Available online at www.sciencedirect.com

ScienceDirect

Journal of Magnesium and Alloys 1 (2013) 169–175
www.elsevier.com/journals/journal-of-magnesium-and-alloys/2213-9567

Full length article

Effects of caliber rolling on microstructure and room temperature tensile properties of Mg–3Al–1Zn alloy

R.L. Doiphode [a], S.V.S. Narayana Murty [b], N. Prabhu [a], B.P. Kashyap [a,*]

[a] *Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai 400076, India*
[b] *Special Materials Division, Vikram Sarabhai Space Center, ISRO, Trivandrum 695022, India*

Received 20 June 2013; accepted 24 July 2013

Abstract

Mg–3Al–1Zn (AZ31) alloy was caliber rolled isothermally in the temperature range of 250–450 °C to develop fine grains of 3.6–12.5 μm. The stress–strain curves obtained from tensile tests at room temperature were found to vary with the temperature employed in caliber rolling. Maximum tensile strength of 290 MPa and ductility of 13.5% were obtained upon caliber rolling at 300 °C as compared to 188 MPa and 15.2%, respectively, in the mill-rolled condition. The variations in tensile properties are explained by the concomitant grain size, texture and twins obtained as a function of caliber rolling temperature.

Keywords: AZ31 Mg-alloy; Caliber rolling; Twinning; Grain size; Tensile properties

1. Introduction

Magnesium alloys are becoming popular in the various sectors like aerospace, automobile, biomedical, architecture and electronic industries [1,2]. However, these alloys have limitation of mechanical processing because of their common hexagonal close packed (HCP) type crystal structure. Rolling is used to form the sheets, bars and rods of various shapes. To improve the mechanical properties of rolled material many changes in the conventional rolling were introduced [3–5], but the same introduce the changes in microstructure and texture of the rolled product [3,4,6–9]. Although it is established that materials with smaller grains exhibit higher yield stress, which can often be described by the Hall–Petch relationship [10,11], there appear some deviations on the effect of grain size [12–15]. Capolungo [16] showed that HCP metals deform plastically via the simultaneous activation of twinning and slip deformation modes. Munroe and Tan [17] studied the orientation dependencies of slip and twinning in HCP metals. Koike et al. [18] reported that the twinning deformation can provide additional slip systems, which can enhance the ductility. Barnett [19] explained that deformation obtained is not homogeneous; the wrought Mg-alloys display inhomogeneous deformation as influenced by the variation in the ease of basal slip orientation amongst grains, micro-textures, shear banding, twinning and grain boundary sliding. Wang et al. [20] reported a significant improvement in properties of Mg-alloy by controlling texture without grain refinement. Twinning behavior depends on the grain size as well as texture [21,22], with finer grained specimens exhibiting lower volume fraction of twins [23]; the formation of twins was enhanced by grain coarsening [24]. Koike et al. [25] explained the role of non-basal slip systems and dynamic recovery at room temperature in fine

* Corresponding author. Tel.: +91 2225767622.
E-mail address: bpk@iitb.ac.in (B.P. Kashyap).
Peer review under responsibility of National Engineering Research Center for Magnesium Alloys of China, Chongqing University

ELSEVIER Production and hosting by Elsevier

2213-9567 Copyright 2013, National Engineering Research Center for Magnesium Alloys of China, Chongqing University. Production and hosting by Elsevier B.V. Open access under CC BY-NC-ND license.
http://dx.doi.org/10.1016/j.jma.2013.07.005

grained Mg-alloy, which were considered to be responsible for large tensile ductility.

The mode of deformation and related micro mechanisms can be influenced by the mechanical processing techniques employed for grain refinement. There does not appear any study to show the effect of caliber rolling (CR) process on microstructure and tensile properties of Mg–3Al–1Zn (AZ31) alloy. In this technique, a pair of grooved rollers is turned in the opposite direction. The work-piece is compressed between the rollers and then reduced to the preset thickness or changed to the desired cross section over several roll passes. It is used to produce the products with high precision and strength in mass production [26]. In the present paper, the effects of caliber rolling at various temperatures on the microstructure, texture, twin formation and room temperature tensile properties of Mg-alloy AZ31 were investigated.

2. Experimental procedures

2.1. Material and processing

Mg-alloy AZ31B, in the form of rolled plate of 50 mm thickness, with chemical composition Mg–Al 3.0, Zn 1.0 and Mn 0.2 wt% was used. The CR was carried out at temperatures of 250, 300, 350, 400 and 450 °C to 12 mm diameter rods in a series of roll passes in a rolling mill.

