Effects of Passes on Microstructure Evolution and Mechanical Properties of Mg–Gd–Y–Zn–Zr Alloy During Multidirectional Forging

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Received: 22 March 2018 / Revised: 27 April 2018 / Published online: 21 June 2018
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Abstract

The multidirectional forging (MDF) process was conducted at temperature of 753 K to optimize the mechanical properties of as-homogenized Mg–13Gd–4Y–2Zn–0.6Zr alloy containing long-period stacking ordered phase. The effects of MDF passes on microstructure evolution and mechanical properties were also investigated. The results show that both the volume fraction of dynamic recrystallization (DRX) grains and mechanical properties of the deformed alloy enhanced with MDF passes increasing till seven passes. The average grain size decreased from 76 to 2.24 μm after seven passes, while the average grain size increased to 7.12 μm after nine passes. The microstructure after seven passes demonstrated randomly oriented fine DRX grains and larger basal (0001)<11 $\bar{2}$ 0> Schmid factor of 0.31. The superior mechanical properties at room temperature (RT) with ultimate tensile strength (UTS) of 416 MPa and fracture elongation of 4.12% can be obtained after seven passes. The mechanical properties at RT after nine passes are inferior to those after seven passes due to the coarsening of DRX grains, which can be ascribed to the static recovery resulting from the repeated heating at the interval of MDF passes. The elevated temperature mechanical properties of the deformed alloy after seven passes and nine passes were investigated. When test temperature was below 523 K, the elevated temperature tensile yield strength and UTS after seven passes are superior to those after nine passes, while they are inferior to that after nine passes as temperature exceeds 523 K.

Keywords Mg–Gd–Y–Zn–Zr · Multidirectional forging · Microstructure · Mechanical properties

1 Introduction

The magnesium alloy has been widely employed as structural components in the aerospace and automotive fields due to its higher specific strength, recyclability and creep resistance [1–4]. However, the further application of the magnesium alloy has been limited due to the poor ductility and strength at RT, which was related to the hexagonal close-packed (HCP) crystal structure of magnesium alloy [5–8]. Recently, the magnesium alloy

containing rare earth (RE) element has attracted considerable researchers' focuses due to its superior elevated temperature and creep behavior than conventional magnesium alloy [9–11]. Particularly, the Mg–Gd–Y–Zn–Zr alloy demonstrates the superior mechanical properties due to precipitation of the second phase with excellent thermal stability and strengthening effect of long-period stacking ordered (LPSO) phase [12–15]. Furthermore, the kink mechanism of intragranular LPSO phase can accommodate strain, which can also contribute to the better mechanical properties of the Mg–Gd–Y–Zn–Zr alloy [16–19].

It is universally acknowledged that the refined grains can effectively improve the mechanical properties at RT and considerable researches are devoted to this field. The severe plastic deformations (SPDs), such as equal-channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll-bonding (ARB), as well as multidirectional forging (MDF), demonstrate a better grain refinement effect than conventional plastic deformation process. Numerous researches concluded that the grains were

Available online at <http://link.springer.com/journal/40195>

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effectively refined due to the dynamic recrystallization (DRX) during the SPD process. Sun et al. [20] found that the initial net-like $\text{Mg}_3(\text{Gd}, \text{Y})$ phases in the cast alloy were effectively broken into small particles with a size of 1–10 μm due to the high strains caused by HPT. Liu et al. [21] investigated the ECAP process of $\text{Mg}_{96}\text{Gd}_3\text{Zn}_{0.5}\text{Ni}_{0.5}$ and concluded that discontinuous dynamic recrystallization (DDRX) firstly occurred at the interface of block 14H-LPSO phases, and the continuous dynamic recrystallization (CDRX) operated at the kink bands of intragranular 14H-LPSO phase. Huang et al. [22] investigated the microstructure and mechanical properties of AZ31 magnesium alloy processed by MDF and concluded that the mechanical properties improved significantly after MDF. Xia et al. [23] investigated the MDF process of coarse-grained Mg-Gd-Y-Nd-Zr alloy and proposed that the volume fraction of the fine recrystallized grains was remarkably increased and grains were significantly refined after six passes. They also concluded that the weakened basal texture can be attributed to the combined effect of dynamic recrystallization, dynamic precipitation and variation of MDF direction. Wu et al. [24] studied the MDF of ZK21 magnesium alloy at different strain rates and found that the novel mixed structure of honeycomb-like coarse DRX grains and island-like ultrafine DRX grains can be obtained at higher strain rate, which can result in the substantial improvements in mechanical properties. Tang et al. [25] found that the Mg-Gd-Y-Zr alloy after six passes demonstrated a superior balance of tensile strength and ductility at RT with ultimate tensile strength (UTS) of 336 MPa and fracture elongation of 21%. Zhu et al. [26] investigated the effects of MDF passes on high-purity aluminum grains and structure uniformity resulting from deformation uniformity and the grain refinement limitation with larger strain. Nakao and Miura [27] investigated the MDF of the austenitic stainless steel and concluded that the grain size was associated with the MDF temperature and the accumulative strain. However, limited researches have been conducted on MDF of Mg-RE alloy with intragranular 14H-LPSO phase and its effect on the microstructure and mechanical properties.

The present work focuses on the effect of MDF passes on the microstructure evolution and mechanical properties of the as-homogenized Mg-Gd-Y-Zn-Zr alloy. Furthermore, the study also illustrates the strengthening mechanism of MDF on the as-homogenized Mg-Gd-Y-Zn-Zr alloy. The results could be the basis of further investigation of the ultrafine grain Mg-Gd-Y-Zn-Zr alloy.

2 Materials and Methods

The as-received material was as-cast $\text{Mg-13Gd-4Y-2Zn-0.6Zr}$ alloy, and its chemical composition is given in Table 1. The as-received material was homogenized at 793 K for 16 h, followed by air cooling.

Furthermore, the billets and dies were heated to 753 and 723 K, respectively, and then reserved for 30 min before MDF. A mixture of graphite and oil was used as lubricant to reduce the friction force between the billets and dies. The billet with an initial size of 30 mm $\times$ 30 mm $\times$ 50 mm was machined from the homogenized alloy. The die for the MDF was constrained mold with cubic mold cavity. The size of the cross section of the mold cavity was 30 mm $\times$ 50 mm, and thus, the size of billet after each pass becomes 30 mm $\times$ 50 mm $\times$ 30 mm. The MDF process was conducted on pressing machine with 200 t capacity and involved the sequential forging along three orthogonal directions, as shown in Fig. 1. The samples obtained from the former pass were reheated to forging temperature before the subsequent pass and then reserved for 20 min. The true strain of each pass was 0.51 calculated through $\varepsilon = \ln(H_0/H)$, where H_0 and H are initial and final height of samples, respectively. The tensile specimens at RT with gauge length of 10 mm were electrical discharge machined from the center of the samples with air cooling after each pass, as shown in Fig. 2a, and the elevated temperature tensile samples were machined from the center of deformed alloy after seven passes and nine passes. The samples were quenched in water immediately after each pass to investigate the microstructure. The location for microstructure observation was selected on the perpendicular to the last pass forging direction, as demonstrated in Fig. 2b.

The optical microstructure (OM) was observed using Leica DMI3000M optical microscopy, and the OM samples were chemically etched in the solution of 5 g picric acid, 5 ml alcohol, 100 ml acetic acid and 10 ml distilled water. The grain size was measured through Image-Pro 5.0 software. The scanning electron microscope (SEM) examination was conducted using Carl Zeiss Merlin Compact, which was equipped with an electron backscatter diffraction (EBSD) system. The specimens for the SEM and EBSD observation were prepared by electro-polishing in the solution of perchloric acid and alcohol with the volume ratio of 1:9. The EBSD results were collected with scanning step size of 1.2 μm . The X-ray diffraction (XRD) using $\text{CuK}\alpha$ radiation was conducted on the ESCLAB 250Xi to analyze the phase constitution. The tensile tests were carried out on Instron-5569 tensile test machine with rate of 1 mm/min at RT and Shimadzu Autograph AG-X Plus tensile test machine with a rate of 2 mm/min at

Table 1 Nominal composition of Mg–13Gd–4Y–2Zn–0.6Zr alloy (wt%)

Element	Gd	Y	Zn	Zr	Cu	Fe	Si	Mg
Content	12.99	4.00	2.00	0.60	0.0060	0.0014	0.0042	Bal.

Fig. 1 Schematic diagram of multidirectional forging process

elevated temperature (ET), respectively. The results reported in the work were calculated from the average value of three repeated tensile tests.

3 Results and Discussion

3.1 Effect of Passes on Microstructure Evolution

The optical microstructures of the as-cast Mg–13Gd–4Y–2Zn–0.6Zr are illustrated in Fig. 3. The microstructure of the as-cast Mg–13Gd–4Y–2Zn–0.6Zr alloy comprises of equiaxed α -Mg matrix with statistically average grain size of 76 μm , the eutectic $\text{Mg}_3(\text{Gd},\text{Y})$ phase, intragranular 14H-LPSO phase and interdendritic 18R-LPSO phase, as well as the precipitated $\text{Mg}_5(\text{Gd},\text{Y})_{24}$ phase [28–30]. The

intragranular thin-platelet lamellar structure can be observed, which precipitates from the α -Mg supersaturated solid solution at high temperatures [29]. The as-cast Mg–13Gd–4Y–2Zn–0.6Zr alloy was homogenized at 793 K for 16 h, followed by air cooling. The optical microstructures of the as-homogenized Mg–13Gd–4Y–2Zn–0.6Zr are demonstrated in Fig. 4. The volume fraction of eutectic $\text{Mg}_3(\text{Gd},\text{Y})$ phase declined and interdendritic 14H-LPSO phase formed due to the dissolution of eutectic $\text{Mg}_3(\text{Gd},\text{Y})$ phase and the transformation from interdendritic 18R-LPSO phase to interdendritic 14H-LPSO phase during the homogenization process [29, 30]. The kink mechanism of 14H-LPSO phase can strengthen the alloy and improve the ductility through inhibiting the initiation and propagation of micro-cracks [29].

Figure 5 shows the XRD analysis of as-cast, as-homogenized and deformed alloy after seven passes. The XRD patterns verified the existences of LPSO phase and precipitated $\text{Mg}_5(\text{Gd},\text{Y})_{24}$ phase in as-cast alloy, as-homogenized and deformed alloy. Furthermore, the homogenized alloy contains more LPSO phase and precipitated $\text{Mg}_5(\text{Gd},\text{Y})_{24}$ phase. Figure 6 illustrates the microstructures of deformed alloy after different MDF passes. Figure 6a demonstrates that the typically bimodal microstructure and primary grains are surrounded with finer DRX grains, indicating that DDRX operates at grain boundaries. The 14H-LPSO can inhibit the lattice rotation and dislocation movement, and the CDRX was suppressed within the deformed grains; thus, the DDRX predominately operates at grain boundaries at lower accumulative strain. Moreover, the interdendritic 14H-LPSO phases were crushed and broken, which can partially contribute to the DRX process through particle-stimulated nucleation (PSN)

Fig. 2 The location for **a** tensile test at RT and ET, **b** microstructure observation

Fig. 3 Optical microstructure of the as-received Mg–Gd–Y–Zn–Zr alloy **a** at low magnification, **b** at high magnification

Fig. 4 Optical microstructures of as-homogenized Mg–Gd–Y–Zn–Zr alloy **a** at low magnification, **b** at high magnification

Fig. 5 XRD patterns of as-cast, as-homogenized and deformed alloy after seven passes: **a** the whole section, **b** the amplified part section of XRD patterns

mechanism [31]. Moreover, the intragranular lamellar 14H-LPSO phases were kinked severely and broken after five passes, as demonstrated in Fig. 6b. Figure 6c shows

the optical microstructure after seven passes. The DRX grains were significantly refined and the average grain size is 2.24 μm as accumulative strain increases to 3.57. The

Fig. 6 Microstructures of deformed alloy after: **a** three passes, **b** five passes, **c** seven passes, **d** nine passes during MDF

broken 14H-LPSO can contribute to the DDRX process through the PSN mechanism, and the reduced inhibition effect of 14H-LPSO phase on lattice rotation facilitates the CDRX process [29, 31]. The residual interdendritic 14H-LPSO phases were distributed at the grain boundaries. However, the statistically average grain size increased up to 7.12 μm , and the intragranular 14H-LPSO and equilibrium β phase ($\text{Mg}_5(\text{Gd,Y})$ phase) precipitate [29] after nine passes, as shown in Fig. 6d, which was due to the static recovery resulting from the repeated heating at the interval of passes.

The inverse pole figure (IPF) maps, pole figures (PF) and Schmid factor (SF) distribution maps of the samples after different MDF passes are illustrated in Fig. 7. The misorientation measured along the black line in primary grain after five passes is demonstrated in Fig. 8, and distribution of crystallographic orientations after different MDF passes is demonstrated in Fig. 9. Figure 7a shows that a great majority of residual coarse grains demonstrate

the basal $\langle a \rangle$ texture after three passes, which can be attributed to that the basal slip system predominately operates due to the lower critical resolved shear stress (CRSS) and continuous slip on basal slip systems results in the rotation of basal slip planes toward perpendicular of compression direction (CD); thus, the c -axis of most deformed grains aligns parallel to CD [29]. The basal $(0001)\langle 11\bar{2}0 \rangle$ Schmid factor after three passes reaches 0.26. For the majority of the deformed grains after five passes, the prismatic slip plane rotates toward normal of CD. The texture density declined slightly to 5.69 and the Schmid factor increased apparently to 0.30 after five passes. The intragranular 14H-LPSO phase can inhibit the movement of basal dislocation; thus, the dislocations cluster along intragranular 14H-LPSO phase as accumulative strain increases. The distinct misorientation can be observed within the primary grains, suggesting that the dislocations are concentrated to form the dislocation cell and thus sub-grains form, which indicates the operation of

Fig. 7 IPF maps **a–d**, pole figures **e–h** and basal (0001) Schmid factor **i–l** distribution of the samples after different MDF passes: **a, e, i** three passes, **b, f, j** five passes, **c, g, k** seven passes, **d, h, l** nine passes

Fig. 8 After five-pass MDF **a** the IPF maps, **b** misorientations measured along the black line in primary grain

CDRX, as illustrated in Fig. 8. In addition, the fraction of low-angle grain boundaries (LAGBs) has increased to 43.38% and fraction of high-angle grain boundaries (HAGBs) declined to 25.28%, which indicates the formation of numerous sub-grain boundaries and operation of CDRX at higher accumulative strain. The intragranular lamellar 14H-LPSO can be zigzagged and broken as accumulative strain increases, which contributes to the DDRX process through the PSN mechanism [31]. The fraction of DRX grains increased significantly after seven passes, and the fine DRX grains demonstrate the random crystallographic orientation, as revealed in Fig. 7c. Furthermore, the texture density declined to 1.70 and the fraction of HAGBs increased to 68.19% after seven passes, as illustrated in Figs. 7k and 9c, which can be ascribed to

the complete DRX process at higher accumulative strain. However, the statistically average grain size increased to 7.12 μm due to the growth of DRX grains and the texture density increased to 3.15 after nine passes. Xia et al. [23] concluded that average grain size declined to 5.1 μm at accumulative strain of 1.5 and then varied slightly for the Mg–Gd–Y–Nd–Zr alloy without 14H-LPSO phase; however, the finer DRX grains were obtained as accumulative strain increased to 3.57 in this study. It can be deduced that intragranular lamellar 14H-LPSO phase can effectively inhibit the DRX process during MDF, which was consistent with the previous research [31].

Fig. 9 Distributions of grain boundary orientation after **a** three-pass, **b** five-pass, **c** seven-pass, **d** nine-pass MDF, **e** misorientation variation for different types of boundaries after different passes

3.2 Effect of Passes on Mechanical Properties

Figure 10 shows the tensile properties of as-cast, as-homogenized and deformed alloy with different MDF passes at RT. The mechanical property at RT for the homogenized alloy was superior to that of the cast alloy, as demonstrated

in Fig. 10. The intragranular 14H-LPSO phase and interdendritic 14H-LPSO phase were remained, and brittle eutectic $\text{Mg}_3(\text{Gd,Y})$ phases at grain boundaries dissolved during the homogenization process, which corresponds to the slight improvement in tensile strength and significant

Fig. 10 Mechanical properties of deformed alloy with different MDF passes

enhancement of fracture elongation during homogenization process.

The tensile properties of the deformed alloy at RT were significantly improved with further passes, as revealed in Fig. 10, which can be attributed to strengthening effect of finer grains. The tensile yield strength (TYS) and UTS at RT after three passes are enhanced compared with the as-homogenized alloy, while the fracture elongation increases slightly. Huang and Zhang [22] ascribed it to the development of high-density twins at lower accumulative strain, and the cracks prone to nucleate at twin boundaries. However, the twins are hardly observed in Fig. 6a, which was due to the inhibition effect of intragranular 14H-LPSO phase on twinning [32]. Furthermore, it is documented that the TYS at RT was related to the operation of basal slip system [32]. The majority of coarse deformed grains demonstrate the stronger basal texture and the basal (0001) plane of deformed grains are parallel to the tension direction; thus, the stress for operation of (0001)<11 $\bar{2}$ 0> slip system increased and thus the TYS increased. Although the volume fraction of DRX grains increased, the basal (0001)<11 $\bar{2}$ 0> Schmid factor was 0.26, and the basal slipping system predominately operates for the Mg-RE alloy, which may result in the approximate fracture elongation compared with the as-homogenized alloy. The deformed alloys after three passes and five passes exhibit the approximate TYS and UTS, while the fracture elongation of the deformed alloy after five passes significantly increases. The prismatic slip plane rotates toward normal of normal direction (ND), which can facilitate the operation of basal slip system at early stage during tensile test; thus, fracture elongation increases after five passes. Besides, the fraction of intragranular 14H-LPSO declined and inhibition effect on the dislocation movement reduced; thus, the TYS after five passes should decrease. The basal (0001)<11 $\bar{2}$ 0> Schmid factor increased to 0.30 after five passes, as revealed in Fig. 7j, which may contribute to the

improvement in fracture elongation and the decline of TYS. However, the volume fraction of DRX grains increased and the fine-grain strengthening effect may compensate for the decline of TYS; thus, the deformed alloy after three passes and five passes showed approximate TYS and UTS. It should be noted that the TYS, UTS and fracture elongation reach their maximum (342 MPa, 416 MPa and 4.12%) after seven passes, which was partially attributed to the strengthening effect of finer DRX grains and weakened texture, as indicated in Fig. 7c, g. Furthermore, since the basal slip system dominates over prismatic and pyramid slip system at RT for the magnesium alloy, the macroscopic plastic strain can be accommodated by the fine DRX grains with relatively random orientation [33, 34]. The Schmid factor reaches its maximum to 0.31 and the fraction of HAGBs increases to 68.19%, as revealed in Figs. 7k and 9c, which is consistent with the superior mechanical properties after seven passes. The strengthening effect of finer grains can compensate for the reduced effect of higher Schmid factor on TYS and UTS; thus, the TYS and UTS after seven passes demonstrate limited improvement compared to that after five passes. Furthermore, the intragranular thin-platelet 14H-LPSO phase can effectively hinder the dislocation movement and consequently strengthen the alloy in certain extent [29]. However, the mechanical properties at RT after nine passes declined compared to that after seven passes. Similarly, the TYS of AZ31 alloy firstly increases till the MDF passes increase up to nine passes, and then reduces with further MDF passes [22]. Wu et al. [24] draw the similar conclusion and ascribe it to the TYS responses. In correlation with Figs. 6 and 7, the grain-refining strengthening effect contributes little to the strength improvement and the statistically average grain size increases to 7.12 μ m with further MDF pass. Furthermore, the fracture elongation also demonstrates the similar trend. Substantial amount of β' precipitates can be observed within the DRX grains after nine passes, as demonstrated in Fig. 6d. The dynamic precipitates exhibit unspecific crystallographic orientation relationship and remain incoherent with the α -Mg matrix [29, 35]. The micro-cracks may incline to initiate around these incoherent precipitates, which results in the inferior ductility of the deformed alloy after nine passes [29]. Furthermore, the basal (0001)<11 $\bar{2}$ 0> Schmid factor decreased to 0.27, as revealed in Fig. 7l, which may partially account for the inferior ductility of the deformed alloy after nine passes.

