

Effect of pre-induced twinning on microstructure and tensile ductility in GW92K magnesium alloy during multi-direction forging at decreasing temperature

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Abstract

Effect of pre-induced twinning on the microstructure evolution and mechanical properties of extruded Mg-9.26Gd-2.08Y-0.36Zr (GW92K) alloy have been investigated during multi-direction forging with large strains at decreasing temperature from 400 °C to 300 °C. The results showed that, whether there pre-induced twinning existing in the initial microstructure by pre-deformation or not, a mixed microstructure of residual coarse grains and notably refined grains formed under both conditions, combing with some residual coarse grains with less deformation inside grains and lots of dispersed nano-precipitates distributed along refined grain boundaries. However, a significant improvement with the tensile ductility was obtained by the pre-induced twinning in the former alloy. It was suggested that, the pre-induced twinning largely contributed to the grain refinement and lead to an increase in the ratio of fine grain structure which would be responsible for the better properties. Furthermore, during subsequent forging deformation in the pre-deformed sample, the grain refinement mechanism by gradual grain orientation rotation around the surface section in residual coarse-grains was a little different from that by the macro-shear deformation in the as-extruded condition.

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Keywords: Magnesium alloy; Pre-induced twinning; Multi-directional forging; Refined-grain microstructure; Tensile ductility

1. Introduction

Mg alloys are promising light-weight engineering materials for application in areas such as automobiles and aeronautics due to their combination of low density, good specific strength. Compared with other conventional structural alloys, its relatively poor ductility and deformation anisotropy due to its hcp lattice structure weaken its ability of being subjected to the same wrought processes, such as, aluminum or steel [1].

Grain refinement is the best way to improve the ductility and strength simultaneously. Fabrication of micron or nano-bulk materials through severe plastic deformation (SPD) has attracted broad interests. Actually Multi-direction forging technology (MDF) is an effective way to obtain fine or even ultra-fine grains, leading to an effective enhancement in ductility [2]. Recently, this method used for wrought magnesium alloys processing has been a concern for researchers, and also won a series of encouraging results [3–6].

Owing to MDF process mentioned above, the grain size can be refined to 1–2 μm in AZ80 magnesium alloy as well as outstanding mechanical properties were observed, i.e. the ultimate tensile strength (UTS) and elongation (ϵ) are up to 345 MPa and 8%, which both are twice than those before being forged [3]. Xing et al. [4] reported that the grain is

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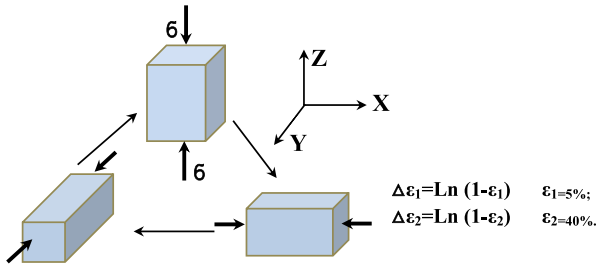


Fig. 1. Schematic of MDF when a pass strain $\Delta\epsilon_1$ and $\Delta\epsilon_2$ is employed, respectively.

further refined to 300 nm in AZ31 alloy by MDF at the decreased temperature, combined with the UTS and ϵ of 530 MPa and 15%, respectively. Furthermore, Miura et al. [5] obtained a 0.8 μm fine grained microstructure in AZ61 alloy, with the UTS of 530 MPa, and the ϵ of about 10%, and even got the superplastic ductility up to 300% at 150 °C. Therefore, this process may provide a feasible way to achieve high performance.

The Mg–Gd–Y system alloys with higher specific strength both at room and elevated temperatures have received intensive research. A GW94 alloy with fully recrystallized microstructure and equiaxed ultrafine grains of submicron size was produced by multi-axial forging (MDF) plus ageing, which is easier than conventional extrusion to improve plasticity [6]. In order to optimize the deformation process in the alloys, it is so necessary to further understand the deformation mechanism and strengthening theory.

The initial microstructures (such as the grain size, texture, second phase morphology, etc.) have a significant effect on thermo-deformation [7–10]. With decreasing the initial grain size in the stainless steel, grain refinement through MDF is accelerated, as a result of the continuous increase in the misorientations between the sub-grains during deformation [11]. Therefore, it is also necessary to investigate the effect of initial microstructures on the evolution of the microstructures and mechanical properties during subsequent MDF process.

