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Edited by: Suveen N. Mathaudhu, Wim H. Sillekens, Neale R. Neelameggham, and Norbert Hort
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EFFECT OF ROLLING TEMPERATURE ON THE AZ31B MAGNESIUM ALLOY MICROSTRUCTURE

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Abstract

The influence of rolling temperature and the effect of strain rate on the microstructure of AZ31B Mg alloy were determined in order to improve its formability. A plate of AZ31 alloy was found to be sensitive to strain rate at high temperature and anisotropy was adversely impacted in cold rolling sheets. Thus, AZ31B has better workability within the temperature range of 200 to 250 °C, due to the grain refinement, caused by recovery and dynamic recrystallization. The effect of rolling temperature was studied on recrystallized sheets (2 mm in thickness) which were deformed by rolling at different temperatures (25, 100, 200 and 250 °C) and the effect of strain rates was evaluated on two different rolling speed (10 and 20 rpm). The microstructural characterization was achieved using several complementary techniques of microstructural analysis, such as optical microscopy, scanning electron microscopy, X-ray analysis by energy dispersive, X-ray diffraction and microhardness.

Introduction

The AZ31B magnesium alloy is a promising structural light material due to its high specific strength and high specific stiffness. Therefore, it is desired to improve the ductility and workability in order to increase its applications, especially in automotive and aerospace industry [1]. Many researches were carried out to enhance the formability of Mg alloy sheets at high temperature. However, there have been a few investigations on the room temperature formability of Mg alloys. It is known that plastic deformation in metals at low temperatures occurs as a result of two mechanisms: slip and twinning. Twin formation depends on the orientation of the crystals which make up the alloy with respect to the applied stresses [1, 2].

Formability of magnesium alloys greatly increases at high temperatures accompanied by a decrease in flow stress, an increase in strain rate sensitivity, and a decrease in normal anisotropy. The value of the critical resolved shear stress (CRSS) of the non-basal slip systems decreases at high temperature improving the ductility. Furthermore, an increase in the deformed temperature also affects the recrystallization behavior of magnesium alloys. Once a critical strain is exceeded, it is possible that during and after deformation occur both dynamic recrystallization and static recrystallization, respectively. Their mechanisms change the grain morphology, and are function of deformation temperature, strain, strain rate, deformation mode, grain size, and grain orientation [3].

Recently, it was showed that grain refinement is effective for activation of prismatic slip at grain boundary, because grain refinement promotes stress concentration at grain boundary, resulting in activation of prismatic slip. Thus, the deformation mechanisms of Mg alloys depend not only of five independent slips, but also the relaxation mechanisms for the stress concentration at the grain boundary and formation of twins, which is produced at higher strain rates and lower temperatures. Their formation strongly depends on grain size [5]. Therefore, it possible to state that during deformation in a coarse-grained Mg alloy, twinning plays a vital role, not in a fine-grained Mg alloy [10]. Yet, effects of the grain size on roles of twinning in the deformation are not still completely understood.

In the present research, the effect of rolling temperature on microstructural evolution is studied at cold and warm plastic deformation on sheets of AZ31B alloys, using microstructural characterization tools.

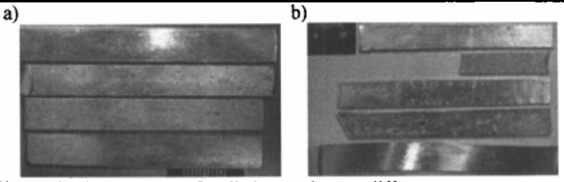
Experimental details

The received material was a plate of 2 mm in thickness of AZ31B magnesium alloy with a nominal chemical composition of 3.34–3.63 wt% Al, 0.45–0.53 wt% Zn, 0.27–0.29 wt% Mn, and balance Mg. It was produced by twin-roll caster by Sinomag Company, China. This sheet was cut into plates with approximate dimensions of 50x10 mm^2 for rolling experiments.

