



Full length article

Effect of asymmetric rolling process on the microstructure, mechanical properties and texture of AZ31 magnesium alloys sheets produced by twin roll casting technique

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Abstract

Symmetric rolling (SR) and asymmetric rolling (ASR) processes were carried out on 6 mm thick AZ31 magnesium alloy sheets that were produced by twin roll casting (TRC) technique. Before rolling processes, sheets were heat treated in order to obtain a homogenized microstructure. In this study, for the ASR process the rolling speed ratio between upper roller and lower was selected as 1.25. Both SR and ASR processes were utilized with 40% reduction per passes using 2 pass schedule for a total reduction ratio of 0.67. Symmetric and asymmetric rolled sheets were characterized using optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. Texture measurements were performed by using X-ray diffraction (XRD) technique and mechanical properties were investigated by tensile tests and also hardness measurements.

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Keywords: Asymmetric rolling (ASR); AZ31 magnesium alloy; Texture; Twin roll casting

1. Introduction

Magnesium alloys have potential as structural materials due to their excellent properties such as low densities, high specific strength, high specific stiffness, good castability and machinability, low heat content per unit of volume, high damping

capacity and good electromagnetic (EMI) shielding. They are therefore very attractive in applications in electronic, transportation (automotive, railway, aerospace and space) and defense industries where the mass reduction is an important issue [1–4]. The use of Mg as a sheet is very limited. The most common magnesium alloy sheet (AZ31 Mg alloy) is produced by rolling of DC (Direct Chill) cast ingots. Conventional magnesium sheet production has a lot of steps such as melting, ingot, homogenization, milling, heating, hot rolling (multi-heating), warm rolling (multi-heating), cold rolling and finish annealing. This multi-step process is not cost-effective in terms of time and energy. However, the demand for decreasing the magnesium sheet prices is high and can be met through twin roll casting [5,6]. The TRC is the most economical process, with investment costs that are 1/3 to 1/4 of the conventional Mg sheet production process [7–9].

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In the last 10–20 years, a lot of companies, institutes and universities produced magnesium alloy sheets as laboratory scale using TRC technology [8,10–25]. Moreover, M. Masoumiet et al. [26,27], D. Choo et al. [28] and B. Engl [29] et al. investigated the effect of thermomechanical processing on the microstructure and texture of TRC-AZ31 magnesium alloy sheets. They observed that the TRC structure is responsible for the texture alteration upon annealing. Magnesium alloys have poor formability and limited ductility at room temperature because of hexagonal close-packed (HCP) crystal structure and low stacking fault energy [30]. Mg alloys only have three independent active slip systems at basal plane. Whereas, according to Taylor criterion at least five independent slip systems are required to achieve homogeneous ductility [31]. The microstructure and mechanical properties of Mg alloys are highly dependent on their severe plastic deformation processes such as, equal channel angular extrusion (ECAE), equal channel angular pressing (ECAP), accumulative roll-bonding (ARB), asymmetric rolling (ASR), cross rolling, high pressure torsion (HPT), etc. [32]. Above all, asymmetric rolling is expected to improve the grain refinement, mechanical properties and formability [33–38]. Press formability is strongly affected by the texture and it can be improved by ASR as a consequence of reduction of basal texture intensity. It is important to improve the formability at low temperatures for a wide use of the wrought Mg alloy sheets by changing or weakening the basal texture [39,40]. In asymmetric rolling (ASR) process, the circumferential velocities of the upper and lower rolls are different so shear deformation can be applied throughout the thickness of the sheets [41–44]. The changes in deformation mode from thickness reduction to shear deformation can change the texture from {0002} texture to other components, and redundant shear deformation can reduce the grain size effectively [45].

In the present work, effect of ASR process on microstructure and texture of an industrial scale AZ31 magnesium alloy sheet produced by twin roll casting technique was investigated. The result of asymmetric rolling process on microstructure and texture was compared with the results of symmetric rolling process. The TRC magnesium alloy sheets used in this study were produced on an industrial scale and 1500 mm wide magnesium alloy AZ31 sheets of 6 mm thickness have been successfully cast [46–50].