2. Experimental procedure

The initial material used in this study was a kind of as-extruded GW92K rod (Mg-9.26Gd-2.08Y-0.36Zr, wt. %)

prepared at 400 °C by an extrusion ratio of 5. Cubic samples with length of 12.5 mm were machined from the extruded rods with one side parallel to the extrusion direction (ED). Then the samples were MDFed at room temperature on a press machine at an initial strain rate of $1 \times 10^{-2} \text{ s}^{-1}$. The forging axis during the first press was parallel to ED of the extrusion rods. Graphite was applied to the faces between the ends of test pieces and grips for lubrication. A part of cubic samples were MDFed by 24 passes with a minor strain of 5% ($\Delta\epsilon_1 = 0.05$) per pass and the axis rotating by 90° in the next pass as shown in Fig. 1 by figured with $z \rightarrow x \rightarrow y \rightarrow z \rightarrow x$ i.e., leading to a cumulative strain of around $\sum\Delta\epsilon = 1.2$, which were designated as “pre-deformed” samples. Then the pre-deformed samples were forged by MDF experiments with a reduction per pass of about 40% ($\Delta\epsilon_2 = 0.34$), under the decreasing temperature from 400 °C to 300 °C by a drop of 25 °C every three successive forging passes. MDF process was repeated by changing the press direction with 90° at a speed of $1 \times 10^2 \text{ s}^{-1}$ after the sample hold under the set temperature for 5 min. Water quenching was used to keep the instantaneous microstructure between passes.

Microstructures were observed on the plane along the last compression direction by optical microscope (OM), Philip XL30 scanning electronic microscope (SEM) with electron backscattering diffraction (EBSD), and transmission electron microscope (TEM) equipped with JEOL 2100F. The specimens for tensile tests were cut along the plane perpendicular to the final forging axis, with a gauge of 10 mm in length, 2.5 mm in thickness and 3.5 mm in width, and tensile tests were carried out under room-temperature at a speed of $1 \times 10^{-3} \text{ s}^{-1}$.

3. Results and discussion

3.1. Initial microstructure

Optical microstructure of the as-extruded GW92K alloy showing an average grain size about 50 μm , and the microstructure of the pre-deformed samples after being MDFed at room temperature were shown in Fig. 2. Second phase particles with a size of several microns were randomly distributed in the matrix of both states, as shown in the embedded pictures in Fig. 2. After being performed by MDF for 24 passes with a

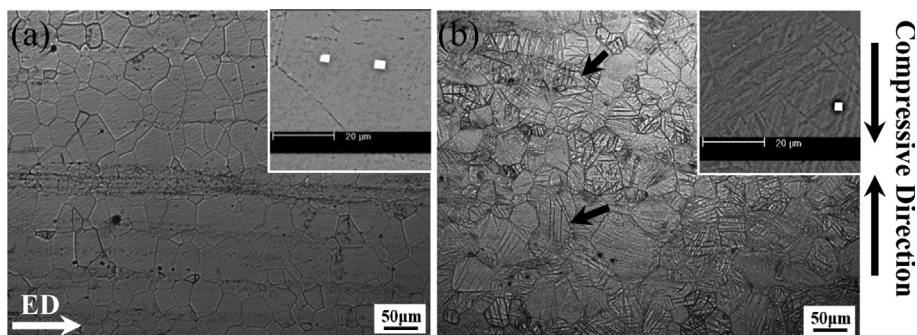


Fig. 2. Microstructures of the GW92K alloy: (a) as-extruded, (b) the pre-deformed at room temperature.

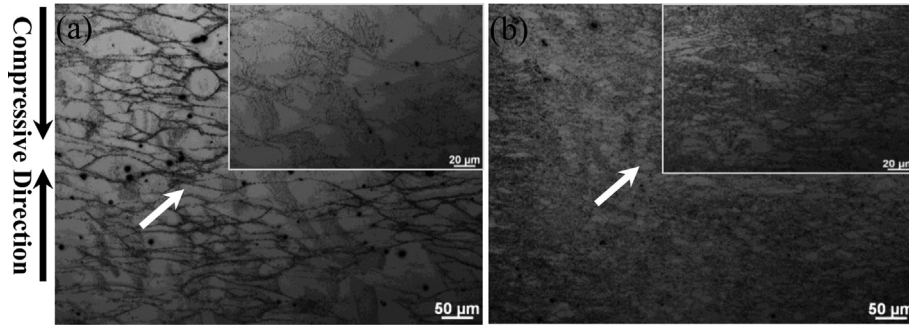


Fig. 3. Microstructures of the GW92K alloys MD Fed 1 pass at 400 °C in different initial states of specimens: (a) as-extruded, (b) the pre-deformed at room temperature.

