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Published in:
Journal of Alloys and Compounds

Link to article, DOI:
[10.1016/j.jallcom.2019.153051](https://doi.org/10.1016/j.jallcom.2019.153051)

Publication date:
2020

Document Version
Peer reviewed version

[Link back to DTU Orbit](#)

Citation (APA):
Luo, X., Feng, Z., Fu, R., Huang, T., Wu, G., & Huang, X. (2020). The synergy of boundary engineering and segregation strategy towards high strength and ductility Mg-3Gd alloy. *Journal of Alloys and Compounds*, 819, [153051]. <https://doi.org/10.1016/j.jallcom.2019.153051>

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Journal Pre-proof

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PII: S0925-8388(19)34297-5

DOI: <https://doi.org/10.1016/j.jallcom.2019.153051>

Reference: JALCOM 153051

To appear in: *Journal of Alloys and Compounds*

Received Date: 21 September 2019

Revised Date: 14 November 2019

Accepted Date: 15 November 2019

Please cite this article as: X. Luo, Z. Feng, R. Fu, T. Huang, G. Wu, X. Huang, The synergy of boundary engineering and segregation strategy towards high strength and ductility Mg-3Gd alloy, *Journal of Alloys and Compounds* (2019), doi: <https://doi.org/10.1016/j.jallcom.2019.153051>.

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Author Contributions

G.L. Wu and Z.Q. Feng conceived the experiments and provided guidance. X. Luo and R. Fu conducted the experiments. X. Luo, T.L. Huang and X.X. Huang analysed the results. X. Luo, Z.Q. Feng and G.L. Wu wrote the manuscript. All authors contributed the discussion.

The synergy of boundary engineering and segregation strategy towards high strength and ductility Mg-3Gd alloy

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Abstract: Nanostructured materials prepared by severe plastic deformation processing were puzzled by the trade-off between strength and ductility. With the synergy of boundary engineering and segregation strategy, a nanostructured Mg-3Gd (wt. %) alloy with simultaneously enhanced strength and ductility was fabricated by accumulative roll-bonding processing and subsequent annealing treatment. Based on transmission electron microscopy observations, clear evidences of Gd segregation at grain boundaries, twin boundaries, stacking faults and dislocations can be observed in annealed samples. Meanwhile, the activation of non-basal slip and intensive interaction between dislocations and Gd atoms were determined in the annealed sample after tensile testing. Boundary segregation and pinning of dislocations were considered to be the main reason for the strength enhancement, while the increase in ductility was attributed to the inhibition of premature failure by Gd segregation at twin boundaries and the activation of non-basal slip.

Keywords: Mg-3Gd alloy; Boundary engineering; Segregation; Non-basal slip; Mechanical properties

1 Introduction

Magnesium and its alloys are superior materials for lightweight automotive applications due to their high strength-density ratio [1-4]. However, low strength and poor formability at ambient temperature immensely limit the application of magnesium alloys [2,5,6]. To enhance the strength of magnesium alloys, the structural refinement by severe plastic deformation (SPD) processing, such as accumulative roll-bonding (ARB) [7,8], equal channel angular extrusion [9,10] and high pressure torsion [11,12], has been recognized as a very effective way. Previous investigations [5,7,13-15] demonstrated that with introduction of high-density defects, such as grain boundaries (GBs), twin boundaries (TBs) and stacking faults (SFs), the dislocation glide can be effectively impeded, resulting in high strength in magnesium alloys. Furthermore, combining SPD processing and subsequent annealing, ultrahigh strength magnesium alloys also can be fabricated through the formation of extensive clusters, nano-precipitates or grain boundary segregation [16-18]. Although high strength magnesium alloys have been reported substantially, it is still a challenge to prepare magnesium alloys with both high strength and ductility [2,3,5,19].

