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Microstructure and Property Relationship Controllable by Thermomechanical Processing of Mg–Al–Zn–Y–Ca Alloy Sheets

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The influence of thermomechanical processing on the microstructure and texture evolution is examined in the present study using an AZ31 alloy modified by Ca and Y addition. AZXW3100 alloy sheets were produced by hot rolling at two different temperatures of 450 °C and 500 °C. Changes in the deformation degree per pass at each rolling temperature were used to analyze the effect of process condition. The sheet rolled at 450 °C and the deformation degree per pass decreasing from 0.3 to 0.1 with rolling step shows a relatively strong texture with a basal pole split into the sheet rolling direction. On the contrary, the sheet rolled at 450 °C and deformation degree per pass increasing with rolling step from 0.1 to 0.3 shows a significantly weak texture with basal pole split into the rolling direction and the transverse direction. The sheets rolled at 500 °C show a further distinct texture type in which the basal poles are largely tilted into the rolling direction. The rolling condition influences also on the grain sizes in the recrystallized sheets, varying within a range of 6 μm and 21 μm after annealing at 400 °C for 10 min of the differently rolled sheets. The sheet formability is dependent on the texture sharpness, i.e. the weaker texture the higher formability. The sheet rolled at 450 °C with the increase of deformation degree per pass with the rolling step shows an excellent formability with the Erichsen Index of 8.1, which is about 3 times higher value than that obtained for a conventional Mg alloy sheet.

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1. Introduction

In the last decades magnesium (Mg) alloys have been widely investigated due to their favourable properties for a lightweight structure, e.g. low density, high machinability, and excellent damping capacity. However, the poor formability of Mg sheets, especially at room temperature, is one of the main reasons which retard industrial application of semifinished products from this lightest structural metallic material. The conventional wrought Mg alloys, e.g. based on the Mg–Al–Zn system such as AZ31, have a strong tendency of developing strong basal-type texture during the sheet rolling process. The basal-type texture, in which most grains have their *c*-axes in the sheet normal direction (ND), causes limited sheet formability owing to the restricted activity of the $\langle a \rangle$ dislocation slip, especially under loading in ND. Though strain along ND can be accommodated by pyramidal $\langle c+a \rangle$ slip with a Burgers vector of $\langle 11-23 \rangle$, this deformation system can be activated only at high temperature because of the high critical resolved shear stress (CRSS) at room temperature. To improve the formability of the Mg sheets, it is essential to provide a way to weak the texture. In

case of a weak basal-type texture, it is expected that $\langle a \rangle$ -dislocations with a relatively low CRSS contribute to accommodating of the imposed deformation. It has been reported that texture weakening can be achieved by alloying Mg with yttrium (Y) and rare earth (RE) elements such as cerium (Ce) or neodymium (Nd) [1–3]. Indeed, an increased ductility and strength have been observed in the sheet having a weaker texture. Even though the alloying addition of RE elements is an effective way of texture weakening and improvement of sheet formability at low temperature, the Mg alloys sheets with reduced alloying amount or without RE elements, which are strategically important raw materials, are much beneficial in ecological and economic viewpoint.

Addition of Ca into Mg alloys is known as an effective way to improve the high temperature mechanical properties and the ignition-proof behaviour. Especially, the simultaneous addition of Ca and Y results in an excellent ignition-proof behaviour. The Mg alloys with highly improved non-flammable characteristics have attracted much attention for research activities and industrial applications [4]. Recent studies showed that Ca addition leads to a significant weakening of the crystallographic texture in Mg alloys after thermomechanical treatments, such as extrusion [5] or rolling [6, 7], and consequently the improvement of the room temperature formability. Ca-containing alloys can be further strengthened by an optimised age hardening scheme [8, 9]. It is to

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be mentioned that the Ca-added alloys investigated for wrought processes handles mostly with Al-free Mg alloys, e.g. Mg–Zn–Ca and Mg–Mn–Ca systems. Moreover, the study on the microstructure evolution and the mechanical behaviour of the newly developed non-flammable alloy sheets containing Ca and Y is limited.

In the present study, the microstructure and formability of non-flammable sheets of AZXW3100, which is an AZ31 alloy modified by Ca and Y addition, produced by hot rolling were examined. A focus of the present study is put on the analysis of the process, microstructure and property relationship.