2. Experimental

The twin roll casting (TRC) system used had a gas-fired chamber furnace with a maximum melting capacity of approximately 3200 kg magnesium. Water-cooled steel twin-

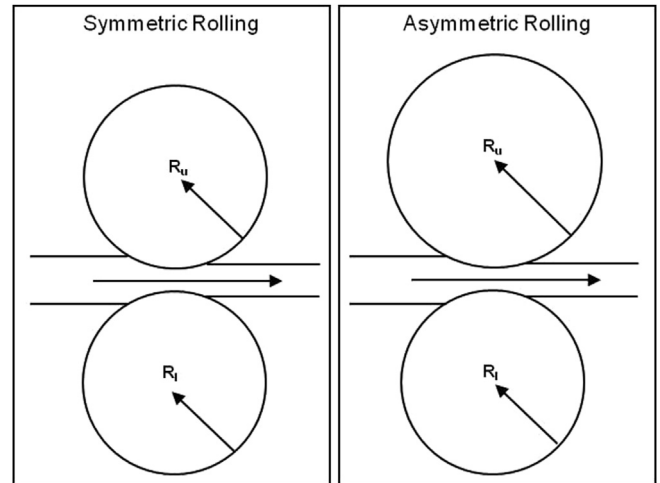


Fig. 1. Schematic pictures of SR and ASR processes.

rolls of 1600 mm wide and 1125 mm diameter with 15° tilt angle were used. The system has a pinch roll unit, a cross-cut shear and a coiler. 1500 mm wide magnesium alloy AZ31 sheets of 6 mm thickness have been successfully cast [46–50]. The alloy composition is given in Table 1. The composition well fits to the ASTM and ISO standards [1]. In the present work, the TRC AZ31 magnesium alloy sheets were used for symmetric rolling and asymmetric rolling processes.

Before rolling processes, sheets were heat treated. Homogenization conditions were set as follows: sheets were heated to 425 °C for 6 h and then cooled to room temperature in air. After homogenization, sheets have been symmetrically and asymmetrically rolled using a laboratory scale rolling mill. Schematic views of symmetric and asymmetric rolling processes were given in Fig. 1; and Table 2 gives the rolling conditions for symmetric rolling (SR) and asymmetric rolling (ASR). Before rolling processes, the sheets were firstly heat treated for 30 min at 350 °C. For the ASR process the rolling speed ratio between upper and lower rollers was selected as 1.25. Both SR and ASR processes with 40% reduction per passes using 2 pass schedule for a total reduction ratio of 0.67 were utilized and the rolling processes were performed at 300 °C. A thermocouple is used to measure the temperature.

The microstructure examination of the as-cast, homogenized and as-rolled sheets was performed using optical microscopy (OM). Rolled sheets were also compared by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

For optical microscopy, the samples were cold mounted in epoxy and polished starting with SiC paper, followed by 3 and 1 μ diamond slurries and colloidal silica. The specimens were etched in an acetic picric acid solution (4.2 g picric acid, 10 ml

Table 1
The chemical composition of TRC magnesium alloy sheet obtained by optical emission spectrometer (mass content, %).

Sample	Al	Zn	Mn	Fe	Ni	Mg
AZ31	2.7	1.03	0.31	0.003	0.0002	Bal.

Table 2
Rolling conditions of asymmetric and symmetric rolling processes.

	R_u (mm)	R_l (mm)	V_u (m/min)	V_l (m/min)	V_u/V_l
ASR	350	300	19.75	15.80	1.25
SR	350	300	15.80	15.80	1

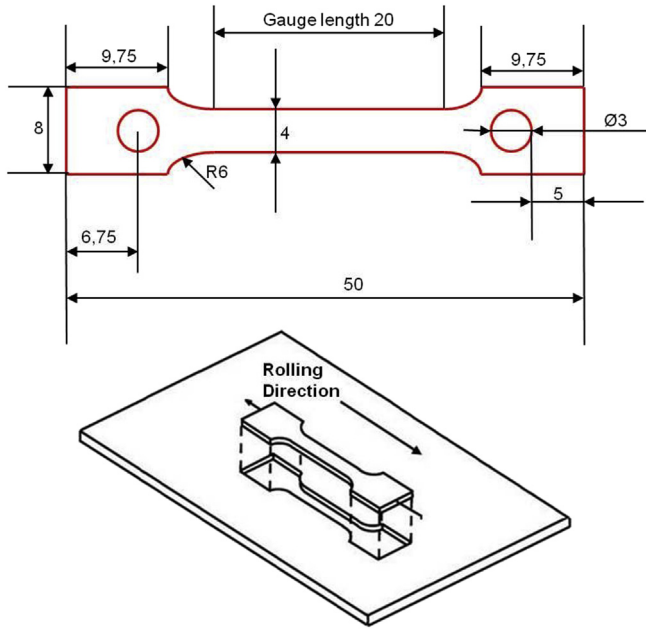


Fig. 2. Tension test specimen (dimensions are in mm).