Recent studies have revealed that the trade-off relationship between strength and ductility in metallic materials can be up-turned by utilizing boundary architectures and complexion strategies synthetically [5,6]. Compared with the precipitation hardening behaviors of traditional magnesium alloys, solute segregation and co-clustering at dislocations and grain boundaries can induce a quick hardening response [19-21]. For instance, Bian et al. [6] reported a rapid age-hardening response in Mg-Al-Zn-Mn-Ca alloy with a significant increase of the flow stress. Moreover, solute segregation and enhanced non-basal slip can release the localized stress concentration generated by deformation twins, and thus postpone the premature failure of magnesium alloys [13,22,23]. However, it is still lack of sufficient knowledge of solute segregation characteristics at various defects including dislocations, SFs, TBs and GBs. Besides, the combined effect of boundary and segregation engineering on the strength and ductility of nanostructured magnesium

alloys are not yet systematically understood.

With a combination of boundary engineering and segregation strategy, here we prepared a nanostructured Mg-3Gd alloy with simultaneously high strength and good ductility via ARB processing and subsequent annealing treatment. The segregation characteristics at various defects were fully revealed and the strengthening and toughening mechanisms of Mg-3Gd samples were discussed. This approach provides a guidance for developing magnesium alloys with simultaneously high strength and ductility.

2. Experimental

An Mg-3Gd (wt. %) ingot with a diameter of 100 mm was prepared by the casting method, followed by homogenization treatment at 450 °C for 24 h. The homogenized ingot was then subjected to hot extruding to a plate with thickness of 3 mm, followed by hot rolling to 1 mm thick sheet at 450 °C and finally annealed at 500 °C for 3 h. Specimens with a length of 70 mm and a width of 25 mm were prepared from the initial 1 mm thick sheet samples for processing using ARB [24,25]. Two such specimens were stacked and preheated at 400 °C for 8 min, and then roll-bonded with a rolling reduction of 50%. The ARB processing was carried out for one cycle without lubrication with the rolling direction (RD) kept fixed during each rolling treatment. The processed ARB samples were annealed for 1h at 190 °C and 250 °C.

Dog-bone-shaped tensile specimens with a gauge length of 13 mm and a cross-section of $5 \times 1 \text{ mm}^2$ were cut along the RD direction. Uniaxial tensile tests were performed using a Shimadzu AGX-50 tensile machine with a strain rate of 10^{-4} s^{-1} at room temperature. A laser extensometer was used to measure the specimen strain during loading. Three tensile tests were carried out for each sample condition to reduce errors. The Vickers microhardness was measured on a MH-3 microhardness tester under a static load of 300 gf for 10 s. The obtained microhardness values were averaged from at least 10 indentations for each sample.

The microstructures of the ARB and annealed samples were characterized by using a JEOL JEM-2100 transmission electron microscope (TEM) and a FEI Titan G2 60-300 TEM in high angle annual dark field (HAADF)-STEM mode. The cross-sectional TEM foil samples composed of normal direction (ND) and RD were prepared by using a Gatan PIPS 691 ion milling after mechanical grinding. Particularly, geometric phase analysis (GPA) method [26] using the STEM_CELL 2.5 [27,28] was employed to measure and illustrate the strain distribution in HAADF-STEM images with atomic resolution.

3. Results

3.1 Mechanical properties

Fig. 1a shows the microhardness curves of the initial and the ARB sample during isochronal annealing for 1h from 60 °C to 500 °C. With increasing annealing temperature, the microhardness of the initial sample decreases slightly, keeping constant at ~40 HV. However, the ARB sample exhibits apparent annealing hardening response with the maximum hardness of ~75 HV at 190 °C. Fig. 1b shows the stress-strain curves of the initial sample, ARB sample, 190 °C and 250 °C annealed samples, respectively. Stress-strain curve of the initial sample shows low yield stress and large elongation. It is seen that the ARB sample shows a high yield stress of ~250 MPa and an elongation of ~1.2%, whereas obvious increase in both yield stress and total elongation can be seen in the annealed samples. In particular, the ultimate tensile strength (UTS) for the 190 °C annealed ARB sample can exceed 300 MPa, while keeping an elongation of ~2.8% (Fig. 1b).

Fig. 1 (a) Microhardness curves obtained from the initial sample and the ARB sample during isochronal annealing for 1 h from 60 °C to 500 °C; (b) Engineering stress-strain curves of the initial sample, ARB sample, 190 °C and 250 °C annealed samples, respectively.