Each samples were heated up at rolling temperature (25, 100, 200 and 300 °C) and kept at this temperature for 10 min, then sent to a small sized rolling mill of 108 mm in diameter for rolling at a single pass. The rolling speeds were maintained at 10 rpm, without any lubricant. Such rolling processes were carried out controlling the compressive reduction in thickness near 10-15% for cold rolling (25 and 100 °C), and near 15-20% for warm rolling (200, and 250 °C). For the same temperature range, the rolling speed was increased to 20rpm and the compressive reduction in thickness was increased to 30% for every rolling temperature.

Table 1 – Table showing the temperature and reduced thickness of the sample sheet with one pass.

10 rpm			20 rpm
Temperature °C	Reduced thickness (%)	strain rates (s^{-1})	Reduced thickness (%)
25	10	1.6	30
100	15	2.3	30
200	15	2.3	30
250	20	2.8	30



a) b)

Figure 1. Appearance of rolled samples at different temperature and rolling speeds: a) 10 rpm and b) 20 rpm for 5.7 (s^{-1}) strain rates.

The appearance of the thinnest sheets after rolling is showed on Figure. 1 and the summary of the reduced thickness, strain rates and variation of rolling speeds are on Table 1. In the figure 1b is evident that the deformation strain is accommodated partly by large amounts of shear bands, which are very common features for AZ31 deformed by rolling.

For metallographic analysis, the as-rolled samples were cut into a square of 10mm×10mm in dimensions. The observed positions were on rolling sections parallel to rolling direction. All samples were ground with SiC papers and etching the samples surface with two mixed solution of nitric acid in diethylene glycol and acetic-picral to reveal the grain structure. Metallurgical structure was observed by using an Olympus PME-3 optical microscopy. The crystal structure and network parameters were determined using X-ray diffraction (XRD) with a Cu Kα radiation (Rigaku). The precipitates were analyzed using scanning electron microscopy (SEM, Philips EM400T) equipped with an energy dispersive X-ray (EDX) spectrometer. Vickers hardness was measured with 0.3 kg load and dwell time of 10 s. The mean linear intercept method was used for grain size analysis.

Results and discussion

Microstructure

The microstructure of the as-received AZ31B-alloy had a mean grain diameter of 13 μm (as seen in Fig. 2b) and Vickers hardness 49 approximately.

a) b)

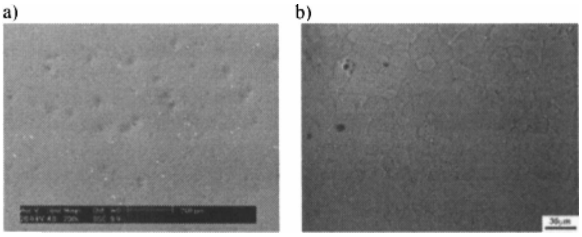
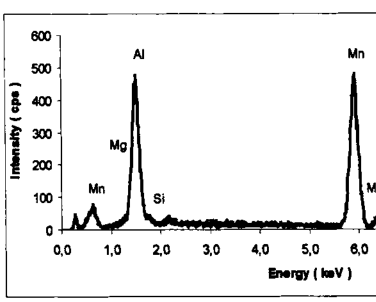


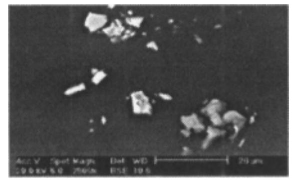
Figure 2. Images (a. MEV; b. OM) the as-received magnesium alloy normal to rolling direction.

The surface of magnesium alloy plate AZ31B as received material presents micro-pores of different sizes and white eutectic precipitates (Figure 2) that according of literature [7]; they are possibly precipitates of $Mg_{17}Al_{12}$, Al_6Mn, Al_4Mn e AlMn.



Elem	Wt%	At%
MgK	2.36	3.88
AlK	31.39	46.47
SiK	2.16	3.08
MnK	61.67	44.84
FeK	2.42	1.73
Total	100.00	100.00

Figure 3. EDX precipitates spectrum at surface as-received material of AZ31B-alloy.