2.2. Tensile tests

The round tensile specimens with gage diameter 4.5 mm and gage length 16 mm were machined. Room temperature tensile tests in rolling direction (RD) were carried out up to failure at a strain rate of $1 \times 10^{-4}\ s^{-1}$ for as received (AR) and caliber rolled materials in the Zwick–Roell Amsler Universal

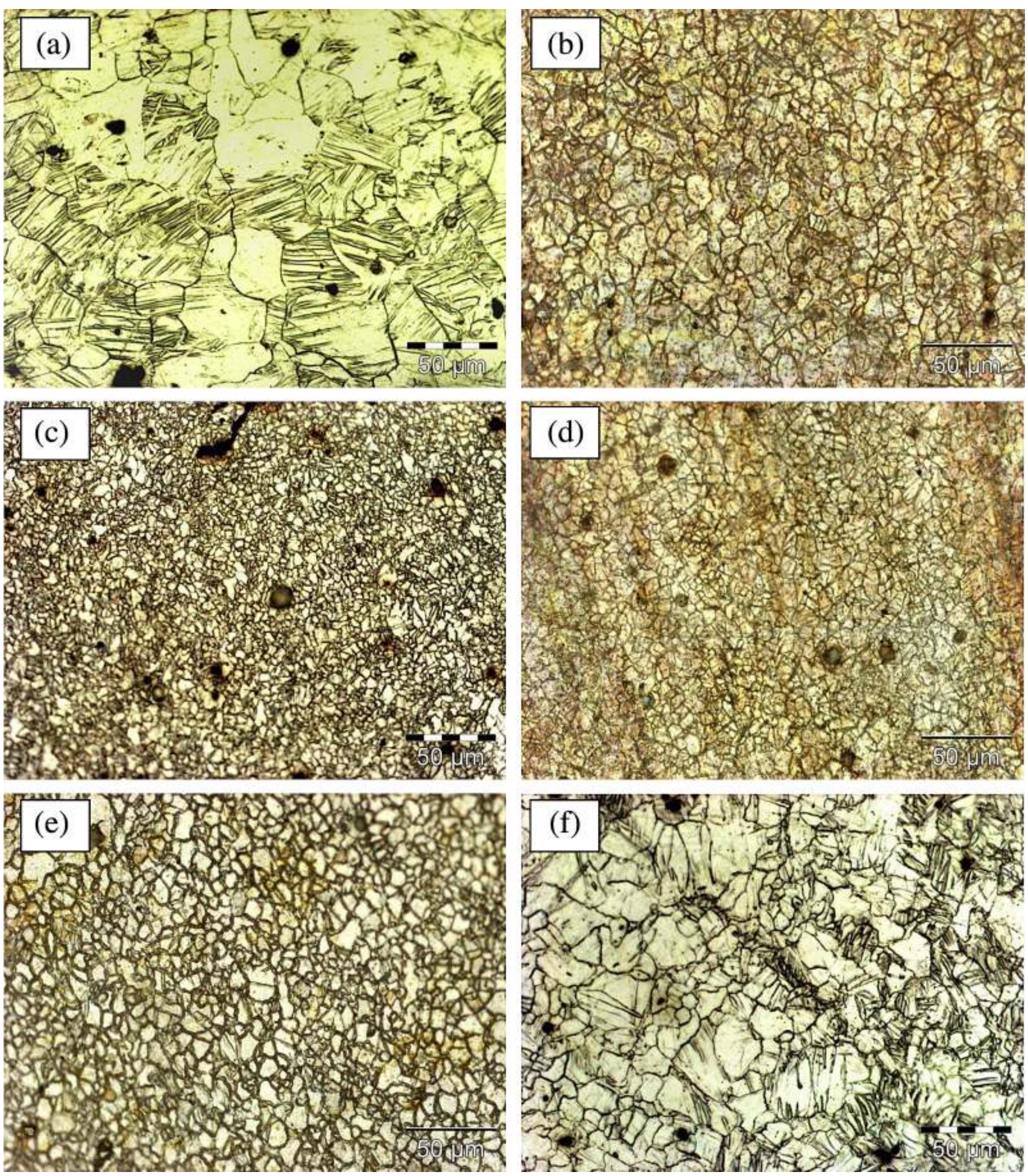


Fig. 1. Optical micrographs of the AZ31 Mg-alloy for (a) as-received plate and (b–f) caliber rolled at (b) 250 °C (c) 300 °C (d) 350 °C (e) 400 °C (f) 450 °C.

Testing machine of 100 kN load capacity. The ultimate tensile strength (UTS) and yield strength (YS) at a permanent elongation of 0.2% were determined.