2. Experimental procedures

From the direct chill (DC) cast ingot of AZXW3100 alloy (Mg–3Al–1Zn–0.5Ca–0.5Y in wt%), the slabs with the thickness of 10 mm were machined for the rolling trials. The corresponding alloy composition is well recognized for its excellent ignition resistance and high strength after extrusion [10], while its properties after rolling, e.g. microstructure evolution during rolling and sheet formability, have not been systematically studied yet. The slabs were rolled to the final gauge of 1.1 mm using 4 different rolling conditions, listed in Table I. Two different schemes of deformation degree per pass were applied at the rolling temperatures of 450 °C and 500 °C; the deformation degree per pass increases with the rolling step ($\varphi = 0.1$ to 0.3), or decreases with the rolling step ($\varphi = 0.3$ to 0.1):

$$\varphi = -\ln \frac{t_{n+1}}{t_n}, (1)$$

where t_n = sheet thickness at n^{th} rolling step. The rolled sheets were annealed at 400 °C for 10 min to the full recrystallization. The optical microstructures of the rolled and annealed sheets were observed by using standard metallographic sample preparation techniques and an etchant based on picric acid [11]. Global texture information on the rolled sheets and sheets after recrystallization annealing was obtained using X-ray diffraction (Panalytical, Cu K_α , 40 kV and 40 mA), and the complete pole figures were calculated by using a Matlab toolbox MTEX [12]. The stretch formability of the sheets was examined by Erichsen tests of the as-rolled and annealed

sheets. The tests were performed in accordance to the DIN 50101 using the blank diameter of 100 mm, punch diameter of 20 mm at the punch speed of 5 mm/min and blank hold force of 10 kN. The Erichsen index (IE) was determined by the punch stroke corresponding to the maximal load. The results from the Erichsen tests are given as the average values from 3 samples, at least, for each condition.

3. Results and discussion

Figure 1 presents the optical micrographs and the recalculated (0002) pole figures of the sheets rolled at 450 °C at the as-rolled condition and after the annealing at 400 °C for 10 min. The numbers given on the micrographs indicate the average grain sizes measured by the linear intercept method. A strongly deformed microstructure with a large amount of twin bands is observed in the as-rolled sheets. The secondary phase particles are homogeneously distributed in the rolled and recrystallized sheets. In case of rolling at 450 °C with increasing deformation degree per pass, the recrystallized sheet has a fine homogeneous microstructure with average grain size of 6 μm . The grain structure of the sheet rolled by the 450-I condition is very stable and no significant grain growth occurs during the subsequent annealing, so that the grain size of the annealed sheet for 60 min at 400 °C is 8 μm . The as-rolled sheet shows a relatively strong texture with the max (0002) pole density of $P_{max} = 6.3$ m.r.d. (multiple random distribution) with a distinct split of the basal poles into the rolling direction (RD). The recrystallization annealing leads to a significant texture weakening such that the max pole density of the annealed sheet is $P_{max} = 2.6$ m.r.d. Interestingly, the texture weakening occurs in such way that the basal pole split into the sheet transverse direction (TD) becomes more visible, while the basal pole developed in the RD rapidly weakens during the recrystallization. The development of recrystallization texture with the basal pole split into the TD has been reported in various RE or Ca containing Mg alloys sheets, but mostly in Al-free Mg alloys, such as Mg–Zn–RE and Mg–Zn–Ca systems [1, 6, 7, 13, 14]. The present results show that the Al-containing alloys can have such texture component by controlling the thermomechanical treatment conditions.

The change of the scheme of the deformation degree per pass, i.e. 450-dec, results in a significantly different microstructure and texture in comparison to the 450-I condition. Firstly the sheet rolled by the 450-D condition has a relatively large grain sizes, 24 μm and 18 μm at the as-rolled and recrystallized states, respectively. Secondly, the slight spread of the basal poles into the RD indicates that the basal planes are largely aligned in the sheet normal plane (ND). Moreover, there is no qualitative texture change during the recrystallization annealing, and the texture weakening degree resulting from the recrystallization is much lower than that observed in the 450-I sheet.

TABLE I

Rolling conditions examined in the present study

Rolling condition	Rolling temperature [°C]	Deformation degree per pass (φ) (total 11 rolling steps)
450 – I	450	0.1 → 0.3 ^a
450 – D	450	0.3 → 0.1 ^b
500 – I	500	0.1 → 0.3 ^a
500 – D	500	0.3 → 0.1 ^b

