Multi-passes warm rolling of AZ31 magnesium alloy, effect on evaluation of texture, microstructure, grain size and hardness

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Abstract. In this study the effect of multi-passes warm rolling of AZ31 magnesium alloy on texture, microstructure, grain size variation and hardness of as cast sample (A) and two rolled samples (B & C) taken from different locations of the as-cast ingot was investigated. The purpose was to enhance the formability of AZ31 alloy in order to help manufacturability. It was observed that multi-passes warm rolling (250°C to 350°C) of samples B & C with initial thickness 7.76mm and 7.73 mm was successfully achieved up to 85% reduction without any edge or surface cracks in ten steps with a total of 26 passes. The step numbers 1 to 4 consist of 5, 2, 11 and 3 passes respectively, the remaining steps 5 to 10 were single pass rolls. In each discrete step a fixed roll gap is used in a way that true strain per step increases very slowly from 0.0067 in the first step to 0.7118 in the 26th step. Both samples B & C showed very similar behavior after 26th pass and were successfully rolled up to 85% thickness reduction. However, during 10th step (27th pass) with a true strain value of 0.772 the sample B experienced very severe surface as well as edge cracks. Sample C was therefore not rolled for the 10th step and retained after 26 passes. Both samples were studied in terms of their basal texture, microstructure, grain size and hardness. Sample C showed an equiaxed grain structure after 85% total reduction. The equiaxed grain structure of sample C may be due to the effective involvement of dynamic recrystallization (DRX) which led to formation of these grains with relatively low misorientations with respect to the parent as cast grains. The sample B on the other hand showed a microstructure in which all the grains were elongated along the rolling direction (RD) after 90% total reduction and DRX could not effectively play its role due to heavy strain and lack of plastic deformation systems. The microstructure of as cast sample showed a near-random texture (mrd 4.3), with average grain size of 44 µm & micro-hardness of 52 Hv. The grain size of sample B and C was 14µm and 27µm respectively and mrd intensity of basal texture was 5.34 and 5.46 respectively. The hardness of sample B and C came out to be 91 and 66 Hv respectively due to reduction in grain size and followed the well known Hall–Petch relationship.

1. Introduction
Magnesium is a very important advanced structural material and has received significant research attention for automobile aerospace and electronic applications due to its low density (1.7 g/cm³, which is 2/3rd of aluminum & 1/5th of steel), high specific strength and adequate specific stiffness [1-3]. The combination of low density and reasonable strength of magnesium helps in weight reduction of components for automobiles and aerospace applications [4]. A lot of research work has been carried out to gain an in depth understanding on the deformation behavior of wrought Mg alloys as compared to cast alloys that normally have low strength and poor ductility. The use of wrought magnesium
alloys in the form of sheet is increasing in recent years. In this regard Mg-Al-Zn type alloy AZ31 has got marvelous industrial applications for sheet production by rolling [5].

One of the most critical issues currently limiting applications of Mg based alloys is their poor formability & plastic flow characteristics at room temperature because of HCP structure and limited available active slip systems [6]. Therefore, the forming processes of AZ31 alloy are carried out at elevated temperatures in order to avoid edge as well as surface cracking. Another obstacle to the formability of the magnesium sheet production is the formation of strong basal texture, which leads to a greater anisotropy and affects the enhancement of its press formability [7]. It is well known that the formability of magnesium alloys can be improved significantly by changing the (0002) basal texture [8,9]. Therefore, many researchers are working to weaken the basal texture either by using severe thermomechanical processes (TMP) or by modifying the well known wrought alloys by addition of alloying elements. Literature reports that some TMP have been proven successful to weaken the basal texture like equal channel angular pressing(ECAP) [10,11], cross-roll rolling [12], differential speed rolling(DSR) [13,14], cyclic extrusion compression (CEC) [15], friction stir process(FSP) [16,17] and repeated unidirectional bending(RUB) [18,19]. Though these processes are helpful in controlling the basal texture, yet all of them are not used for the purpose of manufacturing of sheets.

