe grain coarsening, while systematic studies on low-temperature post-extrusion annealing of Mg–Si–Zn–Ca–Y alloys remain scarce. It should be noted that annealing-induced recrystallization and texture evolution in Mg alloys are highly temperature- and time-dependent. For example, Sheng et al. reported that annealing an extruded Mg–Zn–Y–Nd alloy at 200 °C produced only limited changes in grain morphology and retained a strong extrusion-related fiber texture, whereas annealing at 225–250 °C promoted static recrystallization and obvious texture weakening [12]. Similarly, Wang et al. showed that in a low-temperature extruded Mg–Zn–Ca alloy, grain growth and texture transformation became pronounced mainly under higher-temperature or long-term annealing conditions, especially after annealing at 300 °C for 10 h [13]. Recent modeling work on Mg–Zn–Ca alloys further demonstrated that static recrystallization kinetics are governed by stored energy, grain-boundary mobility, and solute drag, with low-temperature annealing being particularly sensitive to solute-controlled boundary migration [14]. In Mg–Zn–Y–Ca and Mg–Zn–Ca sheets, annealing at 300–360 °C has also been shown to regulate texture, precipitation, and mechanical properties through recrystallization, solute segregation, and particle pinning [15,16]. These studies indicate that pronounced texture modification generally requires higher annealing temperatures, longer holding times, or severe deformation storage energy. Therefore, under the present low-temperature condition of 200 °C, the dominant microstructural response is expected to be phase dissolution, solute redistribution, and second-phase morphology modification rather than extensive texture reorientation. To address both the need for post-extrusion microstructural optimization and the risk of grain coarsening during high-temperature treatment, a low-temperature annealing treatment was applied to the as-extruded Mg-2.5Si-4Zn-0.7Ca-1Y alloy in the present work, with the aim of elucidating the effects of heat treatment on microstructural evolution and mechanical properties.

2. Materials and Methods

Mg-2.5Si-4Zn-0.7Ca-1Y alloy ingots were prepared by melting pure magnesium, pure zinc, Mg-5wt.%Si, Mg-20wt.%Ca, and Mg-30wt.%Y master alloys in an electric resistance furnace under a CO₂/SF₆ (100:1) protective atmosphere and casting in the mold of Φ50 × 100 mm. The ingots were subsequently machined into billets of Φ35 × 70 mm. The billets were extruded at 250 °C with a feed rate of 0.2 mm/s to a final diameter of 12.7 mm (designated as AE).

Heat treatment (annealing) was performed in a forced-air circulation resistance furnace. AE specimens were placed in the furnace after it had reached the target temperature, held for the prescribed time, and then air-cooled to room temperature. To determine the optimal annealing parameters, a two-stage screening was conducted: (i) temperature screening at a fixed holding time of 2 h over the range of 150–250 °C (interval 50 °C), and (ii) holding-time screening at the identified optimal temperature of 200 °C for durations of 4, 8, 12 and 16 h. At each condition, three independently prepared specimens were tested and the

mean Vickers hardness with standard deviation is reported (Figure 1). Based on these results, the optimal annealing condition was identified as 200 °C/12 h (designated as HT), which yielded the peak hardness of ~100 HV_{0.2} within the investigated processing window. For microstructural characterization of the alloy, specimens were cut using wire cutting, followed by mechanical grinding and polishing. Microstructural analysis was performed using an OLYMPUS optical microscope (OM); phase identification and chemical composition analysis were conducted using a field-emission scanning electron microscope (FE-SEM) equipped with an energy dispersive spectrometer (EDS); elemental distribution was analyzed using an electron probe microanalyzer (EPMA); and phase composition was determined via X-ray diffraction (XRD).