The Mg-Gd-Y-Zn-Zr alloy was mostly employed at ET of 473–573 K; thus, it is of vital importance to investigate the elevated temperature mechanical properties of deformed alloy. The elevated temperature stress-strain curves of the deformed alloy after seven passes and nine

Fig. 11 Elevated temperature mechanical properties of the deformed alloy after **a** seven passes, **b** nine passes

Table 2 Elevated temperature mechanical properties of deformed alloy after seven passes and nine passes

MDF passes	Tensile temperature (K)	Tensile yield strength (0.2%) (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
Seven passes	473	292	390	12.7
	523	200	263	44.7
	573	92	147	142
Nine passes	473	267	349	11.9
	523	204	279	27.3
	573	121	174	80.3

passes are illustrated in Fig. 11. The mechanical properties at ET after seven passes and nine passes are demonstrated in Table 2. Results indicated that the TYS, UTS and fracture elongation after seven passes were 292 MPa, 390 MPa and 12.7% at test temperature of 473 K, where the fracture elongation was superior to that at RT. The deformed alloy after seven passes and nine passes demonstrated UTS of 263 MPa and 279 MPa and the fracture elongation of 44.7% and 27.3% at 473 K, respectively. The better elevated temperature mechanical properties can be ascribed to the fine DRX grains and large number of β' precipitates with superior thermal stability, which can inhibit dislocation movement. Furthermore, the TYS and UTS of the deformed alloy after seven passes and nine passes decreased and the fracture elongation improved with temperature increasing as the test temperature exceeds 473 K. The operations of prismatic $\langle a \rangle$ and pyramid $\langle c + a \rangle$ slipping system and the enhancement of solution atom mobility contribute to the decline of the TYS and improvement in fracture elongation as test temperature increases [33]. The elevated temperature mechanical properties of deformed alloy after seven passes were superior to that after nine passes at 473 K, which was due

to the randomly oriented finer DRX grains after seven passes. However, the deformed alloy after nine passes exhibits the higher TYS and UTS compared with those after seven passes as temperature exceeds 523 K. The grain boundary sliding and multiple slip can operate for the fine grains at elevated temperature, and the dislocation was quickly annihilated at grain boundaries due to the finer grains [35, 36]. The grain boundary sliding predominately operates to accommodate the grain deformation, and finer grains prefer to rotate at higher temperature; thus, the elevated temperature UTS and TYS after seven passes are inferior to those after nine passes as test temperature exceeds 523 K.

4 Conclusions

The study investigates the effect of the MDF process on the microstructural evolution and the mechanical properties of the homogenized Mg–13Gd–4Y–2Zn–0.6Zr alloy containing LPSO phase. The conclusions are summarized as follows:

- (1) The microstructure of the deformed alloy demonstrates the bimodal microstructure till five passes and fraction of DRX grains increased with further MDF pass. The fraction of fine DRX grains was significantly improved and homogeneous microstructure with statistically average grain size of 2.24 μm was obtained after seven passes. Due to the static recovery resulting from the repeated heating at the interval of passes, the statistically average grain size increased to 7.12 μm after nine passes.
- (2) The supreme mechanical properties with UTS of 416 MPa and fracture elongation of 4.12% were acquired after seven passes, which can be ascribed to the homogenized microstructure with finer randomly oriented DRX grains. The basal (0001)<11 $\bar{2}$ 0> Schmid factor reached its maximum to 0.31, which was consistent with the superior fracture elongation after seven passes.
- (3) The randomly oriented finer DRX grains after seven passes are responsible for the better elevated temperature mechanical properties at 473 K compared with nine passes. The grain boundary sliding predominately operates to accommodate the grain deformation and finer grains prefer to rotate at higher temperature; thus, the elevated temperature UTS and TYS after seven passes are inferior to those after nine passes as test temperature exceeds 523 K.

Acknowledgements This work was sponsored by the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry.

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