Influences of Asymmetric Reduction Rolling on the Microstructure and Mechanical Properties of AZ91

Yi-Quan Zhao¹ · Hong-Mei Chen² · Jing Zhang³ · Ru Ma¹ · Yan-Dong Liu⁴ · Yi-Nong Wang¹ · Ling Wang⁵ · Qun Zhang⁶ · Wei-Gang Li⁶

Received: 17 July 2017 / Revised: 18 September 2017 / Published online: 13 January 2018
© The Chinese Society for Metals and Springer-Verlag GmbH Germany, part of Springer Nature 2018

Abstract

The influence of asymmetric reduction rolling (ARR) on the microstructure, texture and mechanical properties of AZ91 was investigated. The microstructural characteristics of the AZ91 sheet processed by symmetric rolling (SR) were the twins, intersection of twins and dynamic recrystallization (DRX) grains around the coarse grains and within the twins. However, the amount of twins and DRX grains in ARRed AZ91 was much smaller than that in SRed AZ91. The SRed AZ91 after annealing exhibited fine DRX grains and some coarse grains with a size of $\sim 100 \mu\text{m}$. The grains in ARRed AZ91 after annealing were much finer and more homogeneous than those in SRed AZ91 after annealing. The intensity of basal texture of ARRed AZ91 after annealing was lower than that of SRed AZ91 rolling after annealing. The average Schmid factor of ARRed AZ91 is 0.34, which is higher than that of SRed AZ91. The yield strength and ultimate tensile strength of the ARRed AZ91 sheet were increased to 16.1% and 31.8% compared to SRed AZ91 sheet, from 155 to 180 MPa, and from 220 to 290 MPa, respectively. The improvement of mechanical properties in ARRed AZ91 after annealing was attributed to much finer, more homogeneous DRX grains and weaker basal texture.

Keywords AZ91 · Asymmetric reduction rolling · Microstructure · Texture · Mechanical properties

1 Introduction

Magnesium alloys are attractive structural materials in many fields, such as electronic industries, aerospace, automotive, and other transportation systems due to their low density, high specific strength, good machinability and formability, good damping ability and easy recycling. However, the low strength and high production cost restrict their application wide compared to steels and aluminum alloys. The improved conventional strength by processing technologies, such as rolling, extrusion, forging could not be used widely in the Mg alloys due to their hexagonal close-packed crystal structure and formation of high intensity of basal texture [1–5]. Therefore, the ductility and workability improvement is one of the prevalent issues in Mg alloys research [6–9].

Several thermo-mechanical processes have been reported for obtaining fine grain size and decreasing the intensity of basal texture of Mg alloys. Equal channel angular pressing (ECAP) is one of the most effective methods to tilt basal texture and refine the grains [10–13]. Asymmetric rolling process (ASR), differential speed rolling (DSR),

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

✉ Ling Wang
30732029@qq.com; hill-wang2005@163.com

¹ School of Materials Science and Engineering, Dalian University of Technology, Dalian 116024, China

² Provincial Key Lab of Advanced Welding Technology, Jiangsu University of Science and Technology, Zhenjiang 212003, China

³ School of Metallurgy and Materials Engineering, Jiangsu University of Science and Technology, Zhangjiagang 215600, China

⁴ School of Materials Science and Engineering, Northeastern University, Shenyang 110819, China