2.3. Metallography and structural analysis

Metallographic specimen was prepared as per the ASTM procedure. The etching was carried out by acetic picral. The microstructures of specimens, caliber rolled at different temperatures, were examined and analyzed in RD before and after tensile deformation by Olympus GX51 optical microscope (OM). Grain size measurement was done by mean linear intercept method and error bars in mean intercept length, called grain size (d) here, are reported at 95% confidence level. Electron back scattered diffraction (EBSD) analysis was carried out by scanning electron microscope (SEM) Quanta 3D FEG with EBSD attachment, and TSL software was used for analysis. The variation of UTS, YS, plastic elongation, grain size, mis-orientation angle and twins were measured. The twins were measured in the area 250 × 250 μm^2 with a step size of 0.4 μm.

3. Results

3.1. Initial microstructure

The initial microstructure of the plate, as shown in Fig. 1(a), consists of equiaxed grains, with average size of 33.0 ± 9.0 μm along with a large number of twins. Fig. 1(b–f) shows the microstructures obtained after CR at 250, 300, 350, 400 and 450 °C respectively. The grain sizes after CR at these temperatures are 8.0 ± 1.5, 3.6 ± 0.5, 6.2 ± 1.5, 6.8 ± 1.6 and 12.5 ± 1.5 μm, respectively. The grain refinement was observed in all the CR conditions due to dynamic recrystallization [3]. With increase in temperature, there occurs a reduction in grain size up to CR temperature of 300 °C. However, above this temperature, the grain coarsening started with the rise in CR temperature. The reduction in number of twins was observed upon CR compared to initial microstructure. The number of twins before tensile deformation was found to increase as the grains become coarser, as illustrated in Table 1. However, no uniform relationship between number of twins formed, grain size and CR temperature was observed.

Table 1
Number of twins (in 250 × 250 μm^2 area) and maximum intensity of pole figure (0001) before tensile deformation (BTD) and after tensile deformation (ATD).

Specimen	Twins – $\{10\bar{1}2\}$		Max. intensity of pole figure (0001)	
	BTD	ATD	BTD	ATD
CR at 250 °C (d = 8.0 ± 1.0 μm)	1795	1333	5.136	2.71
CR at 300 °C (d = 3.6 ± 0.5 μm)	2184	853	2.865	2.656
CR at 350 °C (d = 6.2 ± 1.5 μm)	180	853	8.623	6.658
CR at 400 °C (d = 6.8 ± 1.6 μm)	1173	2944	5.627	6.038
CR at 450 °C (d = 12.5 ± 1.5 μm)	2722	851	12.596	4.687
As-mill rolled (d = 33.0 ± 9.0 μm)	4562	2892	13.061	16.67

3.2. Stress–strain curves

Fig. 2 shows true stress–true plastic strain curves from tensile tests conducted up to failure at room temperature at a strain rate of $1 \times 10^{-4}\ s^{-1}$ for all the CR temperatures and as-received material. The UTS for as-received material is found to be 188 MPa with plastic elongation of 15.2%. The UTS increased with simultaneous drop in ductility for the entire caliber rolled conditions. The maximum tensile strength of 290 MPa, 54% rise with respect to that of as-mill rolled material was observed, along with plastic elongation of 13.5% (i.e. 11.2% drop), in the material with CR temperature of 300 °C. Fig. 3(a) shows the variations in grain size and plastic elongation with CR temperature. Fig. 3(b) shows the variations in UTS and YS as a function of CR temperature. These values were also plotted (not shown here) as a function of $d^{-0.5}$, d being the grain size. However, the Hall Petch [10,11] relation was not followed, as evident from the regression coefficient R^2 value (0.33) obtained being very low.

Both the ductility and strength of the caliber rolled bar at 450 °C are noted to be greater than those subjected to CR at other temperatures, except at 300 °C. This is the case in spite of the fact that the grain size upon CR at 450 °C (12.5 ± 1.5 μm) is largest, as listed in Table 1 and plotted in Fig. 3(a) along with ductility. Fig. 3(b) shows the variation in YS and UTS as a function of CR temperature, and the same can be compared with the opposite nature of variation in grain size in Fig. 3(a), except in the temperature range of 400–450 °C. Also included in Fig. 3 are the variations in other microstructural properties to be described later.