acetic acid, 70 ml ethanol, 10 ml water) for 5 s and they were washed with and rinsed in ethanol. Specimens were investigated with a Nikon L150 light microscope in plan view and cross sectional views. According to ASTM E112-96 standard, the Heyn Lineal Intercept procedure was applied for measuring the size of the grains [51]. In this method, a standard line was drawn and the number of grains intercepted by this line was counted. Upon obtaining at least 50 intercepts, the total line length was divided by the number of intercepts in order to obtain the lineal intercept grain size, d .

Scanning electron microscopy (SEM) observations were carried out using a JEOL JSM-6335-F FEG. For transmission electron microscopy (TEM) studies, specimens were cut, 3 mm diameter discs were punched and ground to a thickness less than 60 μm . Punched specimens were dimple ground with Gatan 656 Dimple Grinder. Specimens were ion polished first at 3 kV and after perforation at 1 kV with Gatan 691 Precision Ion Polishing System (PIPS). The foils were examined with a JEOL JEM 2100 HRTEM with a LaB₆ filament operated at 200 kV.

Texture measurements were performed by PANalytical X'Pert Pro operated at 45 kV and 40 mA after specimens were polished to third quarters of sheet thickness. Texture pole figures were determined by normalization with X'Pert Texture Version 1.1a software.

To investigate the mechanical properties of the materials, a Zwick Z250 universal testing device was used. For homogenized condition samples were machined with tensile axis parallel to the rolling direction (RD) with a gage section of 50 mm length and 12.5 mm width. After ASR and SR processes the specimens with 20 mm gage, 50 mm length and 4 mm width were cut along planes coinciding with the rolling direction (Fig. 2). Vickers microhardness measurements were performed with a 200 g applied load with a Zwick ZHV10 device and the average hardness value was calculated from at least five test readings.

3. Results and discussions

In this study, Fig. 3 shows plan view microstructures of as-cast condition, after homogenization heat treatment, symmetrically rolled and asymmetrically rolled AZ31 magnesium alloy sheets.

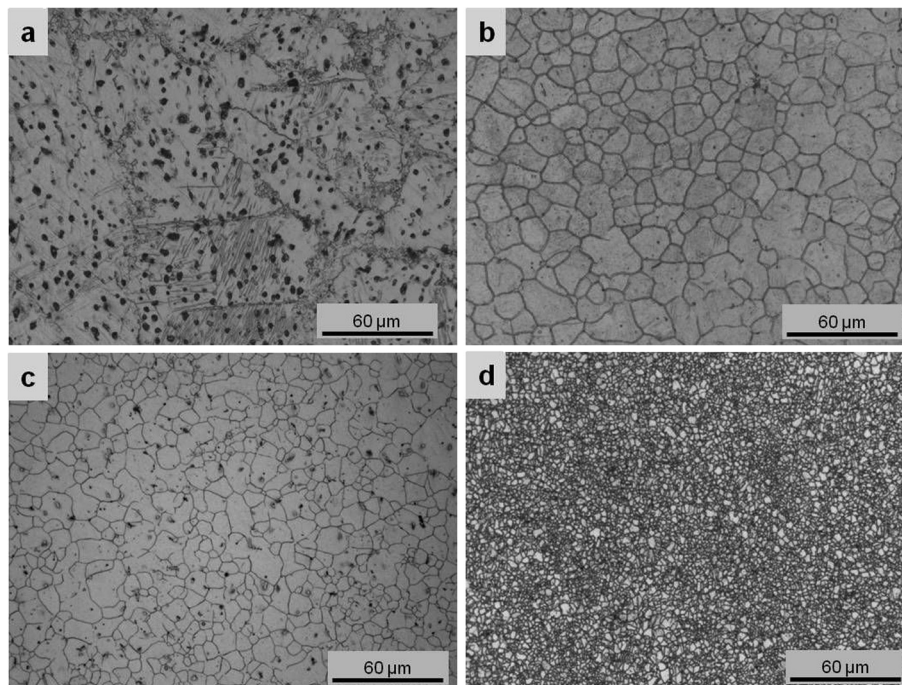


Fig. 3. Plan view microstructures of (a) as-cast and (b) after homogenization at 425 °C for 6 h, (c) symmetric rolled and (d) asymmetric rolled AZ31 magnesium alloy sheets.