⁵ Yingkou Institute of Technology, Yingkou 115014, China

⁶ Bayuquan Entry-Exit Inspection and Quarantine Bureau, Yingkou 115000, China

asymmetric extrusion (AE) and accumulative roll bonding (ARB) could decrease the intensity of basal texture and tilt the basal texture to some extent due to the introduction of shear stress [14–19]. In addition, changing processing routes was a candidate for grain refinement. Yu et al. [20] reported that short flow rolling (a new rolling route combining one pass of hard-plate rolling and two passes of conventional rolling) could obtain uniform fine-grained structure ($\sim 3 \mu\text{m}$) featuring a weakened texture in AZ91–0.4wt%Sn plates after a total thickness reduction of $\sim 86.4\%$. Ma et al. [21] reported that two-stage rolling, high temperature rolling plus mediate temperature rolling, could get high strength and good ductility for AZ31 alloy. However, the techniques mentioned above have some disadvantages, such as high manufacturing cost, low productivity and small product dimension. AZ91 alloys have high strength at room temperature, good cast ability and excellent corrosion resistance. Therefore, AZ91 alloys are used widely as the cast Mg parts. To enhance the strength and ductility of Mg alloys, the plastic working was carried out on low Al-containing Mg alloys and AM series Mg alloys. However, the researches on workability, microstructure and mechanical properties improvement of high-alloyed magnesium alloys-AZ91 were limited [22–28]. Asymmetric reduction rolling (ARR) proposed by our research group was a very effective process for enhancing mechanical properties and ductility of AZ31 simultaneously, with low manufacturing cost, high productivity and big product dimension [29]. It is expected that ARR could improve mechanical properties and ductility, and therefore expand the application range of AZ91. However, there have been few reports on the influence of ARR on microstructure, texture and mechanical properties of AZ91, so these were investigated in this study.

2 Finite Element Stimulation of ARR and SR Process

The schematic of the ARR process is shown in Fig. 1. The ARR machine is composed of SR machine and a support plate in front of the bite entrance. Therefore, the ARR and SR processes have the same roll diameters and rotation speed, and the support plate becomes the unique difference. The support plate is expected to introduce shear stress, which makes the upper and lower parts of the sheet deform asymmetrically. In order to confirm the introduction of shear stress, the ARR and SR processes were stimulated by commercial finite element (FE) software (DEFORM-2D). In the FE stimulation, rotation speed, diameter of rolls and roll barrel are 42 r/min (44 mm/s), 200 mm and 350 mm, respectively. The other necessary parameters for FE

stimulation are listed in Table 1. The details of FE stimulation of ARR were elaborated in Ref. [22].

The effective stress and strain distribution of AZ91 sheets rolled in a 30% reduction were investigated by FE stimulation, and the results are shown in Fig. 2. In SR process, the distribution of the effective stress is symmetric with respect to the middle layer of the sheet as shown in Fig. 2b. While for ARR process, distribution of the effective stress is asymmetric. The effective strain distributions of ARR and SR processes have the similar characteristic with effective stress distributions of ARR and SR processes, as shown in Fig. 2c, d. Compared to the symmetric distribution of the effective strain during SR, the effective strain of the lower layers is higher than that of the upper layers of the ARRed sheets. Based on the FE stimulation, it is confirmed that the shear stress was introduced during the process of ARR.

3 Experimental Procedures

The AZ91 alloy in the present study was composed of 9 wt%Al, 0.8 wt%Zn, 0.3 wt%Mn with Mg at balance. It was melted in an electrical furnace using a mild steel crucible under sulfur powder protection. The melt AZ91 was held for 10 min at 740 °C and then poured into a mild steel mold (coated with BN and preheated to 200 °C) with a size of 185 mm $\times$ 100 mm $\times$ 25 mm. AZ91 blocks with a size of 170 mm $\times$ 85 mm $\times$ 20 mm were cut from the gray block of AZ91 ingots homogenized at 380 °C for 20 h, as shown in Fig. 3. AZ91 rolling sheet was from the gray block in Fig. 3 with the size of 100 mm (rolling direction) $\times$ 50 mm (width) $\times$ 5 mm (thickness). The AZ91 rolling sheet was heated to 400 °C for 10 min and then one pass rolled with a reduction of 30%, from 5 to 3.5 mm. The rolling speed and diameter of roll were 44 mm/s and 200 mm, respectively. Then, the rolled AZ91 sheets were annealed at 400 °C for 30 min.