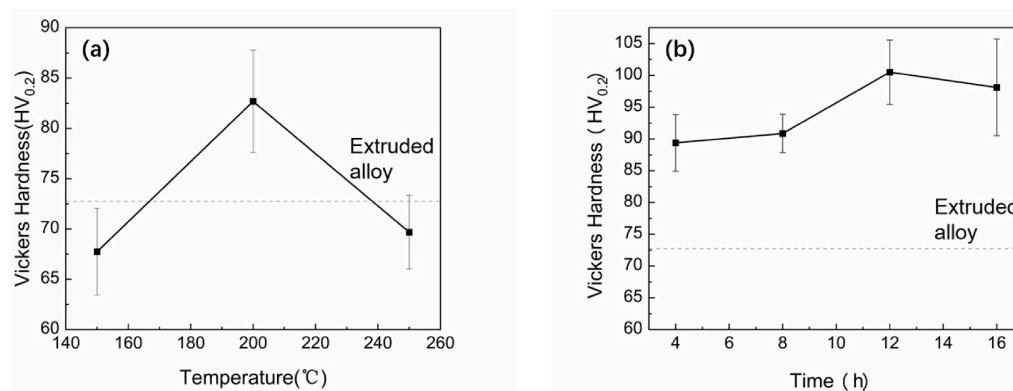


Figure 1. Vickers hardness vs. (a) temperature under the holding time of 2 h and (b) holding time under the temperature of 200 °C. Dashed line: Extruded alloy.

The mechanical properties of the alloys were evaluated using a universal testing machine (SANS-CMT5105, Shenzhen SANS Test Instrument Co., Ltd., Shenzhen, China) equipped with a resistance heating furnace for elevated-temperature tests. Tensile tests were conducted at room temperature (RT) and 200 °C; for the 200 °C tests, specimens were held at the target temperature for 5 min before loading to ensure thermal equilibrium. A minimum of three specimens were tested at each condition, and the reported tensile properties (UTS and elongation) represent the mean values; individual test results are provided in the Supplementary Table S1. Vickers hardness (HV_{0.2}) was measured on polished cross-sections using a load of 1.96 N and a dwell time of 15 s; at least five indentations per specimen were averaged, with results reported as mean ± standard deviation.

3. Results and Discussion

3.1. Microstructural Evolution

3.1.1. Phase Composition and Lattice Distortion

Phase composition and distribution in the alloy were analyzed before and after heat treatment, as shown in Figure 2, where Figure 2a,c show SEM images, Figure 2b,d show EDS results by line scans, Figure 2e show XRD patterns, and Figure 2(e1) show magnified XRD peaks of the α -Mg (10-11). From Figure 2e, the XRD pattern of the AE alloy confirms the presence of α -Mg matrix, Mg₂Si, MgZn, CaMgSi, Mg₂Y, and Y₂Si₂Mg phases. After heat treatment, the diffraction peaks of Mg₂Y are no longer identifiable, whereas the relative intensity of Y₂Si₂Mg peaks increases. Meanwhile, the peaks of MgZn are weakened, reflecting partial dissolution into the α -Mg matrix. Combined with Figure 2b,d, the phases illustrated using purple, green, blue, and sky blue rings can be identified as Y₂Si₂Mg, Mg₂Si, MgZn, and CaMgSi, respectively.

