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To cite this article: Hucheng Pan, Dongsheng Xie, Jingren Li, Hongbo Xie, Qiuyan Huang, Qingshan Yang & Gaowu Qin (2021) Development of novel lightweight and cost-effective Mg–Ce–Al wrought alloy with high strength, Materials Research Letters, 9:8, 329-335, DOI: 10.1080/21663831.2021.1918776

To link to this article: <https://doi.org/10.1080/21663831.2021.1918776>



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Published online: 27 Apr 2021.



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ORIGINAL REPORT



Development of novel lightweight and cost-effective Mg–Ce–Al wrought alloy with high strength

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ABSTRACT

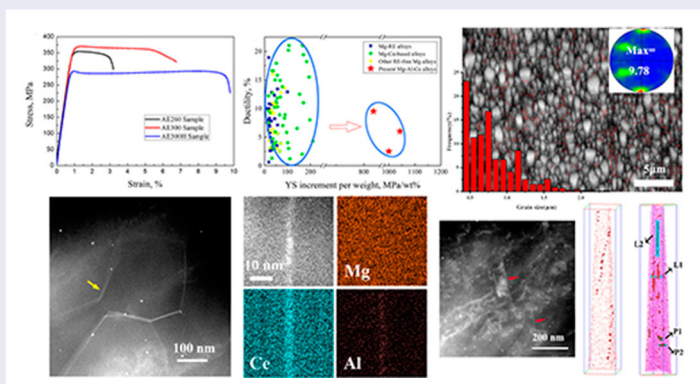
The low microalloying content and high yield strength of 365 MPa have been achieved simultaneously in extruded Mg–Ce–Al alloy which is mainly ascribed to the unexpected grain refinement (mean size of ~ 420 nm). It is found that adding tiny Al (0.05 wt.%) into Mg–0.2 wt.% Ce alloy can promote the formation of Ce–Al-enriched segregation along dislocations and grain boundaries which can effectively counterbalance the thermally activated dislocation recovery and thus guarantee grain refinement. The ultra-fine grains are mainly related to both linear and planar cosegregation of solutes, rather than the traditional planar segregations and/or nanoprecipitations at grain boundaries.

ARTICLE HISTORY

Received 20 February 2021

KEYWORDS

Mg wrought alloy; high strength; compositional plainification; segregations; low cost



IMPACT STATEMENT

A new mechanism of linear/planar cosegregation of solutes for ultrafine DRXed grains formation in Mg alloys has been found, rather than traditional planar segregations and/or nanoprecipitations at grain boundaries.

1. Introduction

Recently, a novel development direction for high-performance metallic materials is to maintain the excellent mechanical properties, based on the concept of compositional 'plainification' or the low-alloying, which was firstly proposed by the Lu group [1]. The purpose is to stabilize the existing defects and thus promote the formation of ultrafine/nanosized grains with no or small

addition of solutes [2]. Magnesium, as the lightest metallic structural materials ($\sim 2/3$ of aluminum and $\sim 1/4$ of steel in density), acts as an important candidate for weight saving, which has great potential for usage in fields of transportation and aerospace [3]. In accordance with the concept of 'plainification', low-alloyed Mg materials have recently attracted much attention because their deformation resistance is low, which represents high

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Supplemental data for this article can be accessed here. <https://doi.org/10.1080/21663831.2021.1918776>

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formability [4]. Moreover, the density of ‘plain’ Mg alloys continues to decrease by 10 ~ 20% compared with those of conventional heavily alloyed Mg counterparts, and the advantage of weight saving becomes more obvious [5]. Recently, the highest strength of rare-earth-free and low-alloyed Mg alloy has reached ~460 MPa in the Mg–2Sn–2Ca wt.% wrought alloy [6]. This means that excessive alloying elements can be avoided by optimizing the choice of solutes and processing routes. In this sense, novel high-strength and low-alloyed (HSLA) Mg alloys, which have better workability and low cost, exhibit broad prospects for industrial applications.