Present study is an effort to increase the formability of AZ31 through conventional rolling carried out in steps with different number of rolling passes in each step. The roll gap in each pass was fixed in a way that the resulting true strain increases very slowly. In this regard warm temperature range (250-350°C) was selected due to the fact that deformation in warm forming temperature range is now well known to generate dynamic re-crystallization (DRX), deformation twins, and dislocation slips. The main objective of this work is to establish the optimum process parameters for multi-pass warm rolling which can help in improving the formability of the AZ31 magnesium alloy by improving (1) basal texture (2) microstructure (3) grain refinement and (4) mechanical properties.

2. Experimental
The Mg–3Al-1Zn (wt. %) magnesium alloy (abbreviated as AZ31) was prepared from high purity elements Mg (99.99%), Al (99.99%) and Zn (99.99%). The charge was melted in an iron crucible using an electrical resistance furnace, in the presence of high purity Argon gas to protect the melt from oxidation. After stabilization at 780 °C for about 5 min, the melt was poured into a copper mold with dimensions (80 × 75 × 8 mm). After cooling in Ar atmosphere the cast ingot was demolded and three samples A, B & C were collected from the ingot from the location shown in figure 1, their dimensions are listed in table 1. The chemical analysis was carried out by inductively coupled plasma atomic emission spectroscopy (ICP), results are shown in Table 2.

Figure 1. Location of the samples taken from warm rolling.
Table 1. Dimensions of two samples before and after multi pass warm rolling.

| Samples   | Initial Dimensions (mm) | Final Dimensions (mm) | Reduction (%) |
|-----------|-------------------------|-----------------------|---------------|
|           | Length | Width | Thickness | Length | Width | Thickness |             |
| Sample B  | 33.12  | 17.79 | 7.76      | 315    | 23.63 | 0.78      | 90          |
| Sample C  | 24.89  | 18    | 7.73      | 88.73  | 29.11 | 1.16      | 85          |

Table 2. Chemical Composition of AZ31 magnesium alloy (wt %).

| Materials | Mg %   | Al % | Zn % | Fe ppm | Cu ppm | Mn ppm | Ni ppm |
|-----------|--------|------|------|--------|--------|--------|--------|
| Pure Mg   | Balance| ND   | ND   | ND     | ND     | 13     | ND     |
| AZ31      | Balance| 3.0  | 1.02 | 51     | ND     | 20     | ND     |

Sample A was retained in the as-cast condition for determination of texture, microstructure, grain size and hardness. A unidirectional multi-pass warm rolling (250-350 °C) of as-cast samples B and C was carried out in JOLIOT rolling machine without any lubricant with roll diameter of 124 mm and roll length of 150 mm. Rolling speed was fixed at 27 rev/min. Samples B & C were preheated at 400 °C in air for 2 hours in induction heating furnace prior to the start of rolling. After two hours the furnace was switched off and samples were allowed to cool in furnace as soon the temperature was dropped to 350 °C, multi-pass warm rolling was started at 300 °C with an intermediate heating time of 5 minutes according to the scheme given in Table 3. The step numbers 1, 2, 3 and 4 consist of 5, 2, 11 and 3 passes respectively while the next 5 steps consist of only 1 pass each. In each discrete step a fixed roll gap was used in a way that value of true strain per step might increase very slowly from 0.0067 in the first step to 0.7118 in the 26th step.

The plane of examination for microstructure and macro-texture examinations of as cast sample was that of solidification direction (SD) whilst in case of rolled samples same study was performed on the plane containing rolling direction (RD) & transverse direction (TD). The samples for microstructure examination were ground by using SiC papers in the sequence of 1200-grit, 1500 grit and 2000 grit. Polishing was carried out by means of solution of alumina 0.05 µm and ethanol. After final polishing the samples B & C were etched by using acetic picral with the composition: acetic acid 10 ml, acetic picral 4.25g, distilled water 10ml, and ethanol 70 ml. The microstructures were analyzed using OLYMPUS optical microscope (OM). Texture evaluation was carried out by X-ray diffraction (XRD) technique using a Bruker D8 diffractometer with CuKα radiation. The hardness measurements were performed by Struers Duramin micro-Vickers apparatus under the load of 1.96 N for the duration of 10 seconds.
Table 3. The multi pass warm rolling schedule for Sample B and C