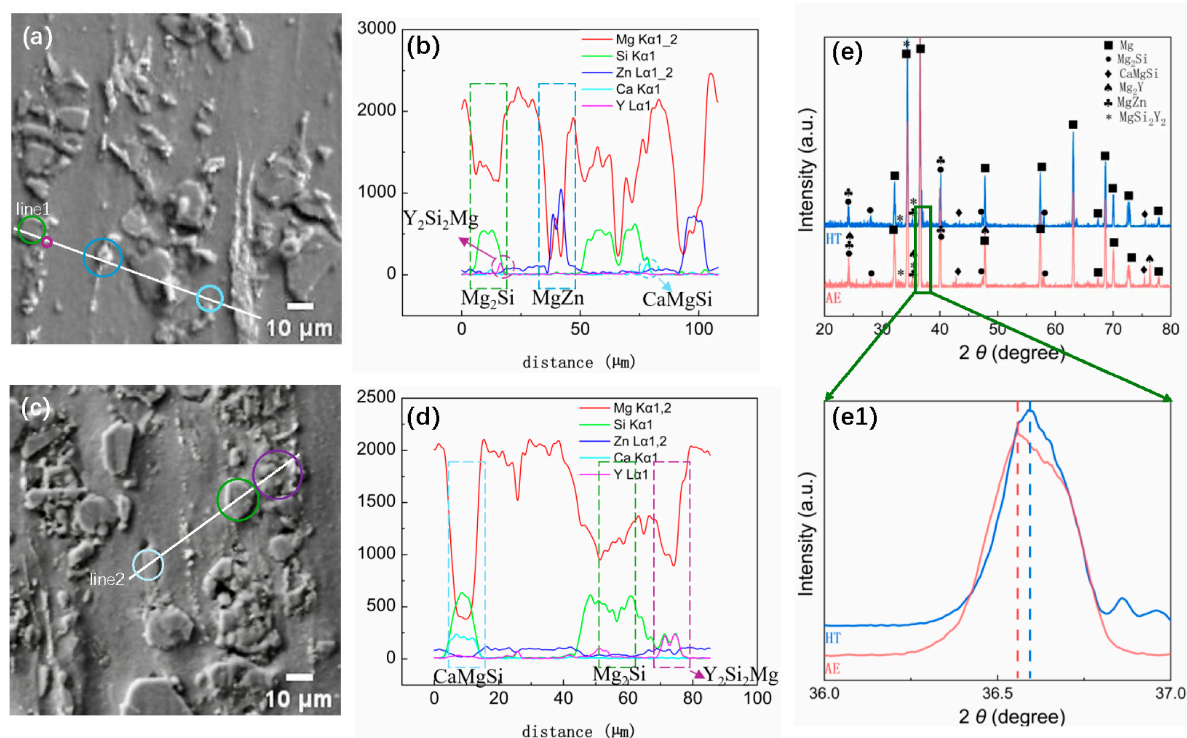


Figure 2. (a,c) SEM images; (b,d) EDS line scans; (a,b) AE alloy; (c,d) HT alloy; (e) XRD patterns; (e1) Magnified XRD peaks of the α -Mg (10-11).

A closer inspection of the (10-11) reflection in Figure 2(e1) reveals a slight shift of the diffraction peak toward higher 2θ angles after annealing, which qualitatively indicates a contraction of the α -Mg lattice and is consistent with the incorporation of solute atoms into the matrix [17]. To move beyond this qualitative observation and quantitatively assess the lattice response, Rietveld refinement of the AE and HT patterns was performed using GSAS-II (<https://advancedphotonsource.github.io/GSAS-II-tutorials/> accessed on 1 May 2026), and the refined lattice parameters of α -Mg are summarized in Table 1. Upon annealing, the basal-plane parameter contracts from $a = 3.21115(5) \text{ \AA}$ to $3.21018(5) \text{ \AA}$ ($\Delta a/a = -0.030\%$), while the c -axis remains essentially unchanged ($\Delta c/c = +0.010\%$), yielding a net cell-volume contraction of $\Delta V/V = -0.052\%$. The refinements converged with R_{wp} values of 6.62% (AE) and 6.55% (HT), confirming the reliability of the extracted parameters.

Table 1. Rietveld-refined lattice parameters of α -Mg in the AE and HT alloys.

Condition	a (Å)	c (Å)	V (Å ³)	c/a	R_{wp} (%)
AE	3.21115(5)	5.21027(4)	46.5277(7)	1.6226	6.62
HT	3.21018(5)	5.21077(4)	46.5041(8)	1.6232	6.55
Change	$\Delta a/a = -0.030\%$	$\Delta c/c = +0.010\%$	$\Delta V/V = -0.052\%$		

The direction and magnitude of this lattice contraction are consistent with Zn substitution into the Mg lattice, since the atomic radius of Zn (1.34 Å) is smaller than that of Mg (1.60 Å); the observed $\Delta a/a$ is in reasonable agreement with the Vegard-type contraction reported for dilute Mg-Zn solid solutions [18]. The pronounced anisotropy of the response, with contraction confined mainly to the basal plane while the c -axis remains nearly unchanged, likely reflects competing contributions during annealing: Zn substitution induces overall lattice contraction, while partial relaxation of extrusion-induced residual strain and possible minor incorporation of Y atoms released from Mg_2Y decomposition ($r_Y = 1.80 \text{ \AA} > r_{Mg} = 1.60 \text{ \AA}$) may partially offset the contraction along the c -axis. It should