Elem	Wt%	At%
MgK	90.76	93.15
AlK	6.26	5.78
CeL	0.68	0.12
MnK	0.56	0.25
FeK	0.35	0.16
ZnK	1.39	0.53
Total	100.00	100.00

Figure 4. Precipitates image (MEV) and composition of Mg matrix at surface as-received material.

Big precipitates were observed on micrographs (Figure 4.) and Al and Mn were detected by SEM-EDX. After rolling at up 200 °C temperature, eutectic phases were partly dissolved, resulting in shape changes of eutectic precipitates from big to small particles by increasing strain and temperature (Figures 5). The precipitates were remarkably refined to less than 3 μm (Fig. 5b) and redistributed on the α-Mg matrix, as the rolled temperature increased to 250 °C.

a) b)

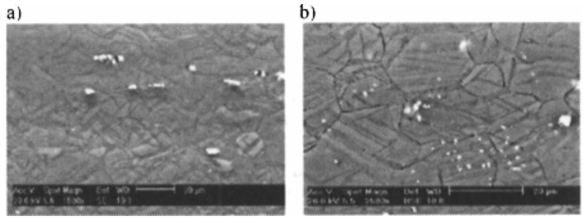


Figure 5. Redistribution of eutectic precipitates of samples rolled at 10 rpm a.) Sample rolled at 25°C and b.) Sample rolled at 250°C

The EDX spectrum from matrix alloy showed that samples rolled at 250 °C contain much more Al and Mn than the as-received material. It confirms that there were lots of eutectic precipitates by dissolution. Otherwise, the atomic percentages of Al and Mn for warm-rolled sample were found to be less than those for cold-rolled one. This fact implies that the precipitates were gradually broken by the large deformation during cold rolling and partly dissolved during the pre-heating and during warm rolling, resulting in redistribution of precipitates.

a) b) c) d)

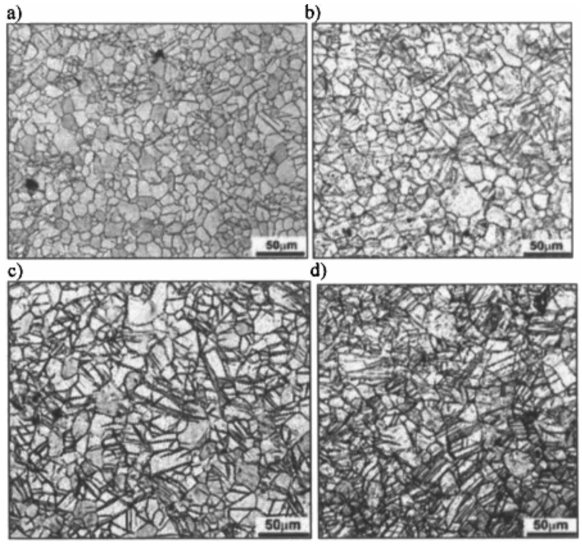


Figure 6. Optical micrographs of single pass rolled AZ31B sheets at 10rpm rolling speed and a) 25 °C, b) 100 °C, c) 200 °C c) 250 °C.

After rolling at 10 rpm, the microstructure of samples was characterized by the mixture of small grains, large amounts of shear bands and twin boundaries (Figure. 6). The grain size of cold rolled sample (25 °C and 10 % reduced thickness) decreased from 13 µm to about 10 µm, inducing inhibition of mechanical twinning. Increasing reduction to 15% and temperatures up to 100 °C, plastic deformation of magnesium produced extensive formation of twinning and dense dislocation pile-ups within grains, especially, on bigger grains where twins were more prominent as was found by Barnett's research. Whereas at temperature up to 200 °C, the microstructures of 15 % and 20% reduced thickness (figure 6c. and 6d.) revealed bimodal distributions of grain sizes with smaller grains about 10 µm and coarser ones about 20 µm. The appearance of these refined grains was mainly induced by recrystallization during the pre-heating and warm rolling. So the static recrystallization may occur during the preheating process for 10 min before the rolling pass.
In order to determine the effect of strain rate, the rolling speed and the reduced thickness were increased to 20 rpm and 30%, respectively. At room temperature (25°C) rolling to 30% reduction in thickness causes cracks, breaking into many small pieces; hence, 10% reduced thickness represents the upper limit of maximum deformation at room temperature.

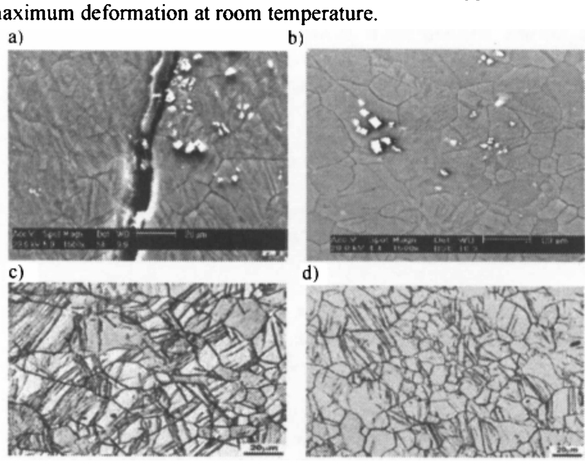


Figure 7. Micrographs of the samples rolled at 20rpm, 30% reduced thickness and a) 25 °C, b) 100 °C, c) 200 °C c) 250 °C.

Comparing samples rolled at 25 °C and 100°C, they had low grain size variations, showing almost a normal distribution of grain sizes about 15 µm (figure 7a and 7b). Increasing temperature up to 200°C, the resultant microstructure became a bimodal mix of finer recrystallized grains (8 µm) and coarser deformed grains (20µm).

Hardness

Table 4. Microhardness values of the as-rolled samples at different temperature and roll speed.

	10 rpm		20 rpm and 30% of reduced thickness
T °C	Reduced thickness (%)	Vickers hardness (kg_f/mm^2)	Vickers hardness (kg_f/mm^2)
25	10	73±1.1	76±1.5
100	15	72±1.8	81±1.2
200	15	73±1.3	84±1.5
250	20	70±1.1	81±1.7

The AZ31B alloy as-received material exhibited a relative low hardness of 49±1.8 HV. After rolling at low roll speed (10 rpm), hardening considerably increased to 73HV, and reached the highest value of 84HV at 200 °C with 20rpm roll speed. It means that AZ31B alloy exhibits a strong hardening behavior by plastic deformation and by age precipitation at temperature up to 100 °C because of redistribution of precipitates. Such as samples with heavy-reduction (30%) at high rolling speed showed 71% increase in hardness (100 and 200 °C). Although at 250 °C, there was a little decrease in hardness due to the nucleation of new strain free grains which removed the strain hardening effects and also likely to be contributed in part from recovery prior to recrystallization.

Normal anisotropy

For small strains and thickness samples, a parameter that can characterize the formability of rolled sheets is the normal anisotropy, which would be determined by invoking the volume (V) constancy, DV/V∓ $\varepsilon_w + \varepsilon_t + \varepsilon_l = 0$, where w, t, and l denote sample width, thickness, and length, respectively, and i and f denote initial and final values [9].

$$r = \frac{\ln(w_f / w_i)}{-\ln(l_f / l_i) - \ln(w_f / w_i)} \qquad (1)$$

Table 3. The planar anisotropy r-value obtained in samples rolled at different rolling speed and temperature.