| No of Steps | No of Passes | The Pass Number | Reduced Thickness (mm) | True Strain (%) | Total thickness reduction in a step [Δx] (mm) | Reduction per pass [Δx/no of passes] (mm) |
|-------------|-------------|-----------------|-----------------------|-----------------|-------------------------------------------|------------------------------------------|
| 0           | 0           |                 | 7.76                  | 0              | 0                                         | 0                                         |
| 1           | 5           | 1-5             | 7.5                   | 0.034           | 0.26                                      | 0.052                                     |
| 2           | 2           | 6-7             | 6.89                  | 0.085           | 0.61                                      | 0.305                                     |
| 3           | 11          | 8-18            | 5.1                   | 0.301           | 1.79                                      | 0.162727                                  |
| 4           | 3           | 19-21           | 4.23                  | 0.187           | 0.87                                      | 0.29                                     |
| 5           | 1           | 22              | 3.79                  | 0.11            | 0.44                                      | 0.44                                     |
| 6           | 1           | 23              | 3.25                  | 0.154           | 0.54                                      | 0.54                                     |
| 7           | 1           | 24              | 2.67                  | 0.197           | 0.58                                      | 0.58                                     |
| 8           | 1           | 25              | 1.94                  | 0.32            | 0.73                                      | 0.73                                     |
| 9           | 1           | 26              | 0.952                 | 0.55            | 0.988                                     | 0.988                                    |
|             | 1           | 27              | 0.44                  | 0.772           | 0.512                                     | 0.512                                    |
| Total       | 27          |                 | 7.32                  | 2.872           | 7.32                                      |                                          |

3. Results and Discussion

Figure 2(a), (b) and (c) shows the external appearance, microstructure and basal planes texture of as cast sample A of AZ31 magnesium alloy respectively. The as-cast ingot microstructure was a typical non-equilibrium cast structure which consists mainly of α Mg matrix (light region) and second phase precipitates (dark region). The microstructure of as cast sample shows an average grain size of 44 µm. The second phase precipitates distribute continuously along the grain boundaries. Some black spots were also observed at grain boundaries as well as within the grains. As-cast microstructure also showed few twins, however their volume fraction was very low. Figure 2(c) shows random distribution of basal poles in as cast condition with a mrd of 4.3.

Figure 2(d), (e) and (f) shows the external appearances, microstructure and associated basal planes texture of sample C respectively after total thickness reduction of 85%. The rolling schedule is provided in table 3. The micrograph of this sample shows an equiaxed grain structure [figure. 2(e)] with an average grain size of 27 µm evaluated by linear intercept method. It is envisaged that these refined equiaxed grains are mainly induced by the dynamic recrystallization (DRX) involved during the repeated pre-heating for about 5 min during multi-pass warm rolling [24]. Another interesting result of sample C is that optical micrograph exhibits twin free grains. Such twin free and equiaxed microstructure is the characteristics of annealed samples followed by deformation either by rolling or extrusion etc. Twin free grains microstructure mostly produce a strong basal texture of as-rolled plate due to the fact that twinning is one of the most important deformation mechanism involved in magnesium alloys [20]. Basal pole figure shown in [figure. 2(f)] revealed that the basal planes are trying to reorient their c-axis parallel to the compression axis. Once most of the c-axes are parallel to the compression axis by repetitive rolling, {1012} twinning will be suppressed because {1012} twinning is an extension twin which is active under loading perpendicular to the compression axis. Thus, the possibility of the occurrence of deformation twinning as an additional deformation mode will decrease with an increase in the number of rolling passes during multipass warm rolling [21].
Similar relation of equiaxed microstructure and basal texture was observed while studying the role of tension twins on warm deformation behavior of AZ31 Mg alloy [20]. However, in the present case the intensity of basal texture could only be lowered down to 5.46 mrd.

The splitting of basal poles from normal direction (ND) towards the rolling as well as transverse direction (TD) is shown in figure 2(f). The resulting double peak texture represents a clear tendency for a broad c-axes distribution in the RD and TD, which supports the findings of Li et al. [22] who related it with improved formability. They reported that the deformation was carried out primarily by basal slip assisted by prismatic and <c + a> pyramidal slip. This splitting of basal poles in front of and behind the RD after final rolling pass on sample B reflects indeed a significant improvement in the sheet formability/rollability by decreasing the basal poles intensity [23-24]. Similar double peak basal poles figures were observed by Kohzu et al. during combination of rolling and high-temperature annealing of AZ31 alloy [25].

![Figure 2](image-url)

**Figure 2.** External appearance, microstructures and associated basal pole texture of AZ31 magnesium alloy before multipass rolling and after rolling with a total of 85 % and 90% thickness reduction: (a, b,c) As cast Sample A (d,e,f) 85 % rolled sample C, (g,h,i) 90% Rolled sample sample B.

The microstructural features shown in figure 2 are due to the fact that a high degree of deformation gives rise to a high fraction of DRX structure. Moreover, examination of the microstructural development in AZ31 for different rolling reductions per pass revealed that rolling reductions larger than 53% at warm rolling temperature result in a high fraction of shear banding [25]. It is well known that a shear band is one of the microstructural features that evolve under inhomogeneous deformation [26]. Since conventional rolling is not a complete homogeneous deformation process, therefore, it is
very difficult to prevent shear bands formation at high strains ($\varepsilon = 0.772\%$). It is however, reported that the formation of shear bands can be delayed by multi-axial deformation [27,28] which claims a much more homogeneous microstructure during deformation. Therefore, the formation of shear bands during deformation is of great importance to the formability of Mg alloys.

The decrease in the intensity of the {0002} basal plane with the increase of the reduction ratio per pass may be due to delayed formation of shear bands. These shear bands are highly localized deformed regions in which more random grain orientations and thus weaker texture are developed as shown in basal pole figure 2(i). The other aspect of heavy reduction per pass is reflected into fine grain size and high hardness values as shown in figure 3. There is a great difference in grain size and microhardness of both the samples B & C. It appears that repetitive rolling at a smaller reduction ratio per pass up to total of 85% produced an equiaxed and coarse grains structure during DRX in both the samples up to 26$^{th}$ rolling pass according to rolling schedule shown in Table 3. However, as the % age reduction per pass was increased with true strain $\varepsilon = 0.772\%$, a drastic change in microstructure, grain size, grain shape and texture intensity was observed. These findings are in good agreement with previous studies [29].

The present results of grain size and experimentally measured micro-hardness are also well in agreement with the well known Hall-Petch relation (for grain size vs hardness plot) as shown in figure 3. The reduction in grain size of sample B is almost half of sample C. Accordingly the hardness showed an increase of up to 91 Hv during last rolling pass at high reduction per pass at $\varepsilon = 0.772\%$. The grain size distribution becomes more homogeneous and the average grain size becomes finer (14 µm) with an increase in the reduction ratio per pass in 27$^{th}$ pass as shown in figure. 1(h). Researchers are in agreement with the fact that a homogeneous and fine-grained structure could be obtained by using larger reductions. Moreover, it is reported that weaker texture results from greater reductions [30, 31]. Microstructure of this sample consists of elongated grains after total reduction of 90%. The basal pole texture intensity of sample B is a bit lower than that of samples C as shown in figure 2(i). It seems that in sample B most of the grains are trying to align their c-axes parallel to the compression axis as a result of a very high strain during the 27$^{th}$ pass. The increase of dislocation density resulting from high reduction in final pass seems to be the main reason for strain hardening due to which the dislocation pileup increases and eventually leading to the failure of sample B. It is well established that the evolution of a very high density of uniformly distributed dislocations provide very high strain hardening. Internal stress fields cause the initiation of non-basal slip [32] which might have lowered the basal planes texture of sample B.

**Figure 3.** (a) The variation of grain size as a result of multipass warm rolling after total reduction of 85% and 90%. (b) effect of grain size on microhardness after same reduction.
4. Summary and Conclusions
It is concluded that both samples B and C exhibited a similar manner up to 85% total reduction in 26 passes. During the 27th pass and reaching a value of true strain of 0.772 %, a drastic change in grain size and grain morphology of sample B was observed and the sample failed. The formation of edge and surface cracks seem not due to strong basal texture but due to a very heavy, 53% thickness reduction, in this pass. In short present study shows that formability can be increased through multi pass warm rolling. Adopting this strategy helped in successful rolling of AZ31 with conventional procedures due to the role of DRX. However, from an industrial point of view the economic feasibility of a long processing time may be taken into account.