which is in between die-casting structure and semi-solid casting structure was observed. Although dendritic boundaries were not very clear, the dendritic grain size was measured as approximately $200\ \mu\text{m}$ and secondary dendrite arm spacing (SDAS) was measured as around $15\ \mu\text{m}$. To obtain a homogenized microstructure, sheets were heat treated at $425\ ^\circ\text{C}$ for 6 h before asymmetric and symmetric rolling processes.

Recently, many studies about ASR process have been carried out. Watanabe et al. [38] used asymmetric rolling, in which the ratio of the circumferential velocities of the upper

and lower rolls was 1.25 with the same roll diameter, to produce the AZ31 sheets with grain sizes of $11\text{--}12\ \mu\text{m}$. Some improvement in tensile elongation from 9% to 15% was achieved. S.H. Kim et al. [52] showed that asymmetric rolling, in which the ratio of the upper roll diameter to the lower roll diameter was 1.5 with the same revolutions per minute, reduced the intensity of the basal texture. Also Lee et al. [53] tried to investigate the ASR process effects on microstructure, texture and formability of AZ31 Mg sheet. As a result of the study they observed that the average grain sizes were about $38\ \mu\text{m}$ for asymmetrically rolled sample and $50\ \mu\text{m}$ for

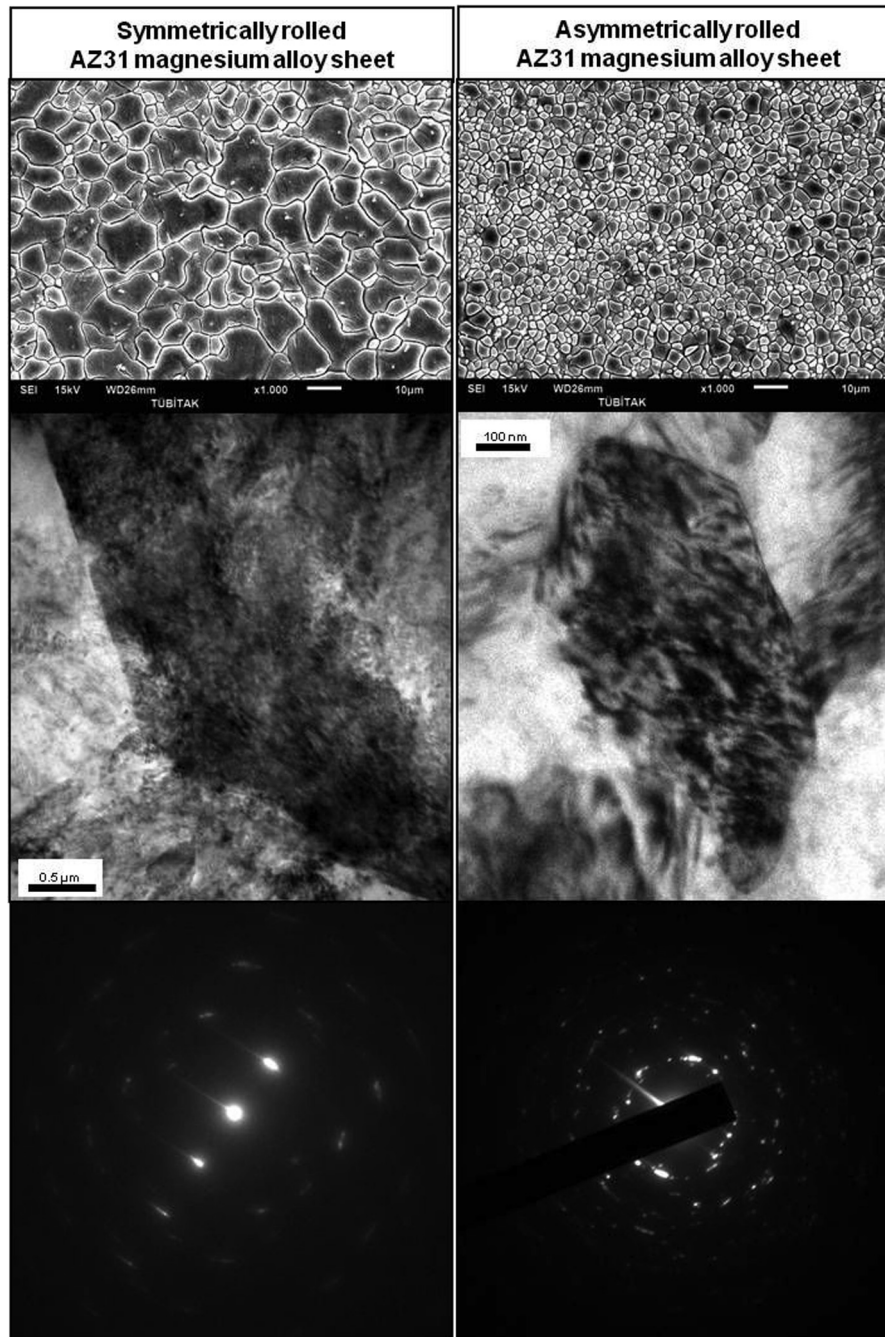


Fig. 4. SEM pictures, TEM pictures and selected area electron diffraction (SAED) patterns of symmetrically and asymmetrically rolled AZ31 magnesium alloy sheets.

symmetrically rolled sample. In their study, to evaluate the formability of both AZ31 Mg alloy sheet, the Erichsen test was used. The Erichsen value was measured to be 8.1 for asymmetrically rolled sample and 5.6 for symmetrically rolled sample. This means that asymmetric rolling process predominant method to improve its plastic anisotropy tendency. W.J. Kim et al. [54] reported that by applying a high ratio of the circumferential velocities between the upper and lower rolls in asymmetrical rolling, a very fine grain size of 1.4 μm could be achieved after a single pass of 70% thickness reduction. Basal fiber texture was notably weakened during the rolling. The rolled sheets exhibited high yield stresses over 300 MPa and a maximum elongation of 35%.

Chang et al. [55,56] investigated about ASR processing of a laboratory scale magnesium alloy sheets produced by TRC technique. They used AM31 magnesium alloy. The chemical compositions of the strip AM31 alloy was Mg–2.7% Al–0.97%Mn (wt.%), respectively. After asymmetric rolling process, in which the ratio of the upper roll diameter to the lower roll diameter was 1.2 and 1.5, the elongation of the TRC–AM31 alloys was improved to be 17–19%. After investigation the pole figures they reported that maximum pole intensity of the ASR (R_s : 1.2–1.5) processed sheets was 10.15 and 13.61, the intensity of the SR processed sheet was 14.76. The mean grain size of the annealed SR-processed sheet was 7.3 μm , while the ASR ($R_s = 1.2, 1.5$) processed sheets showed grain size of 6.9 μm and 4.9 μm , respectively. After the study, they reported that texture characteristics had great influence on the evolution of microstructure and texture during the subsequent rolling and annealing process. Strength and ductility was improved by TRC combined with ASR due to grain refining, inclination of basal poles and weakened texture.

In the present study, after homogenization, the eutectic phases dissolve, equiaxed grain structure was formed and the grain size was refined to 19 μm . For symmetrically rolled sample, the average grain size was achieved as 10 μm and for

asymmetrically rolled sample it was observed as 0.7 μm . SEM and TEM pictures of asymmetrically and symmetrically rolled sheets were given in Fig. 4. Selected area electron diffraction (SAED) patterns of symmetrically and asymmetrically rolled AZ31 magnesium alloy sheets were also presented. SAED pattern of ASR sheet indicates a spreading of the diffracted beams relative to the SAED pattern of the SR sheet which is evidence of grain refinement and larger boundary misorientations by ASR.

(0002) and (1010) pole figures of the commercially available AZ31 1 mm and 2 mm sheets, asymmetrically and symmetrically rolled AZ31 magnesium alloys sheets were given in Fig. 5. Symmetric rolling generally gives rise to a strong basal texture, which exhibits low press formability at near room temperature because the basal slip systems hardly become active. This induces the high normal anisotropy in sheet and increases the difficulty in deformation accompanied with thickness reduction, and consequently leads to a very limited formability near room temperature [57]. Therefore, it is important to change or weaken the basal texture for enhancing the formability. ASR is a processing carried out at different rotation speeds for upper and lower rolls so that intense shear deformation can be introduced throughout the sheet thickness, which can result in grain refinement and change in texture [58].