	10 rpm		20 rpm strain rate: $5.7s^{-1}$
T (°C)	strain rate (s^{-1})	R	R
25	1.5	5.4	-
100	2.3	4.3	5.5
200	2.3	3.9	4.2
250	2.8	3.7	3.4

The magnesium alloy AZ31 showed a significant drop in the normal anisotropy between room temperature and 250 °C (Table 3). This is likely the result of an increase in pyramidal slip activity at higher test temperatures as indicated Koike's researches [4]. This anisotropy provides indirect evidence that there is a change in deformation mechanisms and is attributed to well develop [0002] basal plane, which is a typical texture of warm or cold rolled Mg alloy sheets [10]. Nevertheless, the recovery and dynamic recrystallization processes also decrease r-values at high temperatures on hcp metals, improving the ductility. An increment of roll speed from 10 to 20rpm, improved the strain rate sensitivity from $1.6s^{-1}$ to $5.7s^{-1}$ and induced better formability at temperature up to100°C.

X-ray diffraction

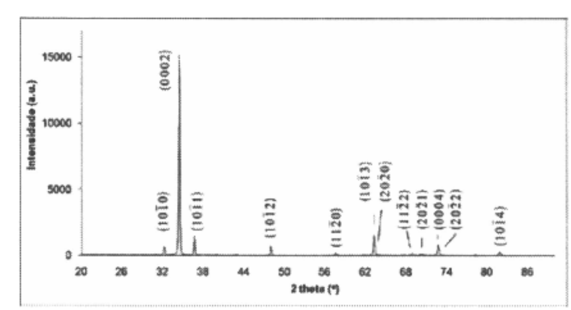


Figure. 8. X-ray diffraction patterns of the as-received AZ31B-alloy.

Figure 8 shows X-ray diffraction patterns of the as-received AZ31B-alloy. All diffraction peaks coincide with those of α-Mg (01-089-4894), suggesting that the AZ31 Mg alloy consists mainly of α-Mg basic phase. The strongest diffraction peak for the AZ31B alloy belongs to (0 0 0 2) and network parameters were calculated as a and c parameters, where a = 0,3198nm; c= 0,5170nm, and c/a = 1,6166.

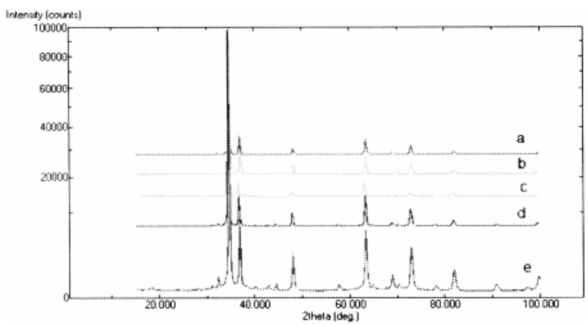


Figure 9. XRD diffraction patterns of AZ31B Mg alloy for rolled samples.

Table 5. Rolling condition and designed name of XRD diffraction patterns.

Temperature °C	Reduced thickness (%)	X-ray diffraction pattern	Rolling speed (rpm)
25	10	d	10
100	15	c	10
200	15	b	10
250	20	a	10
250	30	e	20

The figure 9 shows the X-ray diffraction patterns of rolled samples with different thickness for AZ31 Mg alloys. The varying of intensity showed a transformation of precipitates into magnesium matrix. Comparing X-ray diffraction peaks of the specimens rolled at 10rpm, for 10% and 20% reduction (curve d and a). At high reduction the intensity of peaks decreases resulting on broad peaks diffraction peaks due to mainly temperature increment from 25 to 250 °C. As reported by Peng-Xi Li that a decrease in intensity results on broad peaks.

At 20 rpm rolling speed (curve e), the X-ray diffraction pattern presented broad and sharp peaks due to the precipitation of crystalline phases. The intensity of reflection was increased with increasing speed of the rolling. The microstructures of these alloys showed that the amount of precipitates increases at high rolled temperature as was obtained by X-ray diffraction analyses. The volume fraction of β phase grows during the preheating process before rolling, as shown by the growth of its associated Bragg peaks. The lattice constants change as aluminum exits the matrix to form precipitates, as shown by the Bragg peak's movement to lower 2θ values. However, the X-ray diffraction of warm rolled samples needs more complement data to determine completely the effect of rolling temperature.