Nevertheless, further reducing the content of alloying elements is always demanded, and the development of lower-microalloyed Mg materials with higher strength is more attractive [7]. One apparent reason is that the addition of lower alloying elements can effectively avoid the formation of the second phase, which can promote the uniform corrosion of the Mg matrix and greatly improve the intrinsic corrosion resistance [8]. Moreover, the recycling and sustainable development of Mg resources by microalloying design is highly significant. However, it is still a great challenge to achieve obvious grain refinement in this type of ‘plain’ Mg alloy. In this study, we chose Mg–0.2 wt.% Ce alloy as the model material, which was previously proved to be highly ductile [9]. However, the reported yield strength for the Mg–Ce-based alloy is usually low, less than 100 MPa. In this context, a new microalloyed Mg–Ce–Al alloy has been explored, and the unexpected ultra-fined grains and high strengths (above 350 MPa) were achieved.

2. Materials and methods

As-cast Mg–0.2Ce–0.05Al (in wt.%) ingot was homogenized at 500°C for 24 h, and then the indirect extrusion process was conducted at either 260°C or 300°C with an extrusion ratio of 20:1. The three parallel dog-bone tensile specimens with a diameter of 5 mm and gauge length of 25 mm were machined along the extrusion direction (ED), and measured at the initial strain rate of 0.001 s^{-1} . The grain structures, textures and geometrical necessary dislocations (GNDs) density were observed by electron backscatter diffraction (EBSD). Transmission electron microscopy (TEM, JEOL JEM-2100F) was also applied, operating at 200 kV, and the high-angle annular dark-field (HAADF) observations were conducted. Atom probe tomography (APT) was performed, and the specimens were prepared by mechanical tripod polishing followed by Ar-ion milling in a PIPSTM-II (Gatan) and performed using a local electrode atom probe (CAMECA LEAP4000 HR) at a temperature of 50 K with a pulse fraction of 15% and pulse rate of 200 kHz.

3. Results and discussion

Figure 1 presents the tensile curves of the Mg–0.2Ce–0.05Al wt.% samples, which are prepared by the traditional one-step extrusion. The yield strength (YS) of the sample in tension was measured to be ~350 MPa when the billet was extruded at a low temperature of 260°C and a die-exit speed of ~0.5 mm/s (designated as AE260). The elongation to failure (EL) of the AE260 sample was only ~2.5%. When the extrusion temperature was increased to ~300°C (designated as AE300, with the same extrusion speed of ~0.5 mm/s), the tensile YS of the AE300 sample was increased to ~365 MPa, and the EL was also enhanced up to ~6%. When further increasing the extrusion speed to ~1.5 mm/s for the Mg billet at the same temperature of ~300°C (designated as AE300H), the YS of AE300H sample was decreased to ~286 MPa, while a high EL was obtained ~9.5%.

Importantly, the total solute concentration of the present Mg–Ce–Al alloy is only ~0.25 wt.%, which has been decreased by one magnitude compared with those of commercial Mg alloys, such as AZ31, AM60 and ZK60 alloys (> 4 wt.%, in total). As a result, the YS values divided by the weight percentage (referred to as ‘YS increment per weight’) can reach as high as ~1000 MPa/wt.%, which is approximately one magnitude higher than those of the previously reported Mg alloys [10–14] and our recently reported Mg–Ca-based alloys [6,15]. The extremely large ‘YS increment per weight’ values represent the excellent cost-effectiveness.

EBSD images (Figure 1(c,d), Supplementary Figure S1) show that the AE260 and AE300 samples exhibit a bimodal grain microstructure, i.e. the dynamically recrystallized (DRXed) grains having random orientation and the un-DRXed regions having a typical fiber texture of $\langle 10\bar{1}0 \rangle // \text{ED}$. The un-DRXed grains are separated by the low-angular grain boundaries (LAGBs), and the subgrain lamellae is more widely distributed in AE260 ($0.5 \sim 3 \mu\text{m}$) than those in AE300 ($0.3 \sim 1 \mu\text{m}$). The pole figures indicate that the $\langle 10\bar{1}0 \rangle // \text{ED}$ texture intensity was decreased from ~21.58 multiples of the random distribution (mrd.) in AE260 to ~9.78 mrd in AE300, and the higher volume fraction of un-DRXed regions in AE260 (~66.7%) than that in AE300 (~45.8%) should be the major reason.

TEM images (Figure 2(a,b)) display that the DRXed grains with sizes of $0.2\text{--}1.0 \mu\text{m}$ and subgrain lamellae with thicknesses of $0.5\text{--}2.0 \mu\text{m}$ can be observed in the AE260 (~ $0.64 \mu\text{m}$ in average for subgrains). In the AE300 sample, in contrast, the subgrain thickness drops to only $0.2\text{--}0.5 \mu\text{m}$, with average value of ~ $0.39 \mu\text{m}$ (Figure 2(d)). Numerous linear defects belong to the residual dislocations, and the accompanied nanoparticles

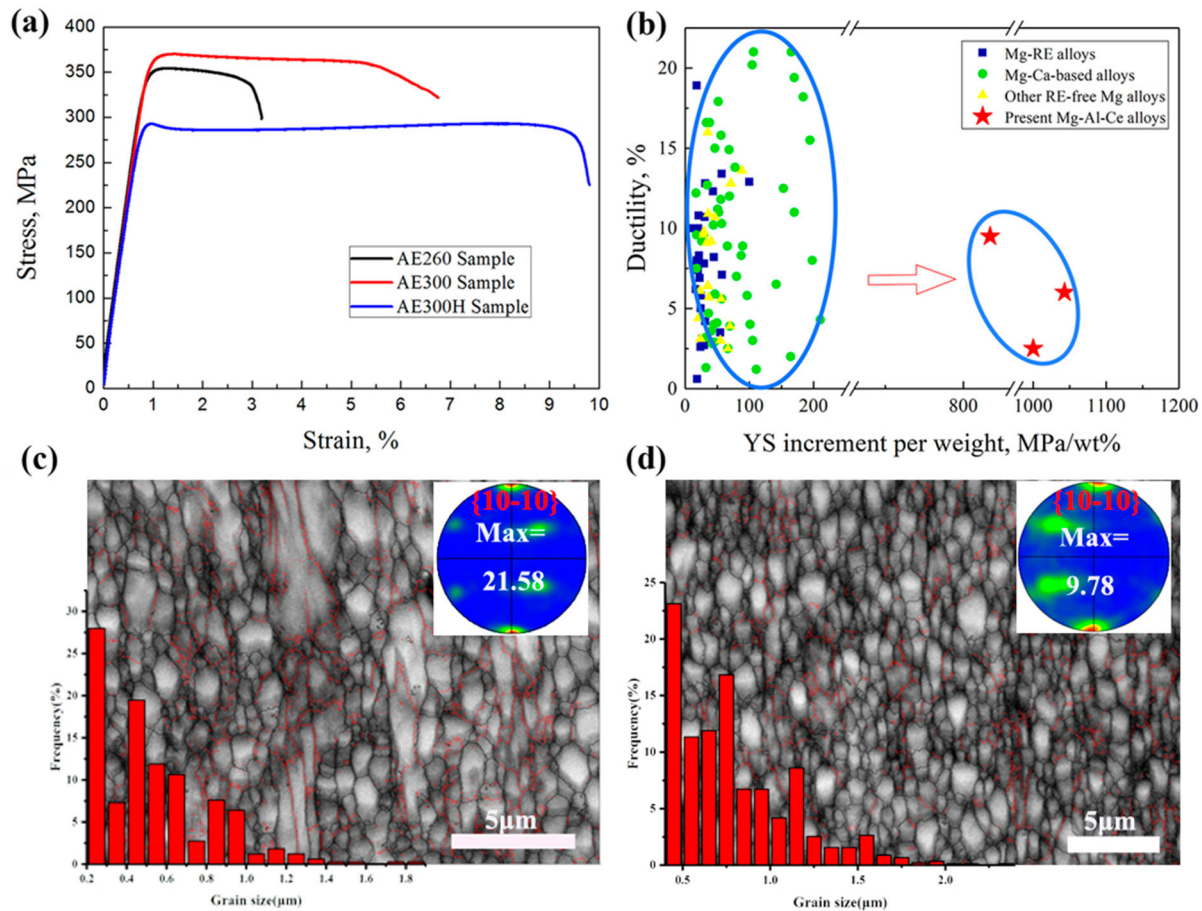


Figure 1. (a) Nominal stress–strain curves of the AE260, AE300 and AE300H samples under tension and (b) the distribution map of ‘YS increment per weight’ and ductility of the present Mg–Ce–Al alloys and previously reported Mg wrought alloys. EBSD images of the (c) AE260 and (d) AE300 as-extruded Mg samples, with the pole figures of 10–10 presented as inset.

can be readily observed in both AE260 and AE300 samples (Figure 2(a–f)). After high-speed extrusion, the DRXed grains in the AE300H sample apparently grow ($1.0 \sim 1.5 \mu\text{m}$), while the number density of residual dislocations was decreased (Figure 2(g–i)). Some nanoparticles are readily precipitated along the LAGBs (blue arrows, Figure 2(i)). The average DRXed grain sizes in AE260, AE300, and AE300H samples are estimated to be ~ 0.47 , ~ 0.61 and $\sim 1.21 \mu\text{m}$, respectively.

HAADF observations were further conducted, as shown in Figure 3. Most GBs in the present samples exhibit a bright contrast (yellow arrows, Figure 3(a,d,g)), and corresponding mapping result in Figure 3(f) confirms the existence of the planar cosegregation with both Ce and Al elements. Some bright nanoparticles can be detected, and the high-resolution TEM image and the mapping result in Figure 3(c) prove that the nanoparticles in the AE300 sample belong to the Mg_{12}Ce phase [16], while some nanoparticles distributed along the LAGBs in the AE300H sample consisted of both Al and Ce atoms, which can be determined to be the Al–Ce phase

according to the Mg–Al–Ce phase diagram [17]. And the nanoparticles are heterogeneously distributed in the Mg matrix with a total volume fraction of less than 0.2%, which is also consistent with the TEM images in Figure 2. Importantly, some linear defects with bright contrast can be also detected in the grain interiors (red arrows, Figure 3(b,e,h)), which suggests the possible solute segregation along dislocations.

APT analysis shows that the nanoclusters are readily distributed along the dislocation lines in the present AE300 sample, Figure 4(a,b) and Supplementary Figure S2. The compositional profiles, taken across the nanoclusters in typical curved dislocations (Figure 4(a) and Supplementary Figure S2), show that both Al and Ce solute elements are evidently concentrated, with 2–5 at.% Al and 1.5–2.0 at.% Ce, together with some Cu and Si impurity. Another APT result in Figure 4(b) suggests that solute segregation along dislocation can be also straight, with a linear length scale of 50–100 nm. The compositional profile in L1 displays that the Al and Ce atoms overlap with each other, with concentrations

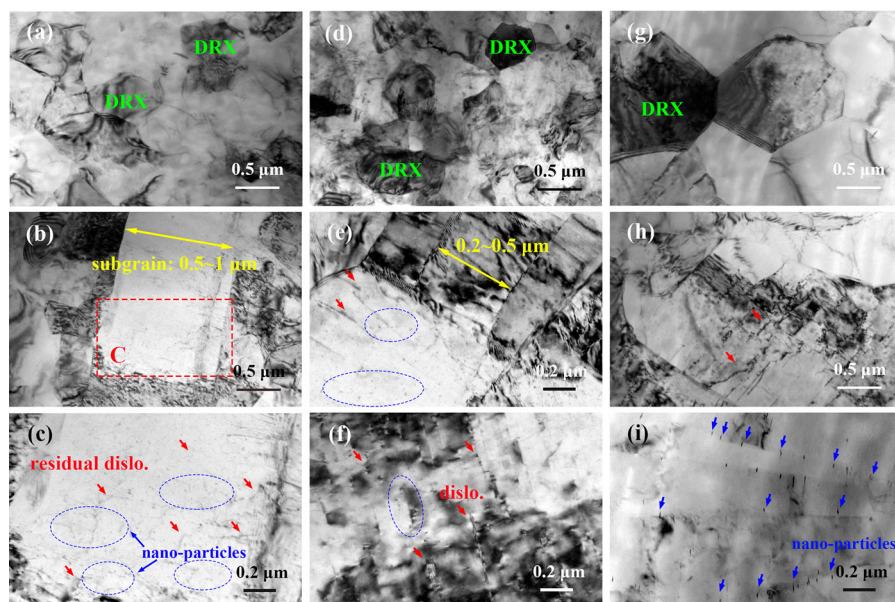


Figure 2. TEM images of the as-extruded (a–c) AE260, (d–f) AE300, and (g–i) AE300H samples, including the (a,d,g) DRXed grains, (b,e,h) subgrains and (c,f,i) residual dislocations and the accompanied nanoparticles. The nanoparticles are marked by blue circles and residual dislocations are marked by red arrows, respectively.

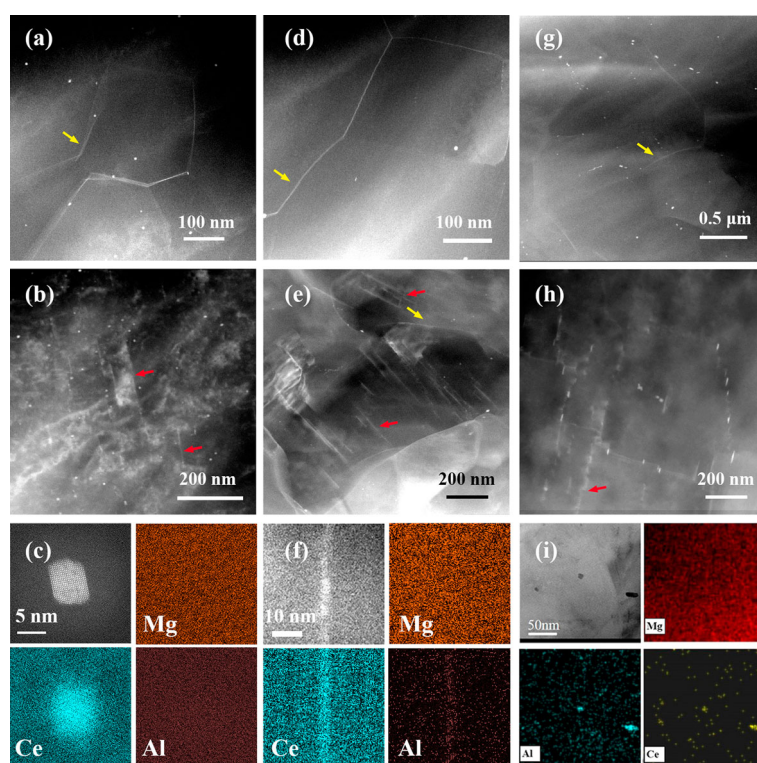


Figure 3. HAADF-STEM and the corresponding mapping images for (a–f) AE300 and (g–i) AE300H samples. Both the GB segregation and existences of the nano-Mg₁₂Ce phase, as well as the Al–Ce phase, have been confirmed.

of ~ 9 at.% Al atoms and ~ 5 at.% Ce atoms. Both the X-axis and Y-axis views are provided for line L2, which proves that the present segregation is indeed linear. Besides that, some random clusters can be also found, e.g. P1 and P2, containing 4–5 at.% Al and 2–3 at.% Ce.

Another APT map involving three typical grain boundaries of GB1, GB2 and GB3 can be found in Figure 4(c), and Supplementary Figure S3. The compositional profiles across GB1 confirms the obvious planar segregations, with ~ 5 at.% Al and ~ 5 at.% Ce. More

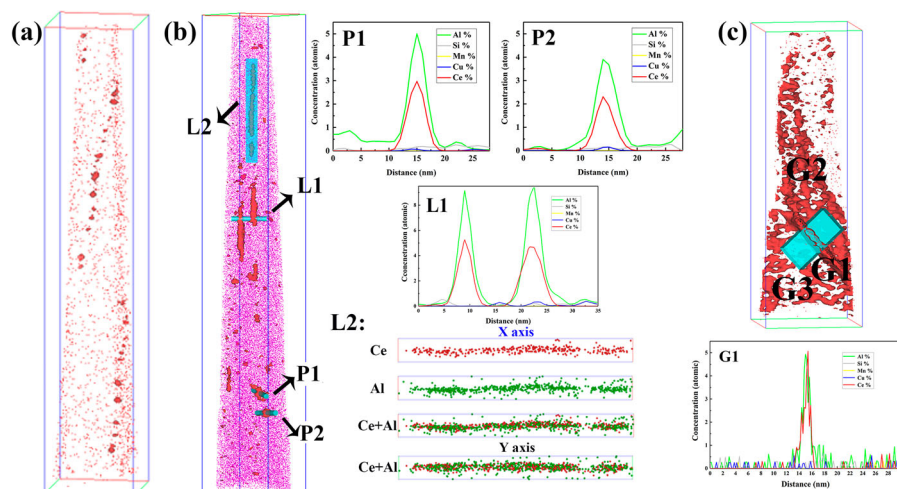


Figure 4. APT maps for the AE300 sample, including (a) the curved dislocations with decorated nano-clusters ($60 \text{ nm} \times 60 \text{ nm} \times 318 \text{ nm}$) and (b) straight dislocations with linear segregations ($74 \text{ nm} \times 74 \text{ nm} \times 477 \text{ nm}$, as revealed by the 1.3 at.% Ce isocompositional surface). Compositional profiles across the typical clusters of P1 and P2, as well as the typical linear profile L1 are provided. Both X-axis and Y-axis views are provided for line L2. (c) The GB segregations are also evidently detected in AE300, with box dimensions of $50 \text{ nm} \times 50 \text{ nm} \times 152 \text{ nm}$.

GB segregation results can be found in Supplementary Figure S3.

In this study, the microalloyed Mg–Ce–Al alloy with a total solute content of $\sim 0.25 \text{ wt.}\%$ was extruded, and ultra-fine grains less than $0.5 \mu\text{m}$ was achieved (Figures 1 and 2). This is unexpected because the DRX temperature of the Mg alloy is usually low due to the low melting point [5]. Thermally activated grain growth would dominate during thermomechanical processing, which can easily lead to grain growth [6]. Consequently, the traditional grain refinement in Mg alloys commonly depends on reducing the deformation temperature. For example, Zeng et al. [18] extruded pure Mg at an extremely low temperature of $\sim 80^\circ\text{C}$, and a minimum grain size of $\sim 1.2 \mu\text{m}$ was achieved because dynamic recovery was suppressed at this low temperature. Another approach was to add more solutes, which can guarantee the formation of high-density nanophases, and the GB migration can thus be effectively hindered. For example, Elsayed et al. [19] reported that the minimum grain size of $\sim 1 \mu\text{m}$ can be produced in the highly alloyed Mg–10Sn–3Al–1Zn wt.% sample, and the full DRX temperature becomes much higher ($\sim 300^\circ\text{C}$), as compared with the $\sim 80^\circ\text{C}$ for pure Mg.

With regard to the low-alloyed Mg samples, the strategy of combining solute segregation and/or nanoprecipitations at GBs can be utilized to achieve ultrafine DRXed grains. For example, our previous results [6] showed that full DRX could not be accomplished in the Mg–2Sn–2Ca wt.% alloy until a high temperature of $\sim 280^\circ\text{C}$ was reached. The Ca segregation along the subgrain boundaries and the homogeneous precipitation of nano-Mg₂Ca

phases can effectively inhibit grain growth, and the minimum grain size can be refined to only $\sim 0.32 \mu\text{m}$. In the commercial AZ31 alloy, the Zn segregation at GBs plays a similar role, and the grain size can be easily refined to $\sim 1 \mu\text{m}$ [18]. More recently, the DRX temperature can be further increased to higher than $\sim 330^\circ\text{C}$ in the low-alloyed Mg–1Al–0.2Zn–0.1Mn–1Ca wt.% alloy, and the mean subgrain size has been further decreased to only $\sim 300 \text{ nm}$. The cosegregation of Al, Ca and Zn elements along the GBs with higher concentrations should play an important role [5].

In the microalloyed Mg alloys, the nanoprecipitation at GBs is usually lacking, and the planar segregation also disappeared due to the lower total solute content. And it is still a great challenge to increase the DRX temperature in ‘plain’ Mg alloy. Here, we found for the first time that adding tiny Al ($\sim 0.05 \text{ wt.}\%$) into Mg–0.2 wt.% Ce alloy can promote the formation of Ce–Al-enriched segregation along both dislocations and grain boundaries, while no segregation was found in Mg–Ce binary alloys, as recently reported by Li et al. [20]. Firstly, this cosegregation at the GBs can contribute to grain refinement of the Mg matrix by both thermodynamically lowering the GB energy and kinetically pinning the GB migration [5,6]. Secondly, the plastic strain in the present Mg alloy is mainly accommodated by dislocation slipping during extrusion. The grain misorientation caused by the high-density dislocations in the grain interior is the direct evidence (Figures 2 and 3). Therefore, the new DRXed grains are formed by following the typical manner of dislocation accumulation and dynamic recovery, which leads to the formation of LAGBs [5,18]. The

LAGBs would then gradually transform into HAGBs by absorbing more new dislocations [6]. In this context, the dislocation behavior can necessarily influence the DRX procedure.

Accordingly, it is found that the addition of minor Al can induce the formation of Ce–Al-enriched clusters, which can significantly promote dislocation accumulation by hindering dislocation movements. As proven by the TEM and APT results in Figures 2–4, numerous dynamically activated dislocations are always present in the grain interiors of as-extruded Mg samples. These dislocations, as well as the GBs above, are readily stabilized in the present Mg–Ce–Al dilute alloy, which can effectively promote dislocation storage and counterbalance the thermally activated dislocation recovery. Consequently, the DRX temperature of the present Mg–Ce–Al dilute alloy has been increased to higher than $\sim 300^\circ\text{C}$. In general, these linear and planar cosegregations of Al and Ce elements should play the key role in achieving the submicron sized grain refinement.

The mechanism for the solute segregation in the Mg alloy is closely related to the atomic size, chemical bonding and electronegativity [21,22]. The atomic size is one reason, and the size of Al ($\sim 1.43 \text{ \AA}$) is smaller than Mg ($\sim 1.60 \text{ \AA}$), while Ce ($\sim 1.82 \text{ \AA}$) is larger than Mg. The cosegregation of Al and Ce atoms can thus compensate for this tension/compression strain field at dislocation cores and become more energy favorable. The large negative mixing enthalpy of -610 kJ/mol between Al and Ce should be the other reason [21], since the mixing enthalpy between Mg and Ce atoms is only -25 J/mol [22]. In this sense, the cosegregation of Al and Ce atoms along the GB and/or dislocations is also thermodynamically feasible.

As a result, a high yield strength above 350 MPa has been achieved in the present Mg–Ce–Al alloy. It is noted that the solid solution hardening and the Orowan hardening should be ignored due to the dilute alloying content, with total second phases fraction less than 0.2%. The grain refinement hardening can be evaluated by the Hall–Petch (H–P) relationship [23], and the parameters of H–P relation slope (k) and the friction stress (σ_0) depend much on the texture and grain size. Accordingly, the grain refinement hardening from DRXed grains and subgrain lamellae can be calculated to be ~ 339 and $\sim 303 \text{ MPa}$ for AE260, and $\sim 323 \text{ MPa}$ and $\sim 330 \text{ MPa}$ for AE300, respectively (see supplementary information).

Importantly, the profuse residual dislocations, which usually belong to the GNDs, have been preserved after extrusion in the present Mg alloy. The residual dislocation density, ρ_{GND} , can be estimated to be 1.3×10^{15} and $0.9 \times 10^{15} \text{ m}^{-2}$, and the dislocation hardening can be evaluated to be ~ 96 and $\sim 85 \text{ MPa}$ for AE260 and

AE300, respectively (see supplementary information). Accordingly, the total yield strengths can be predicted to be ~ 379 and $\sim 365 \text{ MPa}$ for AE260 and AE300, which agrees generally with the experimental values in Figure 1. The over-estimated strength might come from the statistical uncertainties of both grain size and dislocations in the present Mg samples.

The ductility of the Mg alloy is closely related to the movement of $\langle c+a \rangle$ dislocations. For the AE300 and AE300H with higher degree of DRX (Figure 1), the plastic strain in the DRXed region can be also more easily accommodated due to the randomized texture, which is beneficial for ductility [5]. Moreover, the subgrain thickness in the un-DRXed area has been decreased from $\sim 0.64 \mu\text{m}$ in AE260 to $\sim 0.39 \mu\text{m}$ in AE300. In this $\langle 10\bar{1}0 \rangle // \text{ED}$ textured region, the dominant pyramidal slipping has been confirmed to be stable at the early stage of deformation [24]. More importantly, the LAGBs can act as both the dislocation barriers and the dislocation sources [25]. Therefore, the fine subgrain lamellae in AE300 can contribute to the high yield strength via affording grain refinement hardening, and the mobile $\langle c+a \rangle$ dislocations can also easily interact with the nearby LAGBs, and promote the dislocation multiplications, which guarantees the high strength-ductility synergy in AE300. Further increasing forming speed in AE300H leads to a more randomized texture, but also the larger grain size. Accordingly, the elongation is largely increased up to $\sim 9.5\%$, while the strength is decreased to only $\sim 286 \text{ MPa}$.

4. Conclusions

In summary, a lightweight and cost-effective Mg–Ce–Al dilute extrusion alloy has been successfully developed. The minor Al addition can induce the formation of Ce–Al-rich segregation at both dislocations and GBs. These combined effects of linear and planar segregation effectively guarantee the submicron sized grain refinement and high yield strength in the present Mg–Ce–Al alloy. The concepts of high-strength and dilute alloying content for Mg wrought alloys have a significant impact on alloy design and development from the viewpoint of resource-saving sustainable materials.

Acknowledgements

The authors are grateful for the facilities and technical supports provided by the Key Laboratory for Anisotropy and Texture of Materials, Northeastern University.

Disclosure statement

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

Funding

This work is supported by National Natural Science Foundation of China [Grant Numbers 51701211, and 51971053], and funded by the Project of Promoting Talents in Liaoning province [Grant Number XLYC1808038], and Chongqing Municipal Education Commission [Grant Number KJZD-K202001502]. H. C. Pan acknowledges the financial assistance from Young Elite Scientists Sponsorship Program by CAST [Grant Numbers 2019-2021QNRC001, 2019-2021QNRC002], and Joint Research Fund Liaoning-Shenyang National Laboratory for Materials Science [Grant Number 2019JH3/30100040]. Fundamental Research Funds for the Central Universities

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