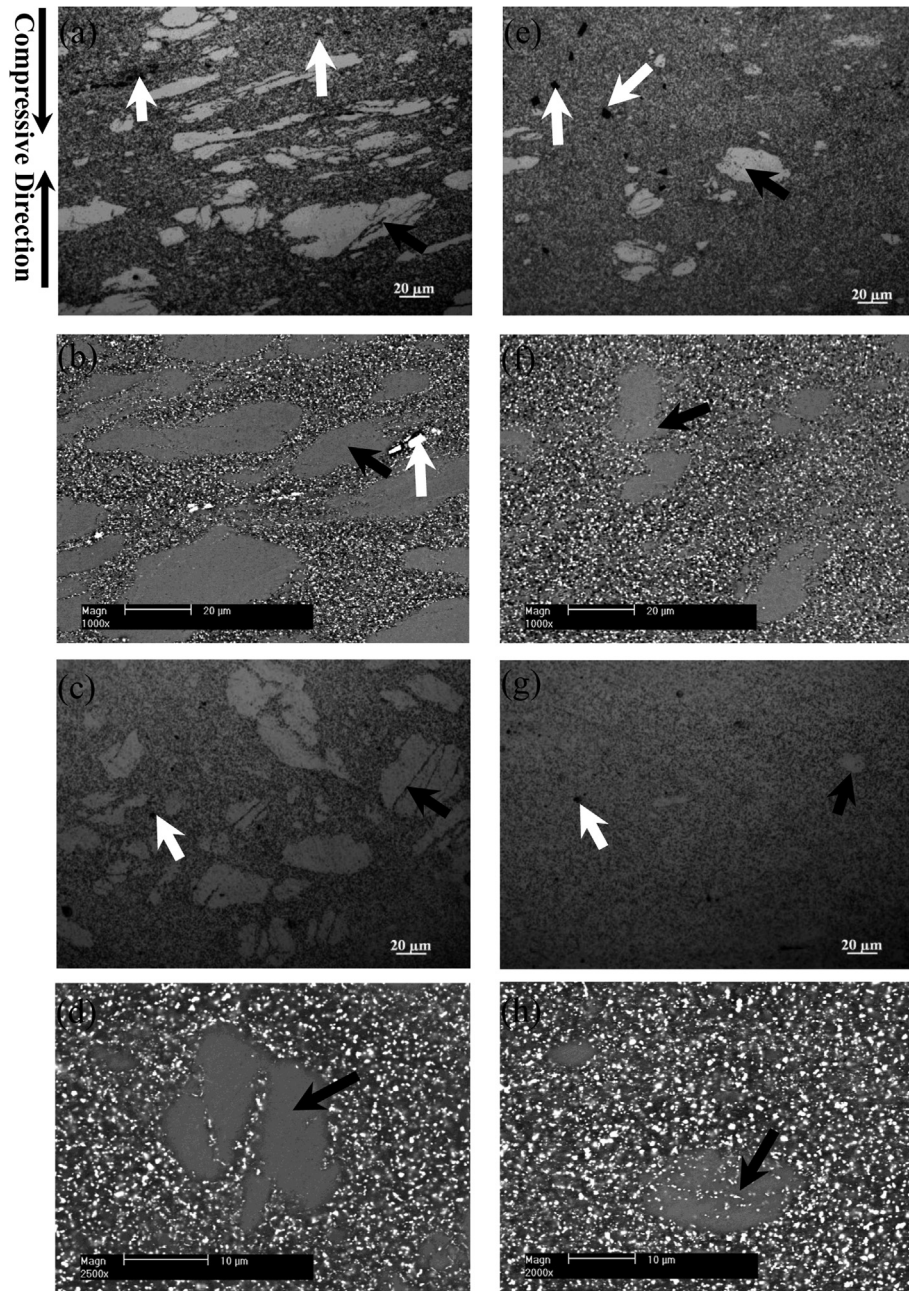


Fig. 4. Microstructures of the different-stated GW92K alloys MD Fed under decreasing temperature with different passes. (a–d) as-extruded, (e–h) the pre-deformed; and (a,b,e,f) 11-pass, (c,d,g,h) 15-pass.

