Evolutions of Mechanical Properties and Macro Textures of AZ91D sheets by Hot Rolling and Subsequent Annealing

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Abstract. The mechanical properties and textures of 75% hot unidirectionally rolled and cross rolled commercial magnesium die-casting alloy AZ91D sheets were investigated for the different rolling temperatures 623K and 673K, respectively. As for mechanical properties, tensile testing results show total elongations of both as-unidirectional and as-cross-rolled sheets are enhanced by the repetition of rolling passes and inter-pass heating. The planar anisotropy of tensile strengths and elongations of cross-rolled at 673K are the smallest among the rolling conditions employed in the present study. Annealed sheets demonstrate the significant elongation to failure due to the static recrystallisation at 623K. Macro textures of the as-unidirectional-rolled sheets show typical double-peak (0001) basal textures with inclined peaks towards ±15˚ in RD. Contrastingly, macro textures of the as-cross-rolled sheets show the significant (0001) basal textures having double peaks at ±10˚ in TD and concentrically broader orientation distribution in RD and TD.

1. Introduction

Magnesium alloys have very attractive features such as a promising potential of light-weight structural metallic material and high-specific strength to be employed for bodies of motor vehicles, high speed rail rolling stocks and aeroplanes in order to reduce emission gas and improve fuel efficiency [1,2]. Magnesium alloys are mostly used as die-cast products in general and the prevalence of wrought magnesium alloy products, especially rolled sheets, are still limited. This is mainly because crystal structure of magnesium is hexagonal close-packed structure which hinders plastic deformation at room temperature in which the basal slip systems only activate. For improvement of cold workability such as sheet metal forming, texture control to weaken (0001) basal texture of magnesium alloys is highly focused for many years [3-5]. Rare earth element added magnesium alloys tend to avoidably have lower tensile strength in exchange for splendid formability at room temperature [5]. High aluminium content (~9%) magnesium alloy AZ91 has higher tensile strength and good corrosion resistance. However, commercial wrought AZ91 materials are seldom manufactured until rapid solidification continuous roll caster designed for thin slab and forging materials is introduced [6]. Only a few attentions on texture changes of rolled AZ91 sheet have been paid even though the other magnesium alloys are evaluated.
It would be preferable that the thin wide AZ91 rolled sheet with weak or double-peak type (0001) basal textures are fabricated using texture control via strain path change in thermo-mechanical process. In this study, the authors investigated the effectiveness and feasibility of strain path change on evolutions of macro textures and anisotropy of mechanical properties of rolled AZ91D sheets.

2. Experimental procedures
A commercial magnesium alloy AZ91D ingot was used in this study. Its chemical composition was listed in the Table 1. The 4mm thick sheets were sliced from an ingot, subsequently being machined into 30mm wide and 60mm long sheets. The sliced sheets were subject to heat treatment in the electric furnace at 703K for 1 hour, followed by water quenching. The quenched AZ91D sheets were rolled at the two different elevated temperatures, 623K and 673K including 30-minutes pre-heating and inter-pass heat treatment for 5 minutes. The total pass numbers of hot rolling were 9 passes and total reduction in thickness was set to 75%. A φ 50 mm 2-high laboratory rolling mill was used without lubrication. Two different rolling patterns explained hereinafter were employed as seen in Figure 1. Unidirectional rolling pattern (A in the figure) was defined as a conventional flat rolling in which a sheet was rolled along the longitudinal direction. Cross rolling pattern (B in the figure) was defined as a combination of unidirectional rolling and transverse rolling. In cross rolling pattern, unidirectional rolling was conducted up to the fourth pass, and from the fifth pass to the final pass, transverse rolling and unidirectional rolling were employed at the odd number and the even number of rolling pass, respectively. The hot-rolled sheets were annealed at 623K for 30 minutes in the electric furnace.

Table 1. Chemical composition of the used AZ91D magnesium alloy (mass%).

| Element | Mg   | Al  | Zn  | Mn  | Si  | Cu  | Fe  | Ni  |
|---------|------|-----|-----|-----|-----|-----|-----|-----|
| bal.    |      | 8.99| 0.63| 0.25| 0.04| 0.01| 0.001| 0.001|

Figure 1. Schematic illustration of rolling patterns used in this study.