3.3. Microstructural evolution

EBSD analysis was carried out for all the CR conditions and the as-mill rolled material before tensile deformation (BTD) and after tensile deformation (ATD) at room temperature. The name of the texture obtained is Harmonic L16,

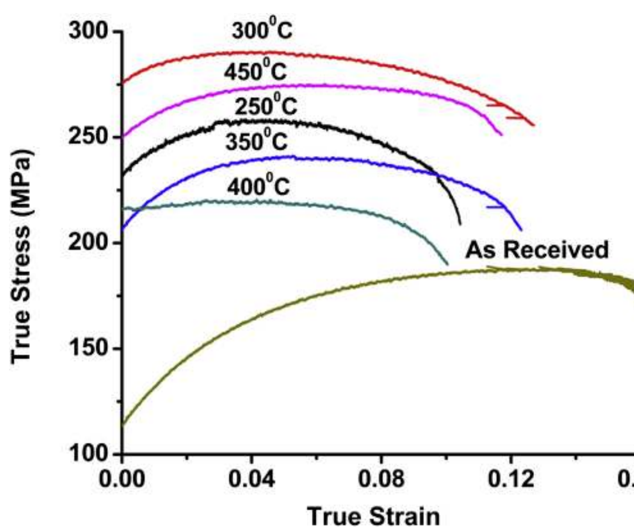
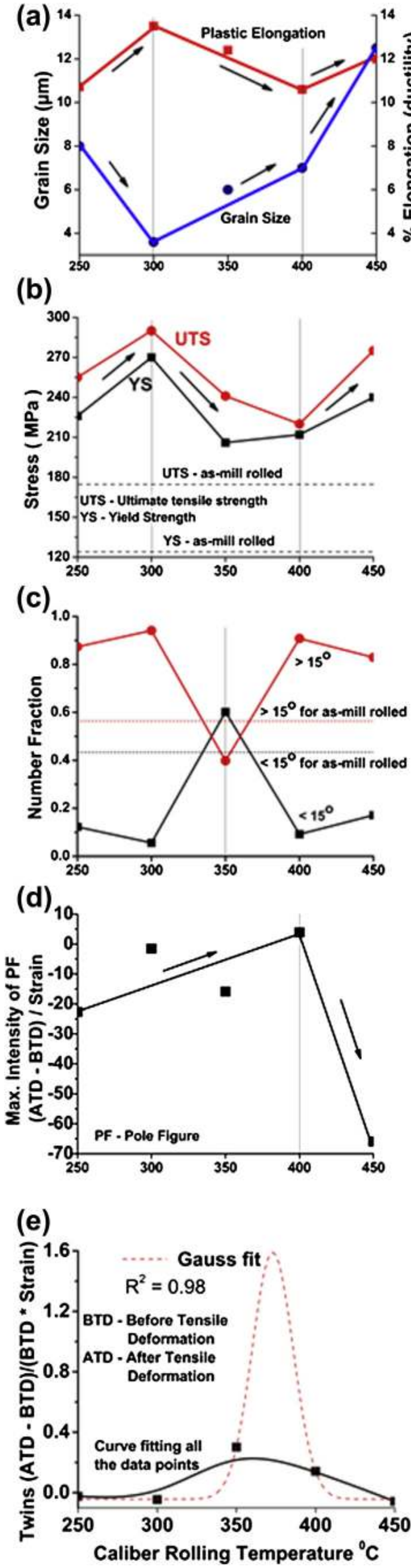


Fig. 2. True stress–true strain plots of AZ31 Mg-alloy for as-received and all the CR temperature conditions in plastic region.



HW5 as calculated by harmonic series expansion [27]. The (0001) basal pole figure along with the maximum intensity for as-received and all the CR conditions are shown in Fig. 4. The distribution of mis-orientation angle as a function of CR temperatures is plotted in Fig. 3(c). It is found that the number fraction of high angle grain boundaries increased with CR temperatures except that with CR at 350 °C. Table 1 shows the values of maximum pole figure intensity before and after tensile deformation for all the CR conditions and as-mill rolled material. This aspect will be discussed later. Fig. 5 shows the microstructure and (0001) basal pole figure upon tensile tests of as-mill rolled and caliber rolled material at 350 °C. Reduction in the grain size is observed in all the conditions of CR. Number of twins are reduced in as-mill rolled condition but it increased in the caliber rolled condition at 350 °C.

4. Discussion

There appears grain refinement after CR process but the variation in grain size with CR temperature is not systematic, Figs. 1 and 3(a). Similarly, the room temperature tensile properties do not follow a systematic trend with respect to CR temperature, Fig. 3(b), and so also with the resulting grain size. As a result, there appeared a deviation from the H−P relationship [10,11]. It is further observed that CR at 450 °C shows more strength and ductility, just below the CR at 300 °C, in spite of larger grain size at the former condition. It indicates that along with grain size there must be other microstructural elements to influence tensile properties. Based on the observations by Barnett [28], the probable reasons for this kind of behavior, upon rolling and subsequent to tensile deformation, are suggested and discussed below.

1. The difference in twin density measured, Table 1.
2. Different mis-orientation angles observed, Fig. 3(c).
3. Difference in texture formation, Fig. 4.

4.1. Interrelationship among various microstructures

Many limitations of magnesium alloys can be overcome by ultrafine grained (UFG) structure. CR at temperature 300 °C is found to be better for grain refinement among all the conditions employed. Probably, at this temperature, there occurs complete dynamic recrystallization, Fig. 1(c), whereas CR below this temperature may be incomplete as noted by partial dynamic recrystallization seen in Fig. 1(b). Processing at higher temperatures above 300 °C causes grain coarsening as observed in the microstructures shown in Fig. 1(d−f) along with the grain size data plotted in Fig. 3(a).

Fig. 3. Plot of structural and tensile properties as a function of CR temperature: (a) variation in grain size and plastic elongation (tensile ductility) (b) Ultimate Tensile Strength (UTS) and Yield Strength (YS) (c) number fraction of mis-orientation angle (d) normalized pole figure intensity with strain (tensile ductility) (e) normalized number of twins.

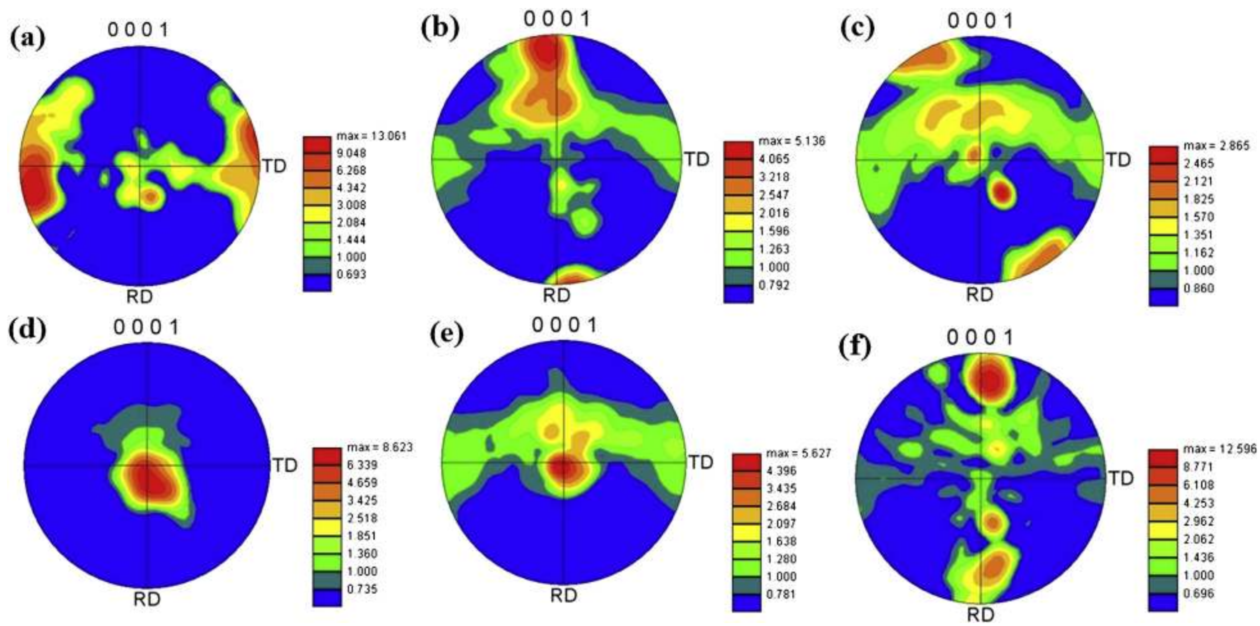


Fig. 4. (0001) Pole figures of the AZ31 Mg-alloy for (a) as-mill rolled plate and (b–f) caliber rolled at (b) 250 °C (c) 300 °C (d) 350 °C (e) 400 °C (f) 450 °C.

The effect of high temperature CR was observed on twin formation in the microstructure. Twins may be formed by deformation, recrystallization, grain growth and the movement of boundaries between different solid phases [27]. According to Christian and Mahajan, $\{10\bar{1}2\}$ mode gives the lower stress for deformation [22], so the same is considered for analysis of the tensile properties here. The number of twins is more for as-mill rolled material but the same decreased for all the CR temperature conditions, Table 1. Chino et al. [24] explained that there occurs an increase in number of twins as the grain size increases and vice versa. However, twins are not found to vary systematically with grain size and so also with CR temperature. A probable reason for this effect could be the different mis-orientation angles observed in the grains,

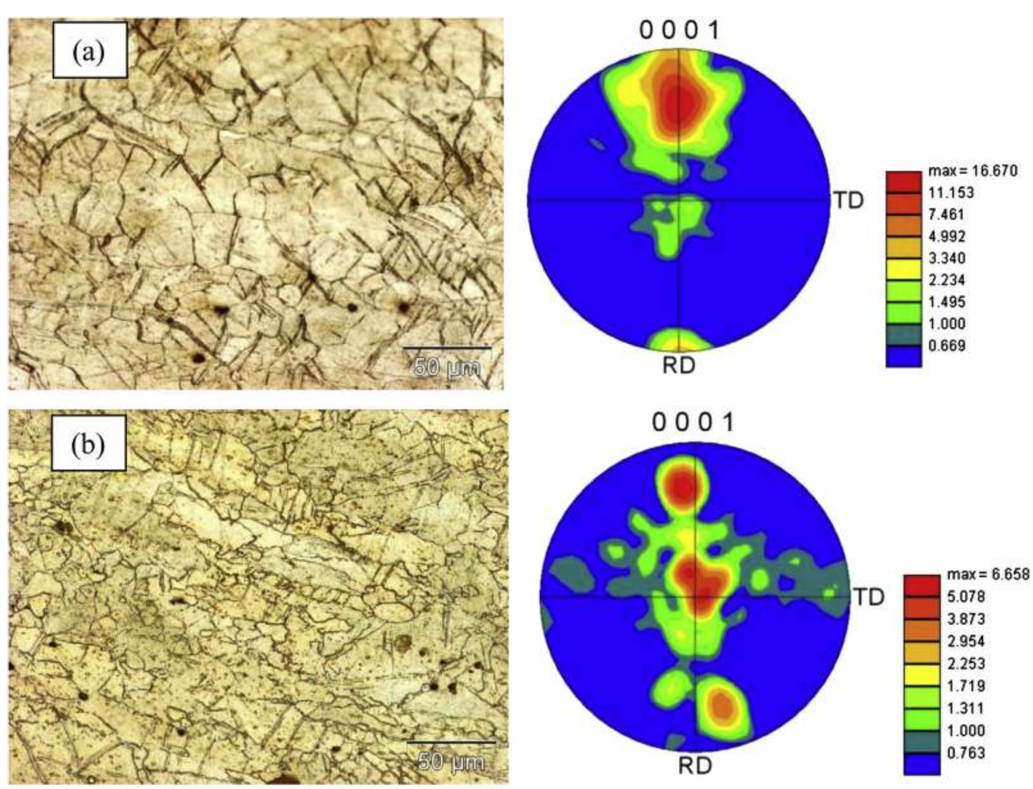


Fig. 5. Microstructure and (0001) pole figures of the AZ31 Mg-alloy after deformation of (a) as-mill rolled plate and (b) caliber rolled samples at 350 °C.

Fig. 3(c), and only the favorably oriented grains could facilitate their formation. It is also found that the grain size after CR at 300 °C is lowest but the number of twins is more, the reason for which could emerge from such variation in grain orientation.

Grain orientations in magnesium are rarely randomly distributed [27] but it does have ability to develop strong textures during processing [29]. The distribution of basal plane played an important role in the enhancement of mechanical properties at room temperature. The as-mill rolled material has (0001) basal texture with maximum intensity of ~13 and *c* axis towards TD, Fig. 4(a). After CR at 250 and 300 °C, there is a shift of *c* axis towards tensile (RD) axis with reduction in maximum intensity, Fig. 4(b,c). At these temperatures, there exists a possibility of dynamic recrystallization, which may replace the deformed structure by the nucleation and growth of recrystallized grains, and can lead to drastic change in texture [30]. Under such crystallographic change, secondary prismatic slip system may become effective through reduction in the critical resolved shear stress (CRSS). In response to the CR at temperatures 350 and 400 °C, there occurs formation of strong basal texture, which is concentrated in the center of pole figure, Fig. 4(d,e). There occurs reduction in the maximum intensity (from 13 in Fig. 4(a) to 5.6 (Fig. 4(e)) and more spread on the TD as seen in this figure. This could be a probable reason for the presence of greater proportion of low angle grain boundaries and less number of twins under these conditions. By increasing the CR temperature to 450 °C, there is possibility of activating first/second order pyramidal slip systems, which is a <$c + a$> type non-basal slip system, towards attaining greater random texture as compared in Fig. 4 for different CR temperatures. This is facilitated with the increase in CR temperature by thermal activation process of deformation and reduction in stress concentration during rolling. Thus, upon subsequent tensile deformation at ambient condition, the prior variation in texture during CR, along with other microstructural changes, can influence the mechanical properties [31]. There is random texture formation with very less reduction in maximum intensity and *c* axis towards RD as shown in Fig. 4(f), which could be the source of improved tensile properties, Fig. 3(b).