be noted that the Y released from Mg_2Y decomposition follows two parallel pathways during annealing: part of it can be incorporated into the $\alpha\text{-Mg}$ lattice as discussed above, while the remainder may be redistributed toward the Mg_2Si -containing regions, as tentatively discussed in Section 3.1.2. A full quantitative deconvolution of these contributions would require complementary characterization such as basal-plane composition mapping and TEM. Overall, the Rietveld results quantitatively support that Zn atoms released by MgZn dissolution are the dominant contributor to incorporation into the $\alpha\text{-Mg}$ matrix during the $200\text{ }^\circ\text{C}/12\text{ h}$ treatment, forming a substitutional solid solution that generates the compressive lattice distortion responsible for the subsequent solid-solution strengthening.

3.1.2. Elemental Distribution and Second-Phase Morphology

EPMA elemental mapping (Figure 3) reveals severe segregation of alloying elements along the extrusion direction in the AE state. The bright phases with higher atomic numbers in the BSE image (Figure 3a) form continuous bands and are identified as MgZn , $\text{Y}_2\text{Si}_2\text{Mg}$, and Mg_2Y . Owing to its low elastic modulus [19], the continuously banded MgZn is prone to cleave the $\alpha\text{-Mg}$ matrix, acting as a preferential site for crack initiation and matrix splitting. After heat treatment at $200\text{ }^\circ\text{C}$ for 12 h, the microstructure underwent two notable evolutions. First, the Zn distribution becomes markedly homogenized (Figure 3(e1)), and the corresponding EPMA signal exhibits a pronounced reduction, suggesting partial dissolution of the matrix-cleaving MgZn phase into the $\alpha\text{-Mg}$ matrix. Meanwhile, the continuous Y-rich bands associated with Mg_2Y also become separated (Figure 3(g) vs. Figure 3(g1)), which, combined with the disappearance of Mg_2Y diffraction peaks and the concurrent intensification of $\text{Y}_2\text{Si}_2\text{Mg}$ peaks in Figure 2e, indicates that the metastable Mg_2Y decomposes during annealing and releases Y atoms that are subsequently redistributed, part of which may participate in $\text{Y}_2\text{Si}_2\text{Mg}$ formation near the Mg_2Si -containing regions.