All optical microstructures (OM) were obtained with the polishing and etching using a solution of picric acid, acetic acid, distilled water and ethanol at room temperature in the transverse direction (TD) of rolled AZ91 sheets. The grain sizes were determined by analyzing the OM using the line-intercept method. Electron backscattered diffraction (EBSD) measurements were taken at the mid-plane in thickness of the rolled sheets, and the samples preparation consisted of mechanical grinding, mechanical polishing, electro polishing and cleaning [19]. The EBSD measurements were taken on a Zeiss Supra 55 FEG-SEM at 20 kV, with a working distance of 20 mm, a tilt angle of 70° and a scanning step of 0.4 μm . The specimens for tensile tests had a gauge length of 15 mm, width of 5 mm and thickness of 3.5 mm. Tensile tests were carried out parallel to the

Fig. 1 Schematic of the ARR process **a** initial stage; **b** stable rolling stage

Table 1 Parameters of ARR FE simulation

Parameters	Unit	Value
Friction coefficient		
Roll-sheet, μ_1		0.3
Support plate-sheet, μ_2		0.1
Thermal properties		
Thermal conductivity	$\text{N}(\text{s } ^\circ\text{C})^{-1}$	159
Heat capacity	$\text{N}(\text{MM}^2^\circ\text{C})^{-1}$	1.7675
Interface heat transfer coefficient	$\text{N}(\text{s MM}^2^\circ\text{C})^{-1}$	4.5
Thermal expansion		
Thermal expansion		2.2×10^{-5}
Convection coefficient	$\text{N}(\text{s MM } ^\circ\text{C})^{-1}$	0.2

rolling direction at room temperature with an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ on a CMT6305-300KN electronic universal testing machine.

4 Results and Discussion

4.1 Microstructure of the as-Cast AZ91 Alloy

The microstructure of the as-cast AZ91 alloy after homogenization at 380°C for 20 h is shown in Fig. 4. The microstructure of homogenized AZ91 alloy was composed of coarse cell structure (with average grain size of $\sim 150 \mu\text{m}$) and eutectic β ($\text{Mg}_{17}\text{Al}_{12}$) phases precipitated along the grain boundaries, which are indicated by the arrows A and B, respectively. The high amount of Al element (in this study 9 wt%) could induce the Al

concentration at the liquid–solid phase boundary during AZ91 alloy solidification process [30]. The concentration of Al element could meet the condition of formation of β phases. Due to the excessive amount of β phases, the homogenization at 380°C for 20 h could not dissolve β phases, higher temperature and longer holding is necessary to dissolve the β phases totally.

4.2 Microstructure of Hot Rolled AZ91

The microstructural characteristics of the AZ91 alloy sheet processed by SR were the formation of twins, intersection of twins and dynamic recrystallization (DRX) grains around the original grains and within the twins, indicated by the arrows A, B, C and D in Fig. 5a. It can be observed that fine DRX grains were mostly free of twins. The twins indicated by arrows A and B were considered as $\{10\bar{1}2\}$ extension twins due to lack of DRXed grains within twins and narrow thickness of twins [31]. The twin indicated by arrow C was intersection of twins. Dislocations were easy to slip in the internal of contraction twin $\{10\bar{1}1\}$ due to the 56° reorientation of crystal lattice [32]. Therefore, plenty of DRX grains was easily observed in $\{10\bar{1}1\}$ contraction twins as shown by arrow D.

Fine DRXed grains and coarse grains with the size of $80\text{--}100 \mu\text{m}$ were the main characteristics of SRed AZ91 sheets after annealing, as shown in Fig. 5b. The coarse grains were considered as the original grains, which were divided by twins or partly be swallowed by DRXed grain growth, in the as-homogenized AZ91. Because the grain size of coarse grains was slightly smaller than that of the as-homogenized AZ91, the grains with basal texture were

Fig. 2 Effective stress and strain distributions of AZ91 sheets with a 30% reduction obtained from FE simulation: **a** effective stress distribution of ARR; **b** effective stress distribution of SR; **c** effective strain distribution of ARR; **d** effective strain distribution of SR

Fig. 3 Schematic illustration of the sample for rolling taken from a cast ingot

hard to be absorbed by DRXed grains, or the process of absorption by DRXed grains was very slow, so the coarse grains after annealing are basal grains [33, 34]. The average grain size (AGS) of SRred AZ91 in annealed condition was about $\sim 30 \mu\text{m}$.

