Relationship between Deep Drawability and Microstructure of Magnesium Alloy*

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Mg–1.5Zr alloy blanks 1.0 mm in thickness, made from cast and rolled materials, were deep-drawn at room temperature and 150°C. The microstructures were examined and deformability evaluated. The grain size of blanks from cast and rolled materials was 40 µm and 15 µm, respectively. The basal texture was confirmed for rolled-blanks, while the microstructures of cast blanks were randomly orientated. The limiting drawing ratio for cast materials was 1.65 at room temperature and 1.75 at 150°C. For rolled-blanks, it was 1.45 and 1.70 at room temperature and 150°C, respectively. The cast-blanks deformed in a similar way at room temperature and at 150°C, exhibiting a decrease in thickness in the punch-shoulder areas and a progressive increase along the side wall toward the rim of the cups. Rolled-blanks tested at 150°C showed the similar trend as cast-blanks and exhibited evidence of dynamic recrystallization in the punch-shoulder areas, contributing to an improvement on the limiting drawing ratio. When the deep-drawing exceeded the limiting drawing ratio, the cast-blanks tested at room temperature and 150°C and also the rolled-banks tested at 150°C fractured in the rim area of the cups, while the rolled-blanks at room temperature fractured from the punch-shoulder areas. [doi:10.2320/matertrans.L-M2020827]

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1. Introduction

Magnesium has a high specific strength, and as an alternative to plastic, enables the fabrication of slimmer and more sophisticated products. Because of its excellent heat dissipation and electromagnetic wave shielding properties, it is used for portable electronic equipment such as notebook computers. However, general-purpose magnesium alloys are known to be difficult to work, and casting methods, such as die casting, are employed in the production of thin-walled parts. When a large amount of lithium is added to magnesium, a body-centered cubic β phase is crystallized, and the formability at room temperature is remarkably improved. Therefore, press-formed products using Mg–Li alloys have been put to practical use in some high-value-added products. This study focuses on hexagonal magnesium alloys (hereinafter referred to as magnesium alloys) but excludes the Mg–Li system.

Magnesium alloys are susceptible to basal slip, and the rolled or extruded material has a basal texture. Sheet metal with a developed basal texture is not easily deformed in the thickness direction and is difficult to press-form at room temperature. On the other hand, at 200–300°C, the formability is significantly improved, and warm press forming technology is applied to shallow and uncomplicated parts.

For magnesium alloy sheets, material development is underway for the purpose of controlling the basal texture i.e. randomization of crystal orientation. However, it is reported that randomization of crystallographic orