Temperature Effects on the Microstructures of Mg–Gd–Y Alloy Processed by Multi-direction Impact Forging

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Abstract

A high strain rate multi-directional impact forging (MDIF) was applied to a solutionized Mg–Gd–Y–Zr alloy in the temperature range of 350–500 °C. Results demonstrate that the dominant deformation mode is twinning at a temperature below 400 °C, whereas at a medium temperature of 450 °C considerable continuous dynamic recrystallization was promoted by {10–12} extension twins. At a higher temperature of 500 °C, twinning activation was suppressed. New DRX grains were observed but their sizes were much bigger than those resulting from the MDIFed 50 passes at 450 °C, which are ascribed to the larger grain boundary mobility and atomic diffusion at 500 °C. Moreover, a non-basal weak texture was gained afterward MDIF at each temperature, which is credited to the MDIF process and the minor strain applied in each pass.

Keywords Magnesium alloys · Twinning · Dynamic recrystallization · Forging · Texture

1 Introduction

Mg alloys attracted significant research interests in aerospace, automotive, power tools, and 3C (i.e., computer, communication, and consumer electronics products) industries [1, 2]. In comparison to the components made from aluminum and iron-based materials, Mg alloys have the capability to reduce their weight by 37% and 78%, respectively [3, 4]. This weight-reducing capacity is eco-friendly and directly related to the decrease in fuel consumption.

In the recent decade, Mg alloys containing Gd and Y specifically (Mg–Gd–Y) have attracted numerous research interests [5, 6], since they possess high strength at the room as well as at ambient temperatures and demonstrate good

creep resistance [7, 8]. To understand their microstructure evolution, several investigations were reported on dynamic recrystallization (DRX) behavior via extrusion [7, 9] rolling [10, 11], and compression [12] processes. Such as Hong et al. [12] confirmed that the long-period stacking-ordered (LPSO) phases play an essential role in the dynamic recrystallization of Mg–11Gd–4Y–2Zn–0.4Zr alloy during the hot compression. Yu et al. [13] observed the increase in the volume fraction of Mg5RE phases along with the improving DRX with the increase in extrusion ratio, while increasing the extrusion temperature decreases the volume fraction of Mg5RE phase and coarsens the DRXed grains. Similarly, Wang et al. [14] confirmed the basal and prismatic slip as dominant, while the pyramidal slip and extension twinning as the complementary deformation modes in Mg–Gd–Y–Zn–Mn alloy sheets processed by large-strain high-efficiency rolling. So far, about multi-directional forging few reports could be found on the temperature effects on microstructure evolution of Mg–Gd–Y alloy which reflects their poor workability during the traditional forging process.

Regarding MDF, the Mg alloys such as Mg–Al–Zn alloys [15], AZ series [16, 17] (RE free Mg alloys) were still concerned via MDF. Wu et al. [18] applied MDF on the ZK21 Mg alloy and due to the different strain rate applied experienced different twinning and DRX behavior. They attributed the island-like ultrafine DRX structure to the higher twinning density while grain coarsening to the

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adiabatic rise in temperature during forging. Miura et al. [15] processed Mg–Al–Zn alloy at room temperature by multi-directional forging and experience the division of initial coarse grains into ultrafine grains by twinning activation. They found twins acting as more preferential nucleation sites than the grain boundaries for recrystallization. Indeed, MDF was also carried out on Mg–Gd–Y alloy [19]; however, with such a low strain rate traditional hydrostatic forging technique, the authors did not perceive twins inside the microstructure which can promote an obvious DRX behavior.

In the present study, we successfully processed a Mg–9.02Gd–4.21Y–0.48Zr wt% (GW94) alloy via high-speed multi-direction impact forging (MDIF) and found it an efficient method to modify the coarse granular structure by twinning activation, which consequently promotes the dynamic recrystallization (DRX) behavior. MDIF also supported the recrystallization process in AZ61 Mg alloy [16], mainly by twin induced DRX behavior and non-basal textures.

In addition, many reports have addressed the topic of strain rate effects on DRX and twinning behavior in Mg alloys [1, 20]; however, little research has been conducted on the temperature effects during the high strain rate forging process especially in Mg–Gd–Y system alloys. Thus, the current work attempts to analyze the effect of deformation temperature on the twinning activity and DRX behavior during the MDIF process which would provide beneficial understanding to obtain a homogeneous and fine-grained Mg–Gd–Y alloy.

2 Experimental

The GW94 Mg alloy samples were extracted in cubic block shape (as-cast ingot) with magnitudes of 70 mm length $\times$ 70 mm width $\times$ 70 mm height. The samples were processed through solutionization treatment at 525 °C for 10 h, which were then cooled in the air down to room temperature.

A hammer (industrial air pneumatic machine) loaded with 400 kg was used to carry out the MDIF process in the open air as mentioned in detail in Ref. [8]. The strain rate was about 50 s⁻¹, and small strain per pass of $\sim$ 0.05 was employed during the MDIF process. Meanwhile, during the MDIF process, the sample direction was changed by 90° every pass and the specimens were forged for 30 passes as shown in the schematic (Fig. 1a). As the pass strain was applied pass by pass from each side, almost no change in the final shape of the samples was observed. In total, four samples were impact forged; however, each one was heated to a different temperature before MDIF, i.e., 350 °C, 400 °C, 450 °C, and 500 °C for 1 h. In addition, the sample forged for 30 passes at 450 °C was extended to 50 passes as exposed

in Fig. 1b. Since MDIF was carried out in an open atmosphere, temperature drop may also occur, however, due to the deformation-induced heating, the temperature loss was not larger than $\sim$ 25 °C of the samples. The specimens for microstructural characterizations were cut from the center of the MDIFed cubic sample. The X-ray diffraction, optical microscopy (OM), and electron backscatter diffraction (EBSD) results were obtained from the central plane perpendicular to the last forging direction as demonstrated in Fig. 1c. Scanning electron microscopy (SEM) model Philips XL 30 ESEM-FEG operational at 20 kV was used to collect the EBSD results. The step size of $\sim$ 0.2 μ m was kept during measuring the EBSD orientation maps. {0002} pole figures were obtained via DIFFRAC plus TEXEVAI software operated by X-ray diffraction Schultz reflection method.

3 Results

3.1 Initial Microstructure

Figure 2a–c reveals the typical optical and SEM microstructures of the as-cast and solutionized GW94 alloy. The as-cast alloy revealed relatively fine grains surrounded by non-equilibrium coarse eutectic β -phases (Fig. 2a). About these non-equilibrium phases including cuboid-shaped particles, we have debated in our previous report [3, 8]. In brief, these non-equilibrium phases were studied in detail by Gao et al. [21] and they confirmed them as Mg₂₄(Gd, Y)₅, by transmission electron microscopy (TEM). The non-equilibrium coarse eutectic phases were almost dissolved in the matrix afterward the solution treatment at 525 °C $\times$ 10 h (Fig. 2b), however, still some cuboid-shaped Mg₅(Gd, Y) [21] particles persisted throughout the microstructure (Fig. 2c). Nevertheless, the same cuboid phases in Mg–Gd–Y alloy were studied by Li et al. [22] and they recognized them as YH₂ and few GdH₂ phases. The average size of grains of the as-solutionized sample increased from $\sim$ 90 to $\sim$ 160 μ m.

3.2 Microstructural Evolution During MDIF at Different Temperatures

The optical microstructures of GW94 alloy with 30 forging passes at temperature 350 °C, 400 °C, 450 °C and 500 °C are illustrated in Fig. 3. As shown in Fig. 3a, b, GW94 alloy MDIFed at 350 °C and 400 °C revealed almost the same microstructure and reflects negligible DRX grains. Twinning seems to be the dominant deformation mode at this temperature, as abundant lenticular twins were observed throughout the microstructure. However, some coarse original grains without twins were also noticed as pointed by the yellow arrows in Fig. 3a, b. At 450 °C, when the alloy was forged, a considerable number of DRX grains appeared

Fig. 1 **a** Schematic diagram of the MDIF process, **b** extension in the forging passes up to 50 at 450 °C, **c** machined out samples position from the center of MDIFed sample

Fig. 2 **a** Cast microstructure of GW94 Mg alloy as shown in OM image, **b** solid solution-treated microstructure, **c** SEM image of solid solution-treated sample

(Fig. 3c) which are likely to reside near the original grain boundaries and also tend to cover the interiors of the grain. After MDIF at 500 °C, almost DRXed microstructure can be found (Fig. 3d), yet the DRX grains are slightly larger than that obtained in sample MDIFed at 450 °C. Overall, the optical microstructures (Fig. 3) exposed that MDIF at a higher temperature such as 450 °C and 500 °C seems more appropriate to achieve a homogeneous microstructure in present GW94 alloy.

In order to analyze the recrystallized or deformed grains, twinning types, and their frequencies, the MDIFed specimens were examined by EBSD measurement. High-angle grain boundaries (HAGBs), low-angle grain boundaries (LAGBs), {10–12} extension, {10–11} contraction, and {10–11}–{10–12} double twinning boundaries are tinted in different colors. The relaxation in angular difference to measure the twin boundaries was considered 5° to the ideal value.

Fig. 3 The optical microstructures of GW94 Mg alloy MDIF 30 passes at different temperatures: **a** 350 °C, **b** 400 °C, **c** 450 °C, **d** 500 °C

Figure 4a, b displays the EBSD IPF map of forged GW94 alloy for 30 passes at 400 °C. Twinning was the main deformation mode as plentiful $\{10\text{--}12\}$ extension twins were detected in the entire microstructure. Lower (CRSS) critical resolved shear stress, i.e., ($\sim 2\text{--}3$ MPa) for $\{10\text{--}12\}$ extension twins may contribute to their existence [23, 24]. In order to explore the microstructure in detail, magnified images are selected in Fig. 4a, b which reveals the parallel twins in some particular regions (Fig. 4c) that are deviated by almost $\sim 86^\circ$ from the matrix. Indeed, the following results are dissimilar with the results obtained in Ref. [25] that $\{10\text{--}12\}$ extension twins including DRXed grains were obtained, however, in this case, the $\{10\text{--}12\}$ extension twins contributed in another way, i.e., they divided effectually the initial coarse grains. Afterward, plenty of LAGBs formed in those twins, accordingly splitting them into numerous parts (Fig. 4d). Besides, the LAGBs in twins few LAGBs parallels with the twin boundaries (TBs) were also noticed (Fig. 4e), thus reflecting not only the partition and finally refinement of the original coarse grains but also specifies an interaction between the $\{10\text{--}12\}$ extension twin boundaries and dislocation slips [26]. Moreover, the grain occupied by LAGBs, i.e., subgrain converted to the new DRX grains with increasing strain has been proposed before in Mg alloys [26].

Indeed, very few regions with fine DRX grains ($3\text{--}5\text{ }\mu\text{m}$) were observed; however, they seem to be twin intersected regions as some residual twin boundaries can be identified beside the DRX grains (Fig. 4f), which has been suggested before to provide effective nucleation sites for recrystallization [27, 28]. Generally, after MDIF 30 passes at 400 °C, a dense twinned microstructure without significant recrystallization was obtained. The $\{10\text{--}12\}$ extension twins vividly divided the coarse initial grains and new DRX grains tends to nucleate at the twinning regions; however, the alloy fails to obtain a fully recrystallized structure and display an incompleteness of the recrystallization process.

The EBSD observations of GW94 alloy at 450 °C after 30 and 50 forging passes were also studied in detail. The comprehensive descriptions about the sample MDIFed for 30 and 50 passes can be found in our previous work [8] while for the purpose of assessment, we will discuss it briefly here. In comparison to the sample MDIFed at 400 °C (Fig. 4), the frequency of the twin boundaries at 450 °C was 12.5%, which is almost double to 7.5% at 400 °C, but almost all the twinning boundaries were recognized $\{10\text{--}12\}$ extension twins. With the increase in forging passes up to 50, the rate of recurrence of those $\{10\text{--}12\}$ extension twins decreases from 12.5 to 3.5%, while a lot of fine DRX grains were also

Fig. 4 **a** Inverse pole figure map, **b** corresponding grain boundary map of the GW94 alloy after MDIF 30 passes at 400 °C, whereas **c–f** the enlarged IPF maps selected in **a**, **b** displaying the formation of extension twins and low-angle grain boundaries

observed [8]. Indeed, the beneficial $\{10\text{--}12\}$ extension twin promoted the DRX behavior during the MDIF process at 450 °C for 30 passes and following 50 forging passes with almost very few twins, the fine DRX grains percent rises up to 25% and occupied most of the microstructure [8]. However, recrystallization process in the present GW94 alloy, even at 450 °C, seems much sluggish than that initiated by the double and contraction twins in AZ61 alloy [16] as a complete recrystallized microstructure was obtained after MDIF 30 passes, following the same forging technique [16].

Figure 5a, b represents the IPF maps of GW94 alloy forged for 30 passes at 500 °C. An inhomogeneous relatively coarse grain microstructure containing very few

twinned grains was obtained after MDIFed for 30 passes at 500 °C. Evidently, the same high strain rate forging process was followed at 500 °C but the absence of significant twin induces DRX because high temperature may result in a relatively coarse-grained inhomogeneous microstructure. Certainly, the 500 °C temperature suppressed the occurrence of twinning during the MDIF; however, few twins were still witnessed (Fig. 5c) which could be attributed to the high strain rate MDIF. In fact, MDIF offers very limited time, during which the activated slips may not alter the crystal orientation suddenly, and however, twinning deviates it abruptly and tends to accommodate the induced strain [29]. Indeed, many LAGBs were detected in

Fig. 5 **a** Inverse pole figure map, **b** corresponding grain boundary map of the GW94 alloy after MDIF 30 passes, where **c–e** the enlarged IPF maps selected in **a**, showing few twins and lots of LAGBs in coarse grain with LAGBs and new DRX grains

some deform grains (Fig. 5d), which reveals the increased level of dynamic recovery (DRV) because of a higher dislocation slipping behavior at 500 °C and hence assist the grain refinement process. Moreover, new grains with HAGBs containing subgrain were also observed (Fig. 5e), which will be transformed progressively into a dynamically recrystallized grain with proceeded deformation (Fig. 5f). Generally, in the deformed structure, LAGBs come from the accumulated dislocations [4]. With the increase in strain, those LAGBs may absorb additional dislocations and transform into HAGBs, i.e., typically a CDRX process [4]. Nevertheless, it should be noted that the average size of those recrystallized grains at 500 °C is approximately ~15 μm, i.e., almost five times greater than that the size of DRX grain (2–3 μm) obtained after 50 passes at 450 °C. Higher grain boundary mobility and diffusion behavior at 500 °C should lead in higher driving

energies, thus resulting in a coarser grain structure than the prior ones [30].

3.3 Texture

Figure 6a–c demonstrates the pole figures of GW94 alloy forged at different temperatures. Generally, a strong basal texture formed during the deformation, e.g., rolling or extrusion, because of the widespread instigation of extension twins and basal slips in wrought Mg alloys [28]. Nevertheless, in the present investigations, a non-basal weak texture was obtained after each MDIF process at a different temperature which could be attributed to the small pass strain and changes in the forging plane at each pass during the MDIF process.

Moreover, twinning may also affect the process of texture development. If the compression stress is perpendicular

Fig. 6 {0002} Pole figures of MDIFed GW94 alloy MDIF for 30 passes: a 350 °C, b 400 °C, c 500 °C

to the *c*-axis of the (hexagonal close pack) hcp unit cell, {10–12} extension twin occurs within a parent grain, ensuring a change in crystal orientation about $\sim 86^\circ$. The basal plane of that particular twinned grains after {10–12} twinning should rotate by $\sim 86^\circ$, which means precisely the basal plane almost perpendicular and *c*-axis parallel to the last forging direction (LFD). Hence, on the bases of arbitrary-oriented initial grains (solutionized conditions), randomly directed twins were produced that consequently developed a non-basal texture during the process of MDIF. Additionally, strain employment during the deformation also has a significant effect on texture formation. Wu et al. [31] found a basal texture in Mg–Gd–Zn alloy even after MDF process and credited it to the large strain employed ~ 0.5 in the final pass of the forging process. However, Yang et al. [32] observed the rapidly amplified grain rotation at the strain greater than 0.1 after hot compression of the AZ31 alloy, thus signifying that deformation by large strain in unidirectional compression source strong basal texture. Therefore, the present results suggest such a minor strain per pass ~ 0.05 combined with the multi-direction forging results in a non-basal texture after 30 passes at each temperature.

4 Discussion

The present experiments reveal evidence for average grain size and twin percentage during the MDIF carried out at different temperatures as shown in Fig. 7. At 400 °C, only {10–12} extension twins were initiated by the MDIF process. The activated twins proficiently segmented and subdivided the parent grains and performed an essential role to offer an additional mode to accommodate the strain [29]; however, they did not succeed in creating an obvious DRX, indicating insufficient driven thermal energy at 400 °C. Meanwhile, twin-assisted continuous DRX mainly

Fig. 7 Relationship among average grain size, twins percentage and forging temperature of MDIFed GW94 alloy obtained from EBSD data. The region inscribed by yellow circle that reveals the twin's percentage and average grain size at MDIF 50 passes

contributed to the grain refinement at 450 °C [8]. Generally, in Mg alloys, {10–12} extension twins are believed to accommodate the internal stresses and grow easily during the deformation process [33]. In comparison to {10–11} contraction and {10–11}–{10–12} double twins, {10–12} extension twins are not considered as influential to DRX nucleation [34–36]. However, with a further increment of forging passes up to 50, the MDIF process was progressively dominated by a {10–12} extension twin-assisted dynamic recovery process that consequently consumed most of the deformed microstructure and almost a fine grain recrystallized microstructure was obtained (Fig. 7). Certainly, the 500 °C temperature suppressed the occurrence of twinning during the MDIF process. The twinning suppression results in a relatively large average grain size, while twins as influential sites for DRX were also reduced. Indeed, new DRX

grain appeared; however, their sizes were much bigger than the ones created after 50 forging passes at 450 °C. With regard to their relatively large average size at 500 °C, Xia et al. [30] proposed that it is governed by Zener–Hollomon parameter, i.e.,

$$Z = \dot{\epsilon} \exp(Q/RT), \quad (4.1)$$

where $\dot{\epsilon}$ is strain rate, Q is activation energy, and R and T are ideal gas and temperature constants, respectively. The Eq. (4.1) suggests that the average size of recrystallized grains will increase with the rise in temperature. Additionally, solute concentrations of RE elements at the GBs or TBs in Mg alloys may also affect their recrystallization process [37]. They tend to retard their mobilities during the recrystallization, which are overcome by sufficient thermal energy during the deformation process [38, 39]. Therefore, consistent with Ref. [40], the diffusion rate of RE in Mg above 400 °C is around $\sim 3 \times 10^{-15}$ m²/s, which is almost thirty times greater than the diffusion rate calculated at 350 °C and must be larger at 500 °C. Therefore, the mentioned Zener–Hollomon parameter and the increased solute diffusion behavior at 500 °C may result in relatively large DRX grains after MDIF.

Furthermore, usually slips and twinning compete during the deformation at elevated temperature, and also, the CRSS values for non-basal slips decrease intensively with the increasing temperature. As reported before in Refs. [33, 41], the pyramidal slip system is easy to be activated above 300 °C, while it occurs more significantly above the 400 °C. In contrast, the grain size, strain rate, also temperature largely affect the twin formation. Indeed, the strain rate during MDIF at each temperature was almost the same but deformation at low temperatures mostly favors twinning while the decreasing grain sizes diminish their formation due to the Hall–Petch effect. i.e., specifically, the twins are difficult to be activated at reduced grain size [42–45]. Therefore, at the lower temperatures such as 350 °C and 400 °C, twinning was the dominated deformation behavior, however, at the intermediate temperature, i.e., 450 °C beside the twinning activation, twin-induced DRX also occurred which significantly contributed to the grain refinement up to 50 forging passes as specified by the yellow circle in Fig. 7. While, during MDIF at 500 °C, non-basal slips were gradually activated due to its decreasing CRSS. It has been well known that due to an increase in the dislocation slips a greater amount of internal energies accumulate, which might perform a key role in DRX nucleation [46]. DRX promoted by different dislocation slips was also studied deeply before [47] and witnessed by Koike et al. [33, 48] in the AZ31 alloy. In addition, the dislocation lines related to the basal slips were also found interrelated to the LAGBs in Ref. [49]. The same LAGBs were also observed after the MDIF 30 passes

at 500 °C which surely contributed to the grain refinement process. Thus, after the MDIF 30 passes at 500 °C, due to the higher temperature, the slipping overawed the dominant twinning behavior. However, due to the higher temperature, the equiaxed DRX grains experienced substantial growth, which deteriorates to obtain a fine grain microstructure.

5 Conclusions

1. The {10–12} extension twins were the dominant deformation mode after the MDIF carried out at temperatures lower than 450 °C, and the onset of dynamic recrystallization was negligible.
2. At the temperature of 450 °C, the {10–12} extension twins divided the initial coarse grain and promoted continuous DRX behavior. With the extension in forging passes up to 50, the twin matrix laminates gradually converted into fine DRX grains.
3. After MDIF at 500 °C, the twinning formation was suppressed due to a higher temperature. New DRX grains were observed, but their sizes were much bigger than the ones formed after 50 passes at 450 °C which is credited to the larger diffusion rates and grain boundary mobilities at 500 °C.
4. A non-basal weak texture with low intensity was obtained after MDIF at each temperature, which is attributed to the multi-directional forging method and the small strain employed in each pass.

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