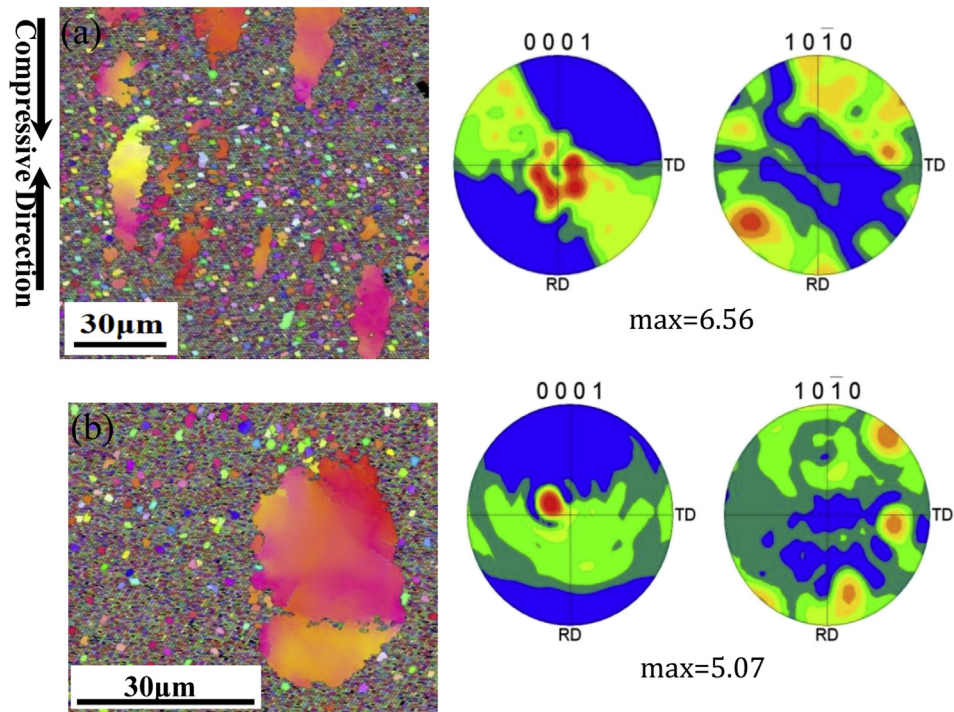


Fig. 5. EBSD analysis for the microstructures of two GW92K alloys subjected to 15-pass MDF (40% per pass): (a) as-extruded, and (b) the pre-deformed, respectively.

total strain of 1.2, high-density twinning intersected with each other (as shown by arrows in Fig. 2b), which consisted of $\{10\text{--}11\}$ contraction twinning and $\{10\text{--}12\}$ extension twinning [12], were detected virtually in most of original grains, as the initial microstructure to subsequent MDF experiments.

3.2. Microstructures after MDF

In the as-extruded samples, deformation features were more obvious at grain boundaries after MDF followed 1-pass at 400 °C as shown in Fig. 3, and dynamic recrystallization in local areas of initial grains even had occurred (as shown by white arrows). However, the ratio of obvious recrystallization significantly increased at the twinning in the pre-deformed specimen.

Fig. 4 indicated the microstructures of the GW92K alloys after being MDFed by accumulated 11-pass at the decreasing temperature from 400 °C to 325 °C, with $\sum\Delta\varepsilon = 4.4$ in the as-extruded samples and $\sum\Delta\varepsilon = 5.6$ in the pre-deformed ones. It

was found that both alloys had been refined remarkably. A large amount of residual coarse grains were remained in the as-extruded (as shown in Fig. 4a) and characterized by smaller ones dividing via shear deformation, however few existed in the pre-deformed samples (as shown in Fig. 4e). SEM results showed that, minor coarse second-phase particles (noted by white arrows), appeared in the initial as-extruded alloy, still remained distributing in the matrix, while merely fewer randomly distributed in the pre-deformed ones. When MDF for 15-pass ($\sum\Delta\varepsilon = 7.7$) at 300 °C, the shear zones remained in the as-extruded (as shown in Fig. 4c), few such areas could be detected in the pre-deformed (as shown in Fig. 4g), but refined grains. Besides, in both different initial states, a large number of dispersed phases dynamically precipitated during deformation.