Fig. 2 (a,b) TEM and HAADF-STEM images showing a lamellar grain structure in ARB sample; (c) HRTEM image showing the atomic structure of the lamellar grain structure.

High magnification HAADF-STEM image of a lamellar grain. The inset FFT pattern corresponding to the white rectangular zone indicates a LAB; (d) A close-up of the cluster (marked by dotted circle) at grain boundary within the green rectangular zone in (c).

3.2 Microstructural characterization

Fig. 2a shows the microstructure of the ARB sample which is characterized by well-defined lamellar structure with a mean lamella thickness of ~ 194 nm, as determined by the line-interception method along the ND (Fig. 3a). HAADF-STEM observation based on atomic number contrast further shows a weak and uniform contrast (Fig. 2b), indicating a homogeneous distribution of solute atoms in the matrix. In the high magnification HAADF-STEM image of a lamellar grain, lots of nano-precipitates can be observed at lamellar grain boundaries (Figs. 2c and 2d), which are similar to that of a previous study [29]. Based on fast Fourier transformation (FFT) analysis, a typical low angle boundary (LAB, yellow line) can be identified within the lamellar grain. However, there is still uniform contrast without apparent indication of solute segregation.

Fig. 3 The distribution maps of boundary spacing in (a) ARB and (b) 190 °C annealed samples.

After annealing at 190 °C for 1h, the microstructure of the annealed ARB sample exhibits a similar lamellar feature (Fig. 4a) with a mean lamella thickness of ~ 200 nm (Fig. 3b), indicating a stable grain structure without apparent grain coarsening. In the

corresponding HAADF-STEM image (Fig. 4b), a continuous bright contrast can be observed along lamellar grain boundaries, suggesting a significant segregation of solute atoms at these grain boundaries.

Fig. 4 TEM and HAADF-STEM images of 190 °C annealed ARB sample showing (a) a lamellar grain structure and (b) solute segregation at lamellar boundaries.

Fig. 5 shows HAADF-STEM images obtained from $[2\bar{1}10]$ direction. As shown in Fig. 5a, the apparent bright contrast can be seen at various grain boundaries. For a LAB segment corresponding to the rectangular zone in Fig. 5b, STEM-EDS mapping strongly evidences clear segregation of Gd atoms. As to another LAB segment with a misorientation angle of 6.7° (Fig. 5c and inset), the detailed Burgers circuit analysis further identifies the dislocation content to be $\langle a \rangle$ and $\langle c+a \rangle$ dislocations. Based on inverse FFT analyses using $(01\bar{1}0)$ and (0002) reflections, two types of dislocations can be clearly observed at the dislocation boundary (Figs. 5d and 5e). The corresponding strain map obtained from GPA analysis using $g=[01\bar{1}0]$ reveals the extension region (in red) and compression region (in blue) around each edge dislocation core (Fig. 5f). Compared with Fig. 5c, it can be observed that most Gd atoms concentrate in the extension region of each dislocation core.

Fig. 5 (a) HAADF-STEM image showing Gd segregation at dislocation structures in the 190 °C annealed ARB sample; (b) HAADF-STEM image and Mg and Gd maps corresponding to the rectangular zone in (a); (c) HAADF-STEM image and corresponding FFT pattern (inset) showing a LAB with Gd segregation at $\langle a \rangle$ and $\langle c+a \rangle$ dislocations; (d,e) The inverse FFT images and (f) GPA strain map showing a periodic array of dislocations at the LAB in (c).

Fig. 6 (a,b) HAADF-STEM images and corresponding FFT pattern (inset) showing Gd segregation at twin structures of the 190 °C annealed sample; (c) SFs with a stacking sequence of “...ABABCBC...” corresponding to the rectangular zone in (b); (d) GPA strain map of the region shown in (b).

Figs. 6a and 6b show HAADF-STEM images of a $\{10\bar{1}1\}$ deformation twin, which can be further identified by FFT analysis (inset in Fig. 6b). The bright contrast along TBs and SFs indicates the segregation of Gd atoms at these defects. High magnification HAADF-STEM image in Fig. 6b shows periodic segregation of Gd atoms at coherent TB (CTB, marked by white solid lines). Gd clusters with bright contrast can also be seen at incoherent TB (ITB, marked by green solid lines) with a

step height of several atom layers. Fig. 6c shows a HAADF-STEM image corresponding to the rectangular zone in Fig. 6b, indicating that SFs with a stacking sequence of “...ABABCBC...”. The GPA strain map shown in Fig. 6d corresponding to the twin in Fig. 6b clearly shows the strain distribution at TBs and SFs. Particularly, it can be seen that most Gd atoms and clusters concentrate around the incoherent TBs with extension strains (in red).

Based on TEM observations in Figs. 4-6, it is seen that high density of Gd segregation can be detected at GBs, TBs and SFs. However, the annealed sample shows a low dislocation density and Gd segregation at dislocations is rarely detected, which may have its cause in the recovery phenomenon by annealing at 190 °C.

3.3 TEM observation of tensile tested sample

To clarify the effect of Gd segregation at defects on deformation, the microstructure of the 190 °C annealed ARB sample after tensile test was characterized by the TEM. High density of dislocations are seen in sample after tensile deformation (Fig. 7). As shown in Fig. 7a, curved non-basal dislocations (marked by green arrows) can be observed within the grain interior. Besides, non-basal dislocations tangled with basal SFs (marked by red arrows) also can be observed within the twin structure (Fig. 7b), implying a strong interaction between non-basal dislocations and basal SFs during tensile deformation.

Fig. 7 TEM images showing non-basal dislocations and SFs within (a) grain interior and (b) twin structure of the 190 °C annealed ARB sample after tensile test.

Fig. 8 (a) HAADF-STEM image showing Gd segregation at SFs and non-basal dislocations; (b) A sketch showing all SFs and dislocations corresponding to (a); (c) HAADF-STEM image and (d) schematic diagram showing the atomic structure of an SF segment; (e) The cross-section of non-basal dislocation within the green rectangular zone in (a).

HAADF-STEM observation was also performed to reveal non-basal dislocations and SFs in the 190 °C annealed ARB sample after tensile test (Fig. 8a). Weak bright contrast can be seen at parallel SFs and the intersected non-basal dislocations (red and green arrows), as schematically illustrated in Fig. 8b. Fig. 8c shows a high-resolution

HAADF-STEM image of a SF segment corresponding to the white rectangular region in Fig. 8a, in which the specific atomic structure can be well determined (Fig. 8d). As to the curved dislocation shown in the green rectangular region in Fig. 8a, the detailed Burgers circuit analysis further identified the dislocation to be $\langle c+a \rangle$ dislocation (Fig. 8e).

4. Discussion

4.1 Gd segregation at various defects

Segregation of solute atoms at TBs usually shows a significant impact on boundary mobility and mechanical properties of magnesium alloys [30,31]. In present study, periodic Gd segregation presents at compressive zone of coherent TBs. On the contrary, high density Gd atoms and clusters appear at incoherent TBs with high extension strain (Fig. 6d). The periodic Gd segregation at coherent TBs (Fig. 6b) is similar to that of previous studies [30-33], which could contribute to boundary energy reduction and microhardness increment. However, the Gd segregation at incoherent TBs shows a totally different segregation behavior (Fig. 6b), i.e. high density Gd segregation presents at extension zone, which may indicate a unique effect on twin boundary migration and mechanical properties [34]. The synergetic effects of various segregation behavior at TBs can better induce the structural stability and strength improvement.

Many SFs with stacking sequence of “...ABABCBC...” were identified within the deformation twins. It is noted that most of the SFs are attached to the TBs with the step height of several atomic layers (Fig. 6b). Linear Gd segregation at SFs can be observed, which may induce a prominent pinning effect on dislocation motion [35]. Similar segregation at SFs has been illustrated to explain the superior strength at elevated temperatures [36]. In addition, the dilute Gd segregation at compressive zone of the SFs is the combined result of chemical and size effects, which is similar to the segregation characteristics at coherent TBs. The segregation behavior and origination of SFs still need further investigation.

In annealed samples, dislocations in LABs show obvious Gd segregation (Fig. 5). High concentration of Gd segregation was formed at the extension zone of the dislocation cores (Fig. 5f), which can well pin dislocation motion resulting in source hardening and high active stress [31,37,38]. In this study, the Gd segregation at TBs, GBs and LABs could significantly contribute to the boundary energy homogenization, which also leads to a weak texture generally observed in Mg-RE alloys [29,39,40].

4.2 Segregation induced high strength and ductility

Segregation hardening phenomenon was usually considered to be negligible in previous studies compared with precipitation hardening of traditional magnesium alloys [30]. In this study, however, a strong segregation hardening response was obtained in nanostructured Mg-3Gd samples (Fig. 1). The Gd segregation contributes to stabilization of nanostructure without apparent grain coarsening (Fig. 4), which is useful to sustain high yield stress by Hall-Petch strengthening.

As to the ductility enhancement of nanostructured magnesium alloys, the inhibition of premature failure caused by deformation twins needs to be taken into consideration. Recent results revealed that active $\langle c+a \rangle$ slip can relax twinning induced stress concentration [13,38]. In present study, not only non-basal slip (Fig. 7) but also clear Gd segregation were detected within twin structure. The synergistic effect of Gd segregation and non-basal slip can effectively release twin boundary stress concentration, which is conducive to ductility improvement. Moreover, the unusual non-basal slip may arise from a lack of active dislocation sources at the initial stage of plastic deformation due to fully pinned basal dislocations by Gd atoms after annealing [41,42]. Therefore, additional non-basal slip is imminently expected, as it is necessary to achieve the five independent slip systems required for an arbitrary deformation. Besides, the high yield stress may also promote the activation of additional slip systems, finally resulting in the inhibition of premature failure and homogeneous deformation.

Furthermore, the interaction between moving non-basal dislocations and other

defects is important for work hardening. Generally, the interplays between dislocations and SFs can either (i) cut or interact with SFs for further plastic deformation or (ii) accumulate around SFs for strain hardening [43-45]. During deformation, parallel SFs not only act as obstacles but also improve the storage and accumulation ability of non-basal dislocations. In our tested tensile samples, non-basal dislocations exhibit a clear segregation of Gd atoms (Fig. 8), indicating a strong interaction of Gd atoms with moving non-basal dislocations. These Gd atoms can drag the moving non-basal dislocations and increase the moving resistance during tensile deformation, which in turn significantly contribute to high strain hardening.

5. Conclusions

To summarize, nanostructured Mg-3Gd alloy with simultaneously high strength and ductility was prepared by ARB processing and subsequent annealing with a synergy of boundary engineering and segregation strategy. Compared with the ARB sample, an apparent hardening response and clear evidences of Gd segregation at various defects, including GBs, TBs, SFs and dislocations, can be observed in the annealed samples. TEM observation of the tensile tested sample well proved the activation of non-basal slip and intensive interaction between dislocations and Gd atoms. The boundary segregation and pinning of dislocations were considered to be the main reason for a significant strength enhancement, while the increase in ductility was attributed to the inhibition of premature failure by Gd segregation at twin boundaries and activation of non-basal slip.

Acknowledgements

This work was supported by the State Key Research and Development Program of Ministry of Science and Technology (MOST) of China (2016YFB0700400), the National Natural Science Foundation of China (No. 51421001, 51971045) and the Fundamental Research Fund of Central Universities of China (Grant No. 2018CDJDCL0019).

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Highlights

- Strong and ductile Mg-3Gd was obtained by boundary and segregation engineering.
- Gd segregation at various defects occurs in annealed ARB sample.
- Strengthening arises from the interaction between Gd and non-basal dislocations.
- Ductility increases due to Gd segregation at TBs and hindering of non-basal slip.

Declaration of competing interest

No potential conflict of interest was reported by the authors.

Journal Pre-proof