The microstructural characteristics of the AZ91 alloy sheet processed by ARR were twinning, intersection twinning and the DRX within the twins, indicated by

Fig. 4 Optical micrographs of the as-homogenized AZ91

arrows A and B as shown in Fig. 6a. Except for the aforementioned, a number of black areas indicated by arrows C and D are shown nearby coarse grains in Fig. 6b. For further study, these thick black areas were investigated by SEM and EDS. Those areas were characterized as fine grains (not fully DRXed grains with AGS of $8\text{--}10 \mu\text{m}$),

Fig. 5 Optical microstructure of **a** as SRed AZ91; **b** SRed AZ91 in annealed condition

Fig. 6 Optical microstructure of **a, b** as ARRed AZ91 sheet; **c** ARRed AZ91 in annealed condition

Fig. 7 Details of black area in ARRed AZ91 rolling sheets: **a** SEM image; **b** EDS result for phase A; **c** EDS result for phase B

Fig. 8 Inverse pole figure (IPF) maps of the rolled AZ91 Mg alloy obtained by EBSD: **a** SRred AZ91; **b** ARred AZ91

Fig. 9 Schmid factor of AZ91 sheets processed by **a** ARred; **b** SR with a 30% reduction

Fig. 10 Stress–strain relations of AZ91 processed by SR and ARR

$\text{Mg}_{17}\text{Al}_{12}$ phase and Al_8Mn_5 phase as demonstrated in Fig. 7. The thick black areas in ARRed AZ91 disappeared after annealing, as shown in Fig. 6c. The disappearance of coarse grains in ARRed AZ91 alloys after annealing was attributed to the consumption of DRXed grains growth. Obviously, the amount of twins, intersected twins and DRX in ARRed AZ91 was much smaller than those in SRed AZ91. The black areas were rarely observed in SRed AZ91. After annealing, the coarse grains and black areas were substituted by homogeneous and fine DRXed grains shown in Fig. 6c.

The compression stress could employ on the rolling sheet during the process of SR and ARR, however, the shear stress would be generated when support plate was introduced to SR machine according to the result of finite element simulation [22]. The shear stress introduced by ARR could make dislocation slip in those grains where twins were accomplished easier during SR process. In another word, the introduction of shear stress make more grains deform by dislocation slip mechanism compared to that of no shear stress introduction. It is proved in Fig. 6a that the amount of twins decreases dramatically in ARRed AZ91.

To examine the microstructure in detail, the inverse pole figure (IPF) was measured by EBSD at the center of the sheets in thickness for SRed and ARRed AZ91 in annealing condition, as shown in Fig. 8. The SRed and ARRed

AZ91 sheets exhibited basal texture. The intensity of basal texture of ARRed AZ91 sheet (Max = 4.74) was lower than that of SRed AZ91 sheet (Max = 5.01). It could be predicted that the intensity of basal texture would be higher than that in Fig. 8a, when coarse grains with basal texture were observed by EBSD in Fig. 8a.

Figure 9 shows Schmid factor (SF) distribution maps for basal $\langle a \rangle$ slip system of AZ91 sheets processed by ARR and SR process, obtained by EBSD analysis. It can be observed that for SR sheet with a stronger basal texture, the average of Schmid factor (ASF) in the basal plane is 0.29, while for ARR sheet with a weakened basal texture that is 0.34, which is slightly higher than that of SR sheet. Thus, it is more easy to activate the basal slip during deformation, which results in an improvement in the plasticity of AZ91 alloys.

4.3 Mechanical Properties

The nominal stress–strain curves obtained at room temperature are shown in Fig. 10. The ARRed AZ91 in annealed condition exhibits higher mechanical properties than SRed AZ91 in annealed condition. The yield strength (YS) and ultimate tensile strength (UTS) of the ARRed AZ91 in annealed condition were increased to 16.1 and 31.8%, from 155 to 180 MPa, and from 220 to 290 MPa, respectively. Simultaneously, the elongation to failure increased by 50% in the ARRed AZ91 (15%) compared to that of the SRed AZ91 (10%).