The EBSD analysis in Fig. 5 showed that, the microstructures of both initial states were composed of refined grains and residual coarse grains. The microstructure in as-extruded alloy was refined dominantly by shear deformation, as say, banded

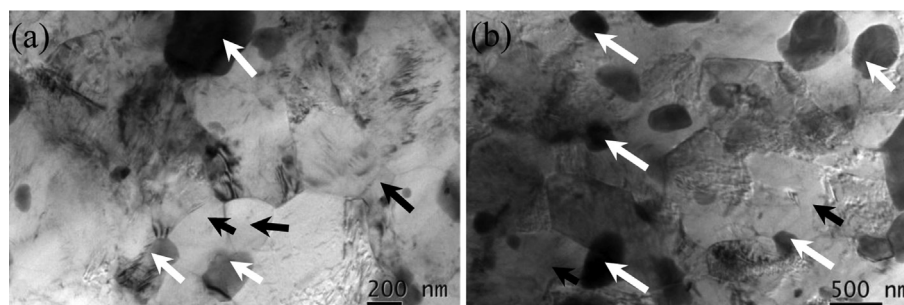


Fig. 6. TEM images of fine-grained regions in both states of alloys being subjected to MDF with 15 passes: (a) as-extruded, and (b) the pre-deformed, respectively.

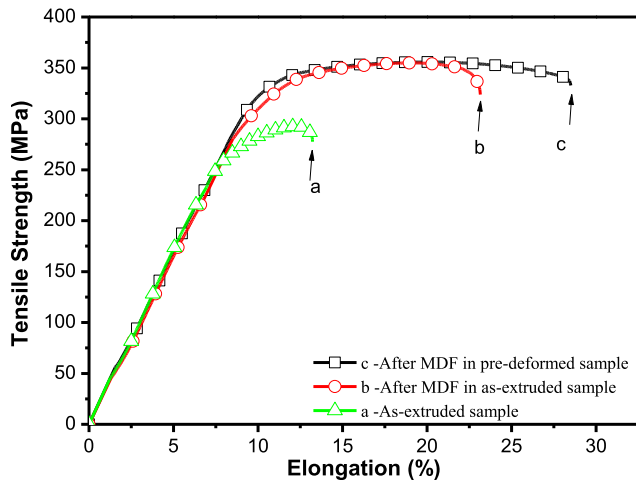


Fig. 7. Tensile properties of GW92K alloys in different states: (a) as-extruded, (b) MDFed in as-extruded and (c) MDFed in pre-deformed samples, respectively.

coarse-grains were mainly distributed their basal planes with shear stress at about 45° (Fig. 5a). During MDF, shear deformation occurred in partial coarse crystals, resulting in these grains gradually divided or crushed, and then distorted their crystals by geometrical rotation in shear regions, which led to grain size gradually decreased. The basal poles in most of the crystals were concentrated 45° with the compressive direction, which may be result from the rotation of the original basal plane by 45° from the compression direction through shear deformation.

In the pre-deformed alloy, a smaller amount of residual coarse grains were also found, and their basal planes mostly oriented approximately parallel to the compressive direction (as shown in Fig. 5b). In such condition, lower part of residual coarse grains had been subdivided by high-angle boundaries.

Also minor grains occurred prior nucleation in deformation belts, which may be caused by the mechanism of rotation dynamic re-crystallization (RDRX) [13]. (0001) pole figure also suggested that, the pre-deformed sample had a weaker texture with an increase in refined grains, as compared to the as-extruded samples.

After 15 passes a large strain of 40% per pass at decreasing forging temperature from 400°C to 300°C , TEM observations in both states of alloys indicated that, some grains have been refined to hundreds of nanometer (black arrows) and minor second phases with a size of hundreds to tens of nanometer dispersed at grain boundaries (white arrows), as shown in Fig. 6.