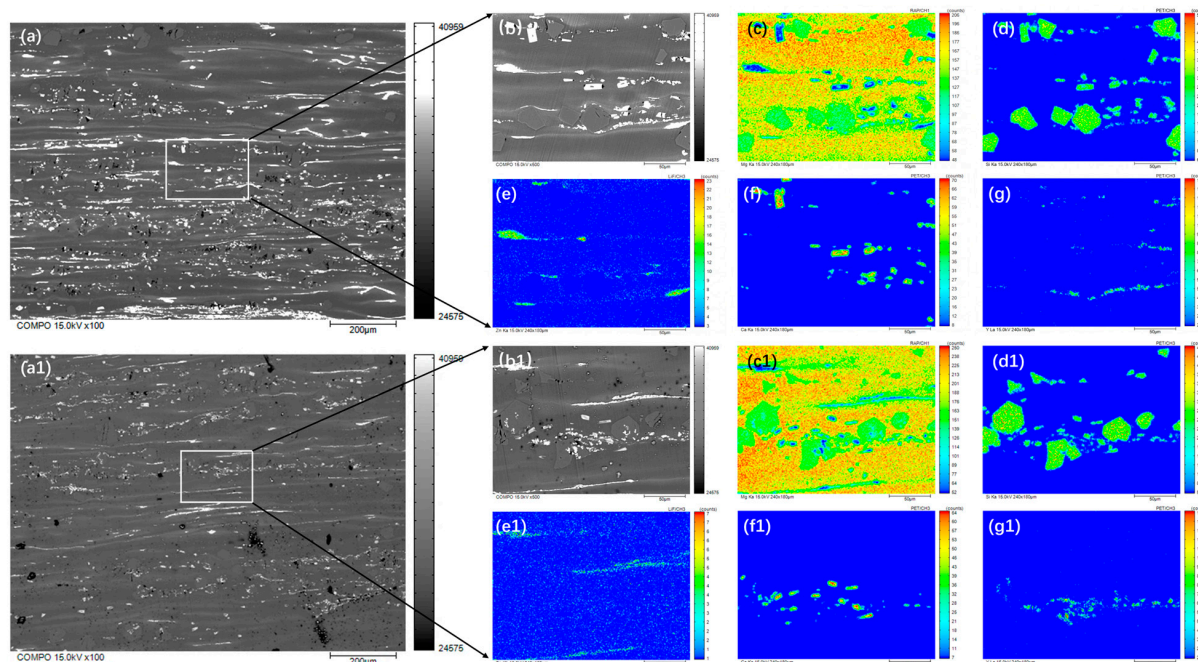


Figure 3. EPMA elemental maps. (a–g) AE; (a1–g1) HT. (a,a1) BSE images; (b,b1) BSE images (amplified); (c,c1) Mg; (d,d1) Si; (e,e1) Zn; (f,f1) Ca; (g,g1) Y.

Second, the continuous Ca- and Y-containing network phases (Figure 3f,g) are transformed into dispersed spheroidized particles (Figure 3(f1,g1)), driven by the minimization of total interfacial energy between the second phases and the $\alpha\text{-Mg}$ matrix. More importantly, under thermal activation, Y atoms are inferred to migrate towards the Mg_2Si particles. The

EPMA maps directly show that the originally continuous Y-rich network is broken up and discretized after annealing (Figure 3g vs. Figure 3(g1)), rather than remaining as connected bands. This discretization of the Ca/Y-containing network is the microstructural change most directly supported by the present results, and it removes the continuous low-strength paths in the as-extruded state. Whether the redistributed Y further reacts at the α -Mg/Mg₂Si interface to modify the local Mg₂Si morphology cannot be resolved by EPMA, and such an interfacial reaction remains a tentative interpretation that would require follow-up TEM/STEM-EDS characterization to confirm. Meanwhile, the blocky primary Mg₂Si particles remain intact (Figure 3b,(b1)), confirming their excellent thermal stability.

Since the alloy was processed by hot extrusion, crystallographic texture may also influence its mechanical response. Nevertheless, the present heat treatment temperature is relatively low compared with the temperature range commonly required for pronounced texture reorientation in Mg-Zn-Ca- or Mg-Zn-RE-based alloys. Previous studies showed that annealing at 200 °C caused little change in the extrusion-related fiber texture of Mg-Zn-Y-Nd alloys, whereas obvious texture weakening occurred only when the annealing temperature increased to 225–250 °C [12]. In low-temperature extruded Mg-Zn-Ca alloys, significant texture evolution was also associated with higher-temperature or long-term annealing, especially at 300 °C for 10 h [13]. Therefore, although a quantitative EBSD texture analysis would be valuable, the strength variation observed in the present 200 °C/12 h treatment is more reasonably associated with phase dissolution, solute redistribution, and second-phase morphology modification rather than texture effect.

3.2. Mechanical Properties and Fracture Behavior

3.2.1. Room-Temperature Tensile Properties

Engineering stress–strain curves of AE and HT alloys at room temperature are shown in Figure 4a. HT alloy exhibits slightly higher ultimate tensile strength with no significant change of elongation compared to AE alloy. Fracture morphologies (Figure 4(a1,a2)) reveal quasi-cleavage fracture characteristics with shallow dimples under both conditions. Coarse blocky phases presented in fracture surfaces (indicated by arrows on Figure 4(a1,a2)) are mainly large undissolved particles of Mg₂Si limiting the room temperature ductility by promoting crack initiation.