Summary

AZ31B Magnesium alloy sheets with about 2 mm in thickness rolled by one-pass with different thickness at cold and warm temperature showed large deformation results with high rolling speed.

The grain refinement was achieved at cold rolling with small reduction in thickness (10%) and low strain rate, twinning was predominantly formed in coarse grains, not in fine grains and with; whereas at temperature up 200°C fine grains were formed by discontinuous recrystallization during the preheating and warm-rolling process. The high speed rolling is very effective for grain refinement.

The AZ31B alloy had a large hardening response on cold and warm rolling temperature resulting of plastic deformation, and precipitation of a transition phase β-($Mg_{17}Al_{12}$), consequently offering a combination of good strength and ductility.

AZ31 alloy was found to be sensitive to strain rate in the high temperature, besides the increase in the flow stress, appears as a decrease in material formability. The anisotropy was adversely impacted in cold rolling sheets and was characterized by high anisotropy at room temperature. Thus, the formability of AZ31B alloy increases with temperature when plastic deformation is performed at low rolling speed, while an increasing of rolling speed leads a decrease in formability at high temperature.

Acknowledgements

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References

[1] B. L. Mordike, T. Ebert, "Magnesium properties-applications-potential," *Materials Science and Engineering A302*, (2001), 37–45.

[2] M. R. Barnett, Z. Keshavarz, A.G. Beer, D. Atwel, "Influence of grain size on the compressive deformation of wrought Mg-3Al-1Zn," *Acta Materialia*, 52, (2004), 5093- 5103.

[3] S. E. Ion, F. J. Humphreys, S. H. White, "Dynamic recrystallization and the development of microstructure during the high temperature deformation of magnesium," *Acta Metallurgica, 30*, (1982), 1909-1919.

[4] J. Koike, T. Kobayashi, T. Mukai, H. Watanabe, M. Suzuki, K. Maruyama, K. Higashi, "The activity of non-basal slip systems and dynamic recovery at room temperature in fine-grained AZ31B magnesium alloys," *Acta Materialia 51*, (2003), 2055–2065.

[5] D. Letzig, J. Swiostek, J. Bohlen, P. A. Beaven and K. U. Kainer, "Wrought magnesium alloys for structural applications," *Materials Science and Technology, Vol. 24*, (2008), 991-996.

[6] Zhao Hong-liang, Guan Shao-kang, Zheng Fei-yan, Li Qing-kui, Wang Li-guo, "Microstructure and properties of AZ31 magnesium alloy with rapid solidification," *Trans. Nonferrous Met. Soc. China 15*, (2005), 144–148.

[7] Soon Chan Park, Jong Dae Lim, Dan Eliezer, Kwang Seon Shin, "Microstructure and mechanical properties of Mg–Al–Ag Alloys," *Materials Science Forum Vols. 419-422*, (2003), 159-164.

[8] Yasumasa Chino, Kensuke Sassa, Akira Kamiya, Mamoru Mabuchi, "Enhanced formability at elevated temperature of a cross-rolled magnesium alloy sheet," *Materials Science and Engineering A 441*, (2006), 349–356.

[9] S.R. Agnew and O. Duygulu, "Plastic anisotropy and the role of non-basal slip in magnesium alloy AZ31B," *International Journal of Plasticity 21*, (2005), 1161-1193.

[10] Matthew R. Barnett, Alireza Ghaderi, Iichat Sabirov, Bevis Hutchinson, "Role of grain boundary sliding in the anisotropy of magnesium alloys," *Scripta Materialia 61*, (2009), 277–280.

[11] Peng-Xi Li, Bo-Ping Zhang, Jing-Yi Chen, Yi-Fan Tu, "Microstructure and Mechanical Properties of AZ31B Magnesium Alloy Sheets Prepared by Extruding and Rolling," *Advanced Materials Research Vols. 11-12*, (2006), 653-656.