Moreover, in the pre-deformed samples, there was a slight increase both in “grain size” and “precipitate size”, also the microstructure appeared few dislocations (Fig. 6b), which meant more fully recrystallization has happened in the pre-deformed alloy.

3.3. Mechanical properties after MDF

Tensile properties of the as-extruded alloy, and the MDFed alloys with two different initial deformation situations were shown in Fig. 7. Compared with the as-extruded specimen, large improvements of ultimate tensile strength (σ_b), tensile yield strength ($\sigma_{0.2}$) were observed in both as-MDFed samples, especially elongation (ϵ) rises so sharply. The pre-deformed alloy specimen showed a better combination of strength and ϵ , its σ_b , $\sigma_{0.2}$ and ϵ were 350 MPa, 320 MPa, and 18.6%, respectively.

The fractography of the MDFed alloy specimens of as-extruded and pre-deformed was shown in Fig. 8, respectively. Compared Fig. 8a and b, more ductile dimples and fewer cleavage fracture surface exhibited in the pre-deformed

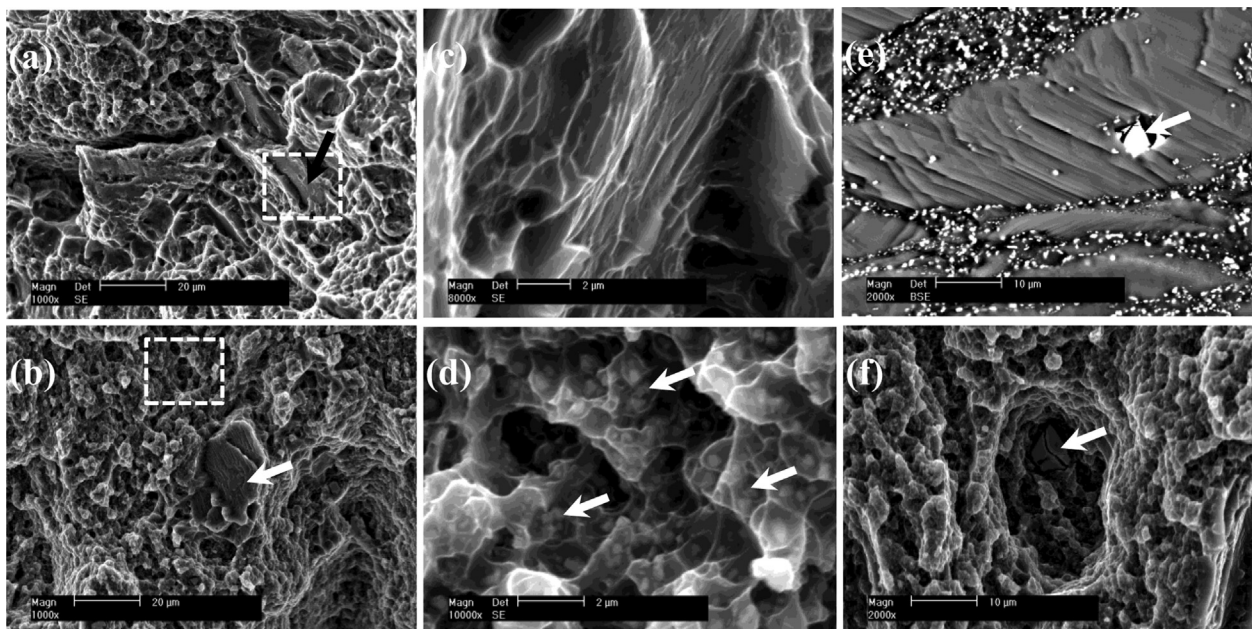


Fig. 8. Tensile fractures of the MDFed GW92K alloys in different states: (a, c, e) as-extruded, and (b, d, f) pre-deformed samples, respectively.

samples, together with a large number of fine precipitates (in Fig. 8d); while cleavage fracture surface and steps are even prominent in as-extruded ones, as enlarged regions shown in Fig. 8c. In Fig. 8e and f, the coarsening second phases of both states were found broken, which may be due to the second phases more brittle than matrix itself.