^aincreasing with rolling step

^bdecreasing with rolling step

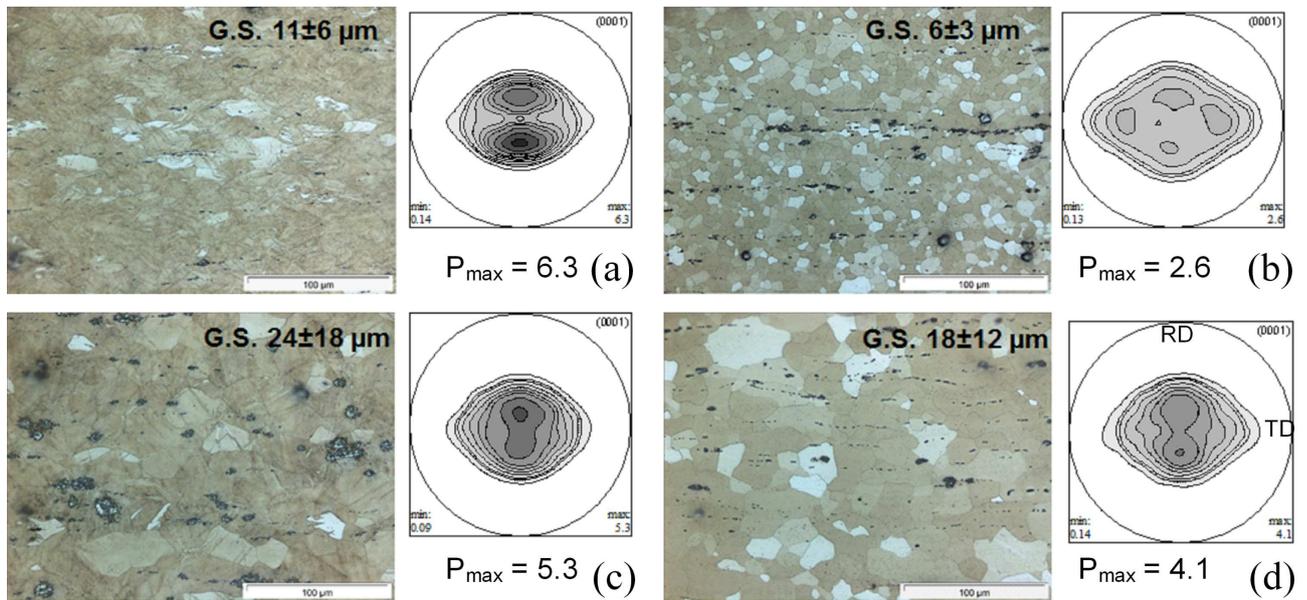


Fig. 1. The optical micrographs and the recalculated (0002) pole figures of the sheets rolled at 450 °C at the as-rolled condition and after recrystallization annealing at 400 °C for 10 min.: (a,b) 450-I sheets and (c,d) 450-D sheets.

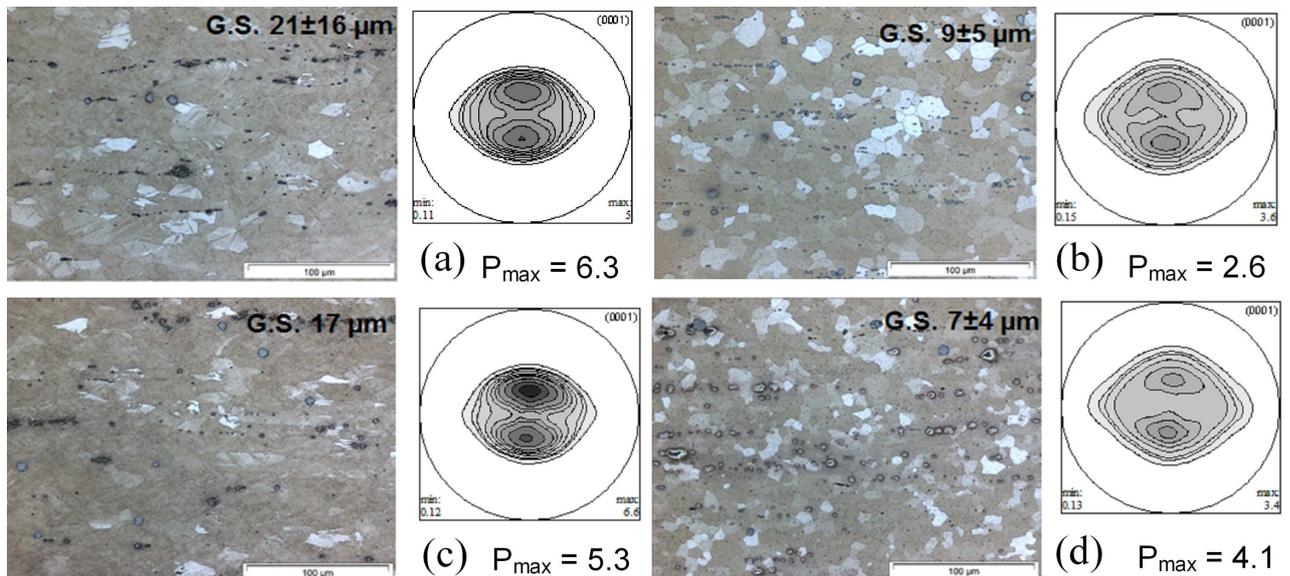


Fig. 2. The optical micrographs and the recalculated (0002) pole figures of the sheets rolled at 500 °C at the as-rolled condition and after recrystallization annealing at 400 °C for 10 min.: (a,b) 500-I sheets and (c,d) 500-D sheets.