Tensile test and Vickers hardness test (results not shown in this paper) were performed in order to evaluate mechanical properties and planar anisotropy of as hot-rolled and 623K-annealed sheets. Dimensions and shape of tensile test pieces were conformed to the 1/5 scale of no.5 specimen JIS Z 2201. Tensile test pieces were spark-machined from the sheets, varying the angles 0˚, 45˚ and 90˚ to the initial rolling direction (RD). The initial strain rate of tensile testing was $\dot{\varepsilon} = 8.33 \times 10^{-4}$ s$^{-1}$. Optical micrographic observation was conducted on TD planes of the as-rolled and annealed sheets. Macro texture measurement of mid-thickness planes of the as-rolled and annealed sheets was performed. For macro texture measurement, pole figure data were measured by the pseudo Schulz reflection method using Rigaku SmartLab multipurpose X-ray diffractometer system equipped with Cu-K$\alpha$ radiation.
Incomplete (0001) pole figures were plotted from collected pole figure data using Mtex toolbox [9] in MATLAB 2019b software.

3. Results

3.1. Microstructures

The optical microstructures observed from the transverse direction (TD) section of the as-hot rolled sheets at 623K and 673K are shown in Figure 2. In the figure, there exist some etched pits at which second phase particles have presumably dropped off during immersion of chemical etching. Mechanical twins, subgrains and elongated recrystallized grains are observed in the 623K unidirectional rolled sheet (a). On the other hand, recrystallized grains and hot-band-like subgrain clusters are found in the 673K straight rolled sheet (b). In the cross rolled sheets of 623K (c) and 673K (d), mechanical twins do not appear in the observed area, and microstructures are covered with partially recrystallized grains.

Figure 3 shows optical microstructures of the sheets annealed at 623K. Microstructures in both annealed 623K- and 673K-unidirectional rolled sheets are covered by fully recrystallized grain and coarsened grains as seen in (a) and (b). Microstructures in annealed 623K- and 673K-cross rolled sheets are also covered by fully recrystallized grains, otherwise there are fewer coarsened grains found in the recrystallisation microstructures. The mean grain sizes measured by intercept counting method along the rolling direction are also shown in Figure 3. There is a tendency that aspect ratios of grain length over grain thickness of cross rolled sheets seem to be smaller than those of unidirectionally rolled sheets.

3.2. Mechanical properties

Nominal stress-nominal strain curves of the as-hot unidirectional (623K (a) and 673K (b)) and the as-hot cross rolled sheets (623K (c) and 673K (d)) are shown in Figure 4. According to the specifications of mechanical properties of as-cast commercial die-casting magnesium alloy AZ91, elongation to failure, 0.2% proof stress and ultimate tensile strength of are around 3%, 160MPa and 230MPa, respectively [10]. Ductility of both the as-unidirectional and the as-cross rolled sheets processed at 623K is enhanced by hot rolling process, showing passable elongation ranging from 4% to 15% at the maximum. Elongations along TD (90°) of both as-unidirectional and cross rolled sheets processed at 623K are distinctly less than 4% due to the early fracture during tensile test. Early fracture also occurs in RD (0°) of both as-hot rolled sheets, which results in 8% total elongation. Larger total elongations around 15% is obtained in the diagonal direction (45°) of both as-hot rolled sheets. Ultimate tensile strengths of the
diagonal direction specimens are 350MPa for 623K-unidirectional rolling and 380MPa for 623K-cross rolling, respectively. At higher rolling temperature, 673K, as-hot-unidirectional rolled sheet shows comparatively uniform and larger elongation to failure, approximately 18%, especially in TD (90°). In contrast, as-cross rolled sheet processed at 673K shows there is a little difference among the three directions, RD (0°), diagonal direction (45°) and TD (90°) in nominal stress-nominal strain curves. It is attributed to that the effect of change in strain path and higher rolling temperature induce to restrain or minimize the planar anisotropy of rolled sheets. Nevertheless, total elongations are limited within a range of around 9–10%.

Figure 4. Nominal stress-nominal strain curves of as-hot-rolled AZ91D sheets.

Figure 5. Nominal stress-nominal strain curves of hot rolled AZ91D sheets annealed at 623K.