As the deformation is carried out in rolling direction there is shifting of *c* axis towards RD as shown in Fig. 5(a). Similarly for caliber rolled material, there is shifting of *c* axis towards RD as shown in Fig. 5(b) for CR temperature 350 °C. To normalize the maximum pole intensity at various CR temperatures the difference (ATD − BTD) was divided by respective plastic strain (tensile ductility) achieved during tensile deformation. The same is plotted against CR temperatures as shown in Fig. 3(d). It is found that there is sharp decrease in pole intensity after tensile deformation of the sample that had undergone CR at 450 °C. This is probably the effect of more twins present under this condition of CR temperature. Fig. 3(d) and (e) appear to exhibit roughly similar variations of maximum pole intensity and twin density respectively, both in the normalized condition. This suggests the change in texture and twins per unit strain increase for the samples CR up to temperature of 400 °C beyond which the same decrease rapidly. This indicates a relationship between the two microstructural features evolved during CR and subsequent tensile tests. This aspect of interrelationship between microstructures is noted to be a topic of detailed study in the literature [27,32].

4.2. Deviation from the Hall–Petch relationship

The Hall–Petch type relationship ($\sigma_y = \sigma_0 + k_y d^{-0.5}$) [10,11] predicts that as the grain size (d) decreases the yield strength (σ_y) increases. Here σ_0 and k_y represent the Hall–Petch constants having significance to the strengthening caused by grain interior and grain boundary, respectively. This strengthening by grain refinement is experimentally found to be true over the grain sizes ranging from 1 mm to 1 μm [27]. In HCP crystals, almost any fraction of grains can be converted to twins [27]. In view of this, it may be possible that the formation of twins could have aided to the deformation of the metals [18]. It was further noted that there is a tendency to decrease the twins ATD for higher twins existing BTD and the increase in twins ATD for lower twins BTD, Table 1. The deformation mechanisms are reported to exhibit different grain size dependencies of yield stress in magnesium alloys [33]. For example, transition from twinning to slip dominated flow occurs with decreasing grain size and increasing temperature. This is accompanied by a lowering of the Hall–Petch slope (k_y) for the yield stress. Further, the degree of work hardening drops, which gives rise to a “reverse” Hall–Petch effect as reported by Barnett et al. [33] for the flow stress at strains greater than 0.15. Consequently, the flow stress would either remain constant or decrease with decreasing grain size. This is probably the reason for the increased strength and ductility for the CR temperature 450 °C, in spite of its larger grain size. Fig. 6 shows the number of twins BTD plotted as a function of grain size after CR. Twins are noted to decrease up to grain size of 5 μm, after which there appears an increase in the number of twins with the increase in grain size. The rate of

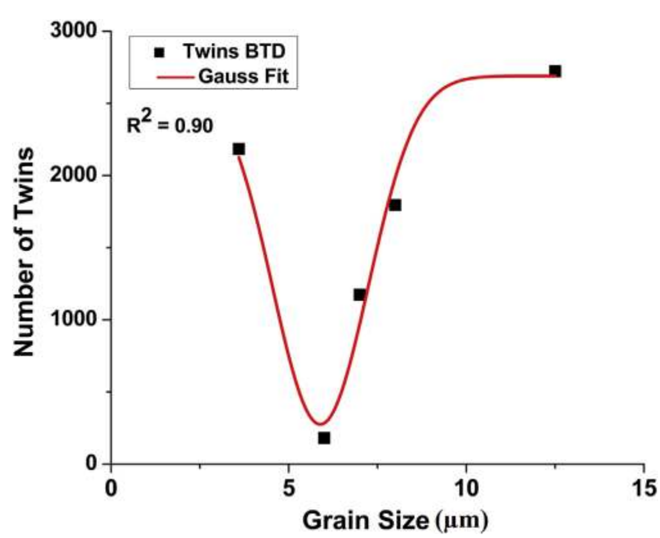


Fig. 6. Number of twins before tensile deformation as a function of grain size after CR.

increase in the number of twins is very high for the grain size increase between 5 and 10 μm, and there appear more of $\{10\bar{1}2\}$ twins (tensile twins) compared to other types of twin. The CRSS for the former twin is known to be less (15 MPa) than that of basal (30 MPa) and pyramidal (90 MPa) slip [34]. So, the formation of twins is suggested to improve the ductility in this material, as was also reported earlier [35].

5. Conclusions

Grain refinement was achieved from 33 μm in as-mill rolled Mg–3Al–1Zn alloy to 3.6 μm upon caliber rolling of about 50% at 300 °C. At this condition of caliber rolling, the UTS of 290 MPa with plastic elongation of 13.5% were achieved. The changes in the properties were found to be the effect of grain boundary strengthening but the deviation from the Hall–Petch relationship was attributed to the vital role played by twinning and texture in influencing the strength and ductility.