In case of the rolling at 500 °C, the influence of the step reduction scheme on the microstructure and texture evolution is negligible, Fig. 2. The rolled sheets by using the 500-I and 500-D conditions show a clear basal pole split into the RD, which is similar to that observed in the 450-I sheet. The recrystallization annealing leads to the formation of the large basal pole split into the RD with the tilting angle of 20°–30° from the ND, while the texture weakens to the $P_{max} = 3.6$ and 3.4 after the annealing at 400 °C for 10 min of 500-I and 500-D sheets, respectively. The grain sizes of the recrystallized sheets, 9 μm

and 7 μm, are comparable to that of the 450-I sheet. It is to mention that a large amount of the stringer structure of the oxide inclusions along the RD is observed in the optical micrographs. These microstructural features are originated from the casting defects, e.g. micro-voids and oxide inclusions, which are elongated during the rolling procedures.

Figure 3 presents the sheet samples after the Erichsen tests and the Erichsen index (IE) of annealed sheets after rolling by 450-I and 450-D schemes. In the case of the rolled samples at 500 °C, some casting defects were found

near the fracture surfaces. The optical micrographs of the sheets rolled at 500 °C show indeed a larger amount of the stringers than the sheets rolled at 450 °C, Fig. 2. Those defects are correlated with early fractures during deformation and a verification of the IE is required. Consequently, only results from the rolled sheets at 450 °C are presented in this section. The annealed sheet after rolling by 450-I scheme shows the IE of 8.1, while the 450-D sheet has the IE of 4.2. The excellent stretch formability of the 450-I sheet after the recrystallization annealing is attributed to the weak texture with the TD split component, which is advantageous for the material flow in the sheet thickness direction during the stretch forming due to the high activity of basal $\langle a \rangle$ slip. Furthermore, the fine grain structure with average grain size of 6 μm and the fine particles distributed homogeneously enhance the sheet formability.

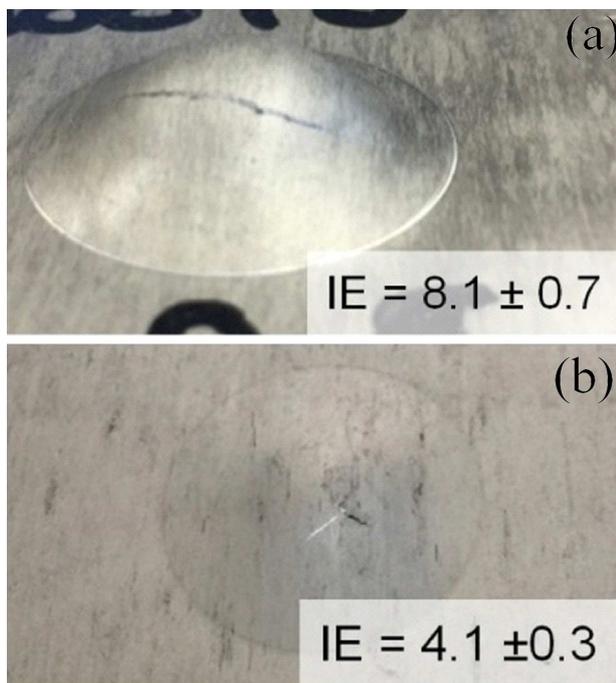


Fig. 3. Appearances of the Erichsen samples. (a) 450-I sheet and (b) 450-D sheet after the recrystallization annealing at 400 °C for 10 min. The Erichsen index (IE) is also given in the figure.

4. Summary

The influence of the rolling parameters on the microstructure and formability of AZWX3100 alloy sheets, which were produced by direct chill casting, warm rolling and recrystallization annealing, was investigated. The results indicate the grain structure and texture are controlled by varying the rolling schedules,

in terms of the rolling temperature and deformation degree per pass. The texture component of the basal pole split into TD develops by rolling at 450 °C with the deformation degree per pass increasing with the rolling step, while a relatively strong basal type texture is formed by the rolling with decreasing deformation degree per pass. It is obvious that the AZXW3100 sheet having the weak texture with TD split shows excellent sheet formability. Further studies on the deformation and recrystallization mechanisms in correlation with thermomechanical treatments will identify the fundamental mechanisms leading such variety in the microstructure and texture development.

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