Figure 5 demonstrates nominal stress-nominal strain curves of the unidirectional rolled and the cross rolled sheets annealed at 623K. As a consequence of recrystallisation annealing, total elongation under all the rolling conditions are well enhanced except for TD (90°) of 623K-unidirectional rolling, 623K- and 673K-cross rolling. Ultimate tensile strength varies from 300MPa to 325MPa in the specimens exhibiting considerably significant elongations. As for 673K-cross rolling, despite recrystallisation annealing, ductility of the annealed sheet does not develop well as compared with the other three rolling conditions, resulting in the total elongation range from 8% to 19%.

Figure 6 shows the variations of r-values (Lankford values or plastic strain ratio) of the annealed sheets. The r-values were calculated from width strain and thickness strain after 4% extension applied in tensile test in order to avoid a fracture. Except for 673K-unidirectional rolling, distributions of r-values show the V-shape or inverse V-shape. Mean r-values varying from 0.80 to 1.79 are generally lower as compared to AZ31. This is because an applied
longitudinal strain (4% elongation) during tensile test is too smaller than 15~20% elongation in the other metallic materials such as steels, aluminum alloys and so on. Regarding to the index of planar anisotropy, 623K-unidirectional rolling is the lowest among the employed rolling conditions, although mean r-value of 623K-unidirectional rolling is also markedly the lowest among these rolling conditions. Accompanied with an increase in ductility, improvement of r-value may arise by applying higher annealing temperature, which may adversely lead to grain coarsening due to the secondary recrystallisation.

3.3. Macro textures

Figure 7 shows the incomplete (0001) pole figures of heat-treated cast starting material and as-hot-rolled AZ91D sheets. Orientation/pole densities in pole figures were normalised with each own X-ray diffraction raw data. Heat-treated starting material does not have an obvious (0001) basal texture because of cast ingot, and the maximum relative intensity lies in nearby the (0112) pole in the pole figure. The pole figure of 623K-unidirectional rolled sheet shows the typical double-peak type (0001) basal texture with the peaks inclined towards ±15° in RD as seen in Figure 7 (b). 673K-unidirectional rolled sheet also shows substantially similar double-peak type (0001) basal texture (Figure 7 (c)). The double-peak type (0001) basal texture is formed in consequence of activation of pyramidal \(<c+a>\) slip system [11]. Contrastingly, macro texture of 623K-cross rolled sheet shows a significant (0001) basal texture with the peaks inclined towards ±10° in TD. Contours of lower relative intensity are concentrically broadened to around ±40° in both RD and TD. Macro texture of 673K-cross rolled sheet resembles that of 623K-cross rolled sheet, exhibiting higher relative intensity towards (0001) pole. This fact agrees with the findings of researches by Xi [7] and Sulkowski [8]. In cross rolling, crystal orientation change by rigid body rotation around ND axis in plane-strain compression imparts the tilt of basal pole in TD as a result of accumulation of strain path change.

Figure 8 depicts the incomplete (0001) pole figures of AZ91D sheets annealed at 623K. In unidirectional rolling at 623K and 673K, the strong (0001) basal textures appear while the inclined peaks towards RD almost vanish from the pole figures as seen in Figure 8 (a) and (b). In cross rolling at 623K and 673K, the strong (0001) basal textures also appear as the peaks inclined towards TD disappear due to dislocation rearrangement and grain growth during the static recrystallisation annealing. However, distributions of lower relative intensities remain moderately concentric in both RD and TD in the pole figures.
4. Conclusions
The following remarks are drawn;
1) The 4 mm thick cast ingot AZ91D materials were successfully rolled into 1mm thick sheets under four different rolling conditions. The as-hot unidirectional and cross rolled sheets demonstrate good ductility ranging from more than 5% up to 15% elongation in tensile test. It is confirmed that the effect of change in strain path and higher rolling temperature induces to restrain the planar anisotropy of cross rolled sheets. The total elongations of annealed sheets under all the rolling conditions are well enhanced.
2) Macro textures of the as-unidirectional rolled sheets exhibit the typical double-peak type (0001) basal texture with the peaks inclined towards ±15° in RD. In contrast to the as-unidirectional rolled sheets, macro textures of the as-cross rolled sheets have double-peak (0001) basal texture with peaks inclined towards ±10° in TD. Contours of lower relative intensity are concentrically broadened to around ±40° in both RD and TD. After recrystallisation annealing at 623K, evolution of (0001) basal texture becomes noticeably predominant under all the rolling conditions. Distributions of lower relative intensities remain moderately concentric in both RD and TD in the pole figures of the annealed cross-rolled sheets.

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