It is known that generally, the symmetrically rolled sample tends to show peak inclined to the rolling direction but the asymmetrically rolled sample tends to show peak slightly inclined to the transverse direction because large shear strain is introduced during asymmetric process, comparing to symmetric process [53]. In this study, when the pole figures of both asymmetrically and symmetrically rolled AZ31 magnesium sheets investigated, it was found that this effect is less pronounced compared to the literature. They showed peak inclined to the transverse direction and this result can be explained that the initial plate used in both rolling processes is

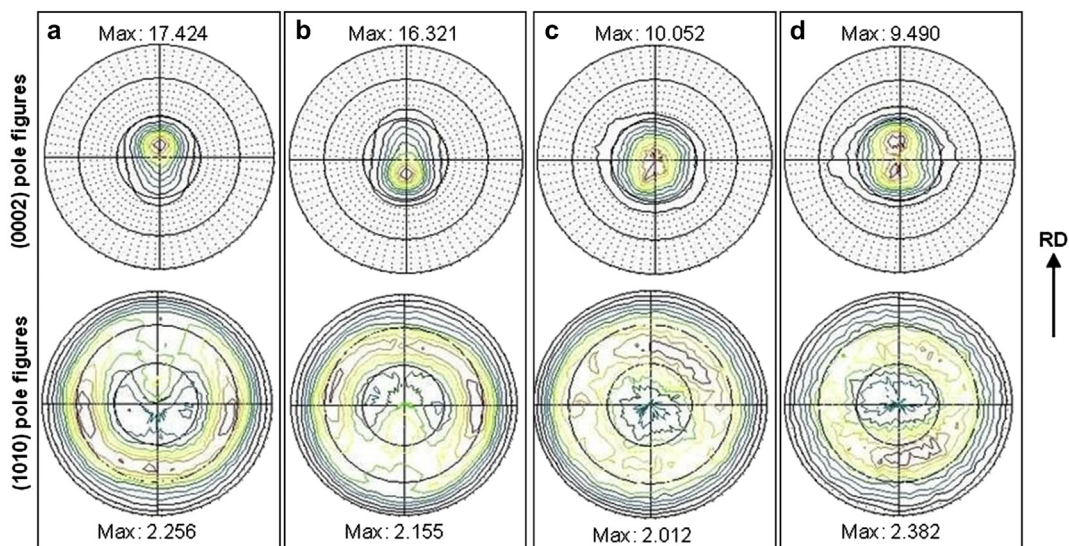


Fig. 5. (0002) and (1010) pole figures of the (a) commercially available AZ31 1 mm sheet, (b) commercially available AZ31 2 mm sheet, (c) symmetrically rolled AZ31 2 mm sheet and (d) asymmetrically rolled AZ31 2 mm sheet.

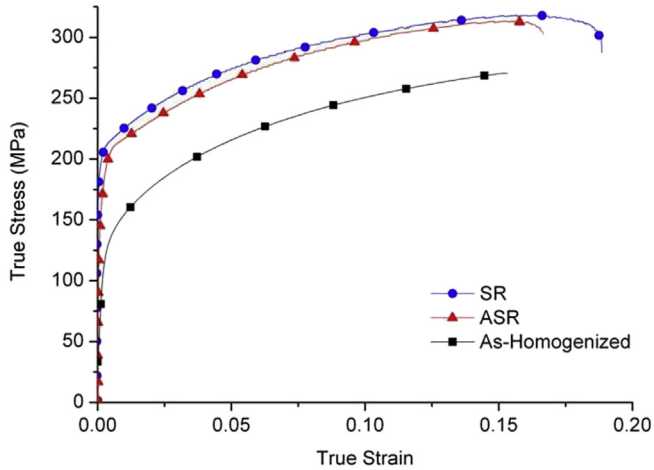


Fig. 6. Tensile test curves for after homogenization at 425 °C for 6 h, asymmetrically and symmetrically rolled AZ31 magnesium alloy sheets.

manufactured by the TRC technique. However, as expected, the texture intensities were decreased after asymmetric rolling process.