5. References
[1] Lee C D 2006 Met. Mater. Int. 12 377
[2] Yim C D, Wu G and You B S 2007 Mater. Trans. 48 2778
[3] Kang S H, Lee Y S and Lee J H 2008 J. Mater. Proc. Tech. 201 436
[4] Easton M, Beer A, Barnett M R, Davies C, Dunlop G, Durandet Y, Blacket S, Hilditch T, and Beggs P 2008 JOM 60 57
[5] Spigarelli S, Mehtedi M E, Cabibbo M, Evangelista E, Kaneko J, Jäger A and Gartnerova V 2007 Mater. Sci. Eng. A 462(1–2) 197-201
[6] Kim H L and Chang Y W 2011 Met. Mater. Int. 17 563 (2011)
[7] Yi S B, Bohlen J, Heinemann F, Letzig D 2010 Acta Mater. 58 592–595
[8] Mukai T, Yamano M, Watanabe H and Higashi K 2001 Scripta Mater. 45 89–94
[9] Chino Y, Lee J, Sassa K, Kamiya A and Mabuchi M 2006 Mater. Lett. 60 173–176
[10] Tong L B, Zheng M Y, Hu X S, Wu K, Xu S W, Kamado S and Kojima Y 2010 Mater. Sci. Eng. A 527 4250–4256
[11] Kim W J, Hong S, Kim Y S, Min S H, Jeong H T and Lee J D 2003 Acta Mater. 51 3293–3297
[12] Chino Y, Sassa K, Kamiya A and Mabuchi M 2006 Mater. Sci. Eng. A 441 349–356
[13] Huang X S, Suzuki K, Watazu A, Shigematsu I and Saito N 2009 J. Alloys Comp. 470 263–268
[14] Lee J B, Konno T J and Jeong H G 2010 J. Alloys Comp. 499 273–277
[15] Chen Y J, Wang Q D, Roven H J, Karlsen M, Yu Y D, Liu M P and Hjelen J 2008 J. Alloys Comp. 462 192–200.
[16] Lee H W, Lui T S and Chen L H 2009 J. Alloys Comp. 475 139–144
[17] Darras B M, Krhaisher M K, Abu-Farha F. K and Omar M A 2007 J. Mater. Proc. Technol. 191 77–81
[18] Huang G S, Xu W, Huang G J, Li H C and Song B 2009 J. Mater. Sci. Technol. 25 365–369
[19] Zhang L, Huang G S, Zhang H, Song B 2011 J. Mater. Proc. Technol. 211 644–649
[20] Park J H, Kim H L, Jung J E and Chang W Y 2013 Met. Mater. Int. 19 389-398
[21] Yim C D, Seo Y M and You B S 2009 Met. Mater. Int. 15 683-688
[22] Li X, Al-Samman T, Mu S and Gottstein G 2011 Mater. Sci. Eng. A 528 7915-7925
[23] Kohzu M, Hironaka T, Nakatsuka S, Saito N, Yoshida F, Naka T, Okahara H and Higashi K 2007 Mater. Trans. 48 64-768
[24] Kohzu M, Nakatsuka S and Higashi K 2008 Mater. Trans. 49 2096-2099
[25] Kohzu M, Kii K, Nagata Y, Nishio H, Higashi K and Inoue H 2010 Mater. Trans. 51 749-755
[26] Yim C D, Seo Y M and You B S 2009 Met. Mater. Int. 15 683–688
[27] Lee C S and Duggan B J 1994 Acta Mater. 42 857
[28] Liu W C, Li X Y and Meng X C 2009 Scripta Mater. 60 768–771
[29] Humphreys F J and Hatherly M 1995 Recrystallization and Related Annealing Phenomena (NewYork: Pergamon) p. 363-392
[30] Sun H, Liang S and Wang E 2009 Trans. Nonferrous Met. Soc. China, 19, 349–354
[31] Jeong H T and Ha T K 2007 J. Mater. Proc. Tech. 187–188 229–561
[32] Muránsky O, Carr D G, Barnett M R, Oliver E C and Sittner P (2008) Mater. Sci. Eng. A 496 14-24