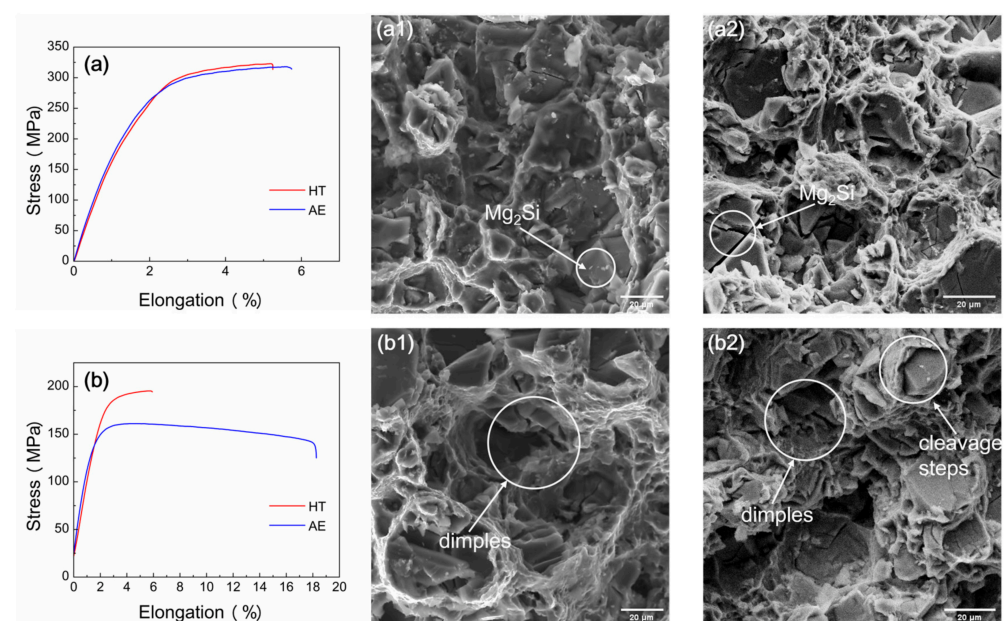


Figure 4. Engineering stress–strain curves and fracture morphologies. (a) RT; (b) 200 °C. Fracture surfaces: (a1,b1) AE; (a2,b2) HT.

3.2.2. High-Temperature (200 °C) Tensile Properties

At 200 °C, the HT alloy exhibits a remarkable enhancement in strength accompanied by a reduction in ductility (Figure 4b). The UTS increases significantly from ~160 MPa to ~200 MPa, while the elongation changes from ~18% to ~6%. At this condition, dislocation climb becomes the dominant deformation mechanism. The dissolution of Zn into the Mg matrix is expected to generate solute atmospheres, which may exert a solute drag effect that hinders dislocation motion and inhibits climb even at 200 °C. In addition, the prolonged holding at 200 °C for 12 h lies within the temperature–time range where precipitation or solute-clustering may occur in Mg–Zn-based alloys [12,20]. Similar Mg–Zn–Ca alloys have also been reported to exhibit precipitation- and segregation-assisted strengthening after post-deformation annealing [15,16]. Therefore, besides Zn solid-solution strengthening, possible fine Zn-rich clusters or precipitates may further impede dislocation slip via a pinning or Orowan-type mechanism. The exact crystallographic nature of these nanoscale features will be resolved by follow-up TEM analysis.

The fracture morphology accounts for the observed loss of ductility. The AE alloy exhibits deep and large dimples at 200 °C (Figure 4(b1)), reflecting extensive plastic deformation. In contrast, the HT fracture surface (Figure 4(b2)) shows a relatively flat morphology with cleavage steps and shallow dimples. Since the matrix is heavily strengthened by solute atoms and fine precipitates, dislocation mobility is severely restricted, leading to dislocation pile-up at the undissolved coarse Mg_2Si particles and grain boundaries. The localized stress concentration cannot be effectively released through plastic yielding of the matrix, resulting in premature micro-crack nucleation and the reduction in ductility.

3.3. Discussion of Strengthening Mechanisms

The property changes after annealing stem from three diffusion-controlled processes occurring within the same treatment process. Zn re-dissolution requires only short-range diffusion in the α -Mg lattice, spheroidization of the Ca/Y-rich network phase is driven by interfacial-energy minimization, and $\text{Y}_2\text{Si}_2\text{Mg}$ formation depends on longer-range transport of Y element released during Mg_2Y decomposition. Despite their differing activation energies, 200 °C/12 h is sufficient to activate these local diffusion processes while remaining below the temperature range that commonly induces extensive recrystallization, grain growth, or strong texture reorientation in Mg–Zn–Ca- and Mg–Zn–RE-based alloys [12,13]. This is also consistent with recent simulations indicating that solute drag can markedly reduce grain-boundary mobility and slow static recrystallization during low-temperature annealing of Mg–Zn–Ca alloys [14]. Therefore, the present annealing treatment enables simultaneous matrix strengthening and second-phase passivation. Based on the annealing behavior of related Mg–Zn–Ca and Mg–Zn–RE alloys, extensive grain coarsening and texture reconstruction are expected to be limited at this relatively low temperature, although they cannot be fully quantified without EBSD [12–14].