The comparisons of room properties of AZ91 using different processing method are listed in Table 2. It is clearly shown that the ARR process had obvious enhancement of YTS, UTS and elongation of AZ91 simultaneously. Grains size and texture are the two key factors influencing the mechanical properties. According to Hall–Petch relation, $\sigma_{0.2} = \sigma_0 + kd^{-\frac{1}{2}}$, where $\sigma_{0.2}$ is the yield stress of a polycrystalline metal, σ_0 is the yield stress relating to materials of infinite grain size, which is similar to the single crystal, k is a constant representing the grain boundaries as an obstacle to slip across the grain boundaries, and d is the grain size [35]. From the grain size point of view, it is easy to understand that the YS of ARRed AZ91 is higher than that of SRed AZ91 due to finer and

Table 2 Comparisons of the room temperature properties of AZ91 alloy using different processing

Processing method	YTS (MPa)	UTS (MPa)	Elongation (%)	Reference
T4 treated AZ91	150	210	10	[27]
SRed AZ91	155	220	10	Present work
ARRed AZ91	180	290	15	Present work
As rolled AZ91	175	260	6	[28]
MDF AZ91-350-1P	135	220	3	[29]
MDF AZ91-400-1P	150	250	3.5	[29]

more homogeneous grains. The dislocations in the grains with soft orientation could be activated more easily compared to the dislocations in hard orientation [36]. The Mg alloys, which have more grains with soft orientation, would show lower mechanical properties than the alloys with more grains in hard orientation. In other words, the resistance of the grain boundaries as an obstacle to dislocation slip across the grain boundaries in Mg alloys with high intensity of basal texture is higher than that in alloys with lower intensity of basal texture in the condition of same grain size. In a word, fine grains and low intensity of basal texture are two competitive mechanisms affecting the mechanical properties. According to the results of mechanical properties, the grain size is more effective to affect the mechanical properties than the intensity of basal texture in this study.

Furthermore, the increase in elongation of ARRed AZ91 sheets could attribute to weak intensity of basal texture, homogeneous and fine grains. Dislocation in Mg alloy with weak intensity of basal texture could slip easily. Fine grains means that the proportion of grain boundary is big and the deformation energy absorption is sufficient. Moreover, homogeneous grains could make deformation uniformly and avoid the appearance of excessive concentration of local stress.

5 Conclusions

The ARR process had higher efficiency to refine grains than the SR process. The ARRed AZ91 in annealed condition exhibited finer and more homogeneous grains compared the SRed AZ91 in annealed condition.

The intensity of basal texture of SRed AZ91 was higher than that of ARRed AZ91. The ASF of ARRed AZ91 was higher than that of SRed AZ91.

ARR process is an effective way to obtain good combination of high strength and ductility of AZ91 without loss of productivity and cost increase. The YS and UTS of the ARRed AZ91 sheet were increased to 16.1% and 31.8%, from 155 MPa to 180 MPa, and from 220 to 290 MPa, respectively. The elongation to failure increased by 50% in the ARRed AZ91 (15%) compared to that of the SRed AZ91 (10%).

Acknowledgement This work was financially supported by the National Natural Science Foundation of China (No. 51271046).