The reduction of (0002) texture intensity and grain size after ASR is also expected to lower yield stress and higher elongation values [52,53]. However, in this study, it was determined that the mechanical test results of asymmetrically and symmetrically rolled sheets were almost the same. AZ31 homogenized sheet, SR and ASR processed sheets detailed tensile test curves and corresponding results were given in Fig. 6 and Table 3. Despite the grain size improvement, lack of change in mechanical properties can be explained that the sheets used for rolling process produced by twin roll strip casting technology as well. Moreover, it should be noted that the samples used in this study were industrial scale sheets and the center line segregation and surface segregation may affect the mechanical properties. The reason also can be related with the long range inhomogeneous microstructure of the twin roll casted AZ31 magnesium alloy sheet that was used for the rolling processes.

Table 4 shows micro Vickers hardness measurement results from plan view surfaces of asymmetrically and symmetrically rolled and also homogenized Mg alloy sheets. Asymmetrically rolled Mg AZ31 alloy has the highest hardness, with a

Table 3
Tensile test results for after homogenization at 425 °C for 6 h, asymmetrically and symmetrically rolled AZ31 magnesium alloy sheets.

Material	YS (MPa)	UTS (MPa)	EL (%)	ϵ_{UTS}
After homogenization at 425 °C for 6 h	122	231	17.3	0.153
Asymmetrically rolled AZ31 magnesium alloy sheet	188	272	21	0.153
Asymmetrically rolled and homogenized AZ31 magnesium alloy sheet	171	265	20	0.157
Symmetrically rolled AZ31 magnesium alloy sheet	203	275	23	0.163
Symmetrically rolled and homogenized AZ31 magnesium alloy sheet	188	267	21	0.163

Table 4
Hardness measurements results of as asymmetrically and symmetrically rolled and homogenized magnesium AZ31 alloy sheets (HV–200 g).

Material	Vickers hardness (HV)
After homogenization at 425 °C for 6 h	56
Asymmetrically rolled AZ31 magnesium alloy sheet	78
Asymmetrically rolled and homogenized AZ31 magnesium alloy sheet	69
Symmetrically rolled AZ31 magnesium alloy sheet	62
Symmetrically rolled and homogenized AZ31 magnesium alloy sheet	59

hardness value of 78HV. Whereas, the hardness value of symmetrical rolled sheet is lower.

Also, the tensile test results and hardness measurements of the homogenized sheets after rolling processes were given in Tables 3 and 4. As shown in Table 3, there are no significant differences for tensile test results after homogenization treatment of ASR and SR processed sheets. However, when the homogenization was applied, it was seen that all the hardness values dropped.

4. Conclusion

TRC–AZ31 alloys were heat treated to 425 °C for 6 h and then cooled to room temperature in air. After homogenization, sheets have been symmetrically and asymmetrically rolled at a warm temperature of 300 °C. Before rolling processes, the sheets were firstly heat treated for 30 min at 350 °C. For the ASR process the rolling speed ratio between upper and lower rollers was selected as 1.25. Microstructures, textures and mechanical properties of the rolled AZ31 sheets were examined. The results are summarized as follows:

- (1) After microstructural investigation, the mean grain size of the homogenized TRC–AZ31 alloy sheet was calculated as 19 μm , while, for symmetrically rolled sample, the average grain size was observed as 10 μm and for asymmetrically rolled sample it was achieved as 0.7 μm .
- (2) When the pole figures were examined, as expected, it was found that the basal texture intensity of both asymmetrically and symmetrically rolled sheets were reduced compared to conventional rolling. According to texture studies, the maximum pole intensity values were 17.424, 16.231 and 10.052 for commercially available AZ31 1 mm sheet, commercially available AZ31 2 mm sheet and SR processed sheet respectively. However, minimum value of 9.490 could be achieved for the ASR processed TRC–AZ31 alloy sheet.
- (3) The reduction of (0002) texture intensity and grain size after ASR is also expected to lower yield stress and higher elongation values. However, in this study it was determined that the mechanical test results of asymmetrically and symmetrically rolled sheets were almost the same. Despite the grain size improvement, lack of change in mechanical properties can be explained that the sheets

used for rolling process produced by twin roll strip casting technology as well. Moreover, it should be noted that the samples used in this study were industrial scale sheets and the center line segregation and surface segregation may affect the mechanical properties. The reason also can be related with the long range inhomogeneous microstructure of the twin roll casted AZ31 magnesium alloy sheet that was used for the rolling processes.

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