These three effects contribute unequally to the mechanical response. Zn solutes, distributed throughout the matrix, are the main contributor to strength retention at 200 °C by impeding both dislocation glide and climb. Spheroidization of the Ca/Y network phase provides little direct hardening, but eliminates a continuous, low-strength crack path rather than increasing the tensile strength of the matrix. Similar post-deformation annealing studies on Mg–Zn–Ca alloys have shown that solute segregation and fine precipitates can suppress grain-boundary migration and contribute to microstructural stability through solute-drag and Zener-pinning effects [13–16]. $\text{Y}_2\text{Si}_2\text{Mg}$ plays an intermediate role, improving local load transfer at the Mg_2Si interface. Since these mechanisms operate at different length scales—lattice, network, and interface—their contributions are largely additive, consistent with the concurrent gain in strength and moderate loss in ductility.

This also explains the remaining ductility loss. Coarse primary Mg_2Si is unaffected by any of the above mechanisms—too large to dissolve, too stable to spheroidize, and insufficiently reactive with Y under the present annealing conditions. As the matrix hardens, the load previously relieved by matrix flow is transferred directly to these particles, promoting earlier interfacial failure and a shift toward cleavage fracture. Thus, annealing strengthens all microstructural constituents accessible at low temperature, while coarse, thermally stable Mg_2Si remains the limiting factor for high-temperature ductility.

This indicates that further gains in elevated-temperature ductility would require upstream modification of the primary Mg_2Si phase—through adjusted extrusion parameters or targeted alloying additions—rather than further refinement of the annealing schedule.

4. Conclusions

Low-temperature annealing at 200 °C for 12 h was applied to the extruded Mg-2.5Si-4Zn-0.7Ca-1Y alloy, and the following conclusions are drawn:

- (1) The UTS at 200 °C increased from ~160 MPa to ~200 MPa after annealing, while elongation decreased from ~18% to ~6%. Room-temperature strength showed a marginal improvement with negligible change in ductility.
- (2) Different alloying elements exhibit distinctly different thermally activated behaviors during the same low-temperature annealing cycle. Zn atoms, originally locked in the continuously banded MgZn phase formed during extrusion, partially re-dissolve into the α -Mg matrix, generating a compressive lattice distortion that is considered a major strengthening contribution via solute drag and dislocation pinning at 200 °C. Simultaneously, Ca/Y-rich network phases spheroidize driven by interfacial energy minimization, eliminating continuous crack propagation paths without directly hardening the matrix.
- (3) After annealing, the identifiable Mg_2Y diffraction peaks disappear while the relative intensity of the $\text{Y}_2\text{Si}_2\text{Mg}$ peaks increases, and the Y distribution becomes less continuous. These combined XRD and EPMA results suggest that metastable Mg_2Y decomposes during annealing and that the released Y is redistributed, part of which may be associated with $\text{Y}_2\text{Si}_2\text{Mg}$ near the Mg_2Si -containing regions. However, the present data cannot establish the location or continuity of an interfacial layer, and its proposed role in blunting the sharp edges of eutectic Mg_2Si requires confirmation by TEM/SAED or STEM-EDS.
- (4) The concurrent loss of ductility is primarily attributed to stress concentration at the unrefined coarse primary Mg_2Si particles: as the matrix is progressively hardened, load redistribution toward these thermally stable particles promotes premature interfacial cracking and a transition toward cleavage-dominated fracture.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/cryst16080529/s1>: Table S1: Ultimate tensile strength (UTS) and elongation (El) of the HT state tested at room temperature and elevated temperature.

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Conflicts of Interest: The authors declare no conflicts of interest.

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