References

- [1] I.J. Polmear, *Mater. Sci. Technol.* **10**, 1 (1994)
- [2] H.M. Chen, Q.H. Zang, H. Yu, J. Zhang, Y.X. Jin, *Mater. Charact.* **106**, 437 (2015)
- [3] H.M. Chen, Q.H. Zang, H. Yu, J. Zhang, J.H. Cho, Y.X. Jin, Y.K. Shi, *Magnes. Tech.* **2015**, 267 (2015)
- [4] W.L. Cheng, Q.W. Tian, R. Huo, L. Tian, S.F. Rong, *China Foundry* **13**, 151 (2016)
- [5] W.L. Cheng, Q.W. Tian, H. Yu, B.S. You, H.X. Wang, *Mater. Des.* **85**, 762 (2015)
- [6] M.M. Myshlyaev, H.J. McQueen, A. Mwembela, E. Konopleva, *Mater. Sci. Eng. A* **337**, 121 (2002)
- [7] G.T. Bae, J.H. Bae, D.H. Kang, H. Lee, N.J. Kim, *Met. Mater. Int.* **15**, 1 (2009)
- [8] S.S. Park, G.T. Bae, D.H. Kang, B.S. You, N.J. Kim, *Scr. Mater.* **61**, 223 (2009)
- [9] K. Yamada, Y. Okubo, M. Shiono, H. Watanabe, S. Kamado, Y. Kojima, *Mater. Trans.* **47**, 1066 (2006)
- [10] A.B. Ma, J.H. Jiang, N. Saito, I. Shigematsu, Y.C. Yuan, D.H. Yang, Y. Nishida, *Mater. Sci. Eng. A* **513–514**, 122 (2009)
- [11] V.M. Segal, *Mater. Sci. Eng. A* **271**, 322 (1999)
- [12] R.B. Figueiredo, T.G. Langdon, *Mater. Sci. Eng. A* **501**, 105 (2009)
- [13] W.L. Cheng, Q.W. Tian, H. Yu, H. Zhang, B.S. You, *J. Mater. Alloys* **2**, 299 (2014)
- [14] Y. Chino, M. Mabuchi, R. Kishihara, H. Hosokawa, Y. Yamada, C. Wen, K. Shinojima, H. Iwasaki, *Mater. Trans.* **43**, 2554 (2002)
- [15] H. Watanabe, T. Mukai, K. Ishikawa, *J. Mater. Sci.* **39**, 1477 (2004)
- [16] X. Huang, K. Suzuki, A. Watazu, I. Shigematsu, N. Saito, *J. Alloys Compd.* **470**, 263 (2009)
- [17] C.M. Sellars, W.J.M. Tegart, *J. Mater. Sci. Eng.* **63**, 731 (1966)
- [18] L.L. Chang, Y.N. Wang, X. Zhao, J.C. Huang, *Mater. Sci. Eng. A* **496**, 512 (2008)
- [19] M.T. Pérez-Prado, D. Valle, O.A. Ruano, *Scr. Mater.* **51**, 1093 (2004)
- [20] Z.P. Yu, M. Zha, Z.H. Li, C. Wang, H.Y. Wang, Q.C. Jiang, *Mater. Sci. Eng. A* **695**, 1 (2017)
- [21] R. Ma, Y.Q. Zhao, Y.N. Wang, *Mater. Sci. Eng. A* **691**, 81 (2017)
- [22] S.W. Xu, N. Matsumoto, S. Kamado, T. Honma, Y. Kojima, *Mater. Sci. Eng. A* **523**, 47 (2009)
- [23] W.J. Kim, J.D. Park, W.Y. Kim, *J. Alloys Compd.* **460**, 289 (2008)
- [24] Y.H. Wei, Q.D. Wang, Y.P. Zhu, H.T. Zhou, W.J. Ding, Y. Chino, M. Mabuchi, *Mater. Sci. Eng. A* **360**, 107 (2003)
- [25] S.W. Xu, N. Matsumoto, S. Kamado, T. Honma, Y. Kojima, *Mater. Sci. Eng. A* **527**, 52 (2009)
- [26] K.B. Nie, K.K. Deng, X.J. Wang, T. Wang, K. Wu, *Mater. Charact.* **124**, 14 (2017)
- [27] L.W. Zheng, H.H. Nie, W. Liang, H.X. Wang, Y.D. Wang, *J. Magnes. Alloys* **4**, 115 (2016)
- [28] K.B. Nie, K.K. Deng, X.J. Wang, F.J. Xu, K. Wu, M.Y. Zheng, *Mater. Sci. Eng. A* **624**, 157 (2015)
- [29] R. Ma, L. Wang, Y.N. Wang, D.Z. Zhou, *Mater. Sci. Eng. A* **638**, 190 (2015)
- [30] M.C. Flemings, *Flemings Solidification Processing* (McGraw-Hill Inc, New York, 1974), pp. 58–92
- [31] D. Drozdenko, J. Bohlen, S. Yi, P. Minarik, F. Chmelík, P. Dobron, *Acta Mater.* **110**, 103 (2016)
- [32] Y.N. Wang, J.C. Huang, *Acta Mater.* **55**, 897 (2007)
- [33] R.D. Zhou, Dissertation, Dalian University of Technology, 2013
- [34] L. Wang, Y.M. Kim, J.H. Lee, B.S. You, *Mater. Sci. Eng. A* **528**, 943 (2011)
- [35] L.L. Chang, Y.N. Wang, X. Zhao, M. Qi, *Mater. Charact.* **60**, 991 (2009)
- [36] Y.N. Wang, C.I. Chang, C.J. Lee, H.K. Lin, J.C. Huang, *Scr. Mater.* **55**, 637 (2006)