Acknowledgment

We express our thanks to DST for funding under FIST program SR/FST/ETII – 054/2000 for purchase of Universal Testing Machine and high temperature test facility.

References

[1] F. Mordike, Magnesium Technology, Metallurgy, Design Data Applications, Springer Berlin Heidelberg, New York, 2006.
[2] C. Blawert, N. Hort, K. Kainer, Trans. Indian Inst. Met. 57 (2004) 397–408.
[3] M. Qing, H. Lianxi, W. Guojun, W. Erde, Mater. Sci. Eng. A 528 (2011) 6694–6701.
[4] X. Huang, G. Haung, D. Xiao, Q. Liu, Mater. Sci. Forum 686 (2011) 40–45.
[5] W. Xia, Z. Chen, D. Chen, S. Zhu, J. Mater. Process. Technol. 209 (2009) 26–31.
[6] L. Guo, Z. Chen, L. Gao, Mater. Sci. Eng. A 528 (2011) 8537–8545.
[7] T. Mukai, H. Somekawa, T. Inoue, A. Singh, Scr. Mater. 62 (2010) 89–94.
[8] T. Inoue, F. Yin, Y. Kimura, Mater. Sci. Eng. A 466 (2007) 114–122.
[9] Y. Cheng, Z. Chen, W. Xia, T. Zhou, J. Mater. Eng. Perform. 17 (2008) 15–19.
[10] E.O. Hall, Proc. Phys. Soc. London B 64 (1951) 747–753.
[11] N.J. Petch, J. Iron Steel Inst. 174 (1953) 25–28.
[12] J. Koike, Y. Sato, D. Ando, Mater. Trans. 4912 (2008) 2792–2800.
[13] A. Jain, O. Duygulu, D. Brown, C. Torve, S. Agnew, Mater. Sci. Eng. A 486 (2008) 545–555.
[14] M.R. Barnett, C. Davies, X. Ma, Scr. Mater. 52 (2005) 627–632.
[15] M.R. Barnett, N. Stanford, Scr. Mater. 57 (2007) 1125–1128.
[16] L. Capolungo, Acta Mater. 59 (2011) 2909–2917.
[17] N. Munroe, X. Tan, Scr. Mater. 36 (1997) 1383–1386.
[18] J. Koike, R. Ohyama, T. Kobayashi, M. Suzuki, K. Maruyama, Mater. Trans. 44 (2003) 445–451.
[19] M.R. Barnett, Mater. Sci. Forum 618-619 (2009) 227–232.
[20] H. Wang, P.D. Wu, M.A. Gharghouri, Mater. Sci. Eng. A 527 (2010) 3588–3594.
[21] M. Meyers, O. Vohringer, V. Lubarda, Acta Mater. 49 (2001) 4025–4039.
[22] J.W. Christian, S. Mahajan, Prog. Mater. Sci. 39 (1) (1995) 1–157.
[23] N. Ecob, B. Ralph, J. Mater. Sci. 18 (1983) 2028–2035.
[24] Y. Chino, K. Kimura, M. Mabuchi, Acta Mater. 57 (2009) 1476–1485.
[25] J. Koike, T. Kobayashi, T. Mukai, H. Watanabe, M. Suzuki, K. Maruyama, K. Higashi, Acta Mater. 51 (2003) 2055–2065.
[26] Y. Tanno, T. Mukai, M. Asakawa, M. Kobayashi, Mater. Sci. Forum 419 (2003) 359–364.
[27] C.S. Barrett, T.B. Massalski, Structure of Metals, Crystallographic Methods, Principles and Data. Int. Ser. Mater. Sci. Tech, third ed., 1980.
[28] M.R. Barnett, Mater. Sci. Eng. A 464 (2007) 1–7.
[29] N. Ecob, B. Ralph, J. Mater. Sci. 18 (1983) 2419–2429.
[30] Y.N. Wang, J.C. Huang, Mater. Chem. Phys. 81 (2003) 11–26.
[31] F. Kang, L. Zheng, J. Wang, P. Cheng, H. Wu, J. Mater. Sci. 47 (2012) 7854–7859.
[32] Y. Chun, S. Yu, S. Semiatin, S. Hwang, Mater. Sci. Eng. A 398 (2005) 209–219.
[33] M.R. Barnett, Z. Keshavarz, A.G. Beer, D. Atwell, Acta Mater. 52 (2004) 5093–5103.
[34] S.R. Agnew, M.H. Yoo, C. Tome, Acta Mater. 49 (2001) 4277–4289.
[35] M.H. Yoo, J.R. Morris, S.R. Agnew, Metall. Mater. Trans. 33A (2002) 813–822.