In this study, plenty of twinning were induced through pre-deformation at room temperature, as similar to increasing the grain boundary density, which could lead to accelerate the process of grain refinement in subsequent MDF [11,12], and be beneficial for forging by the induced softening [14]. Furthermore, MDF under decreasing temperature at below $0.5 T_m$ could prevent the refined grains excessive growing [5,15], which can ascribe to reducing the dynamic recrystallization temperature by the accumulation of great plastic strains [16]. Accumulated strain by multi-passes contributed to induce more dislocations and other crystal defects around grain boundaries, which could also result in precipitation occurred within deformed regions by storing larger energy [17] and grain refinement [18] by a large of grain nucleation induced through particle stimulated nucleation (PSN) mechanism [10]. It is also suggested that, more precipitates appeared in the microstructure may be responsible for the more stable fine-grained structure and the weaker texture. In addition, precipitates were effective in impeding dislocation slip, which could further strengthen tensile properties. As for the ductility, the pre-induced twinning could further weaken texture with alternate the forging direction [14], and even the crystal orientation was slightly changed in remaining grains interior, as EBSD analysis in Fig. 5, therefore a larger elongation achieved in the pre-deformed alloy.

4. Conclusions

- 1) The pre-induced twinning by pre-deformation is beneficial for obtaining a finer microstructure and weaker texture during subsequent multi-directional forging (MDF).
- 2) A better ductility can be achieved in as-extruded GW92K alloy especially after being pre-deformed followed by MDF than those through directly forging, in spite of strength slightly improved.
- 3) Pre-deformed samples exhibit ductile fracture features of dimples, which could be chiefly attributed to the refined grain size, and the weaker texture, together with fine dispersed precipitates.

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References

- [1] F.S. Pan, E.H. Han, *Wrought Magnesium Alloy and High-Performance Processing Technology*, Science Press, Beijing, 2007.
- [2] R.M. Imayev, V.M. Imayev, G.A. Salishchiv, *J. Mater. Sci.* 27 (1992) 4465.
- [3] Q. Guo, H.G. Yan, Z.H. Chen, H. Zhang, *Acta Metall. Sin.* 42 (2006) 739–744.
- [4] J. Xing, H. Soda, X.Y. Yang, H. Miura, T. Sakai, *Mater. Trans.* 46 (2005) 1646–1650.
- [5] H. Miura, G. Yu, X. Yang, *Mater. Sci. Eng. A* 528 (2011) 6981–6992.
- [6] L. Gao, R.S. Chen, E.H. Han, *Trans. Nonferrous Met. Soc. China* 21 (2011) 863–868.
- [7] M.R. Barnett, A.G. Beer, D. Atwell, A. Oudin, *Scr. Mater.* 51 (2004) 19–24.
- [8] W.N. Tang, R.S. Chen, J. Zhou, E.H. Han, *Mater. Sci. Eng. A* 499 (2009) 404–410.
- [9] S.W. Xu, N. Matsumoto, S. Kamado, T. Honma, Y. Kojima, *Mater. Sci. Eng. A* 523 (2009) 47–52.
- [10] T. Li, K. Zhang, X.G. Li, Z.W. Du, Y.J. Li, M.L. Ma, G.L. Shi, *J. Magnesium Alloys* 1 (2013) 47–53.
- [11] A. Belyakov, K. Tsuzaki, H. Miura, T. Sakai, *Acta Mater.* 51 (2003) 847–861.
- [12] H. Miura, T. Maruoka, X. Yang, J.J. Jonas, *Scr. Mater.* 66 (2012) 49–51.
- [13] Y. Qiao, X. Wang, Z. Liu, et al., *Mater. Sci. Eng. A* 568 (2013) 202–205.
- [14] M.R. Barnett, *Mater. Sci. Eng. A* 464 (2007) 8–16.
- [15] W.W. Jian, Z.X. Kang, Y.Y. Li, *Chin. J. Nonferrous Met.* 18 (2008) 1005–1011.
- [16] X. Zhao, J.W. Gao, Y. Nan, T.F. Jing, *Mater. Rev.* 17 (2003) 5.
- [17] D. Lin, L. Wang, Y. Liu, J.Z. Cui, Q.C. Le, *Trans. Nonferrous Met. Soc. China* 21 (2011) 2160–2167.
- [18] O. Sitdikov, T. Sakai, A. Goloborodko, R. Kaibyshev, *Mater. Trans.* 45 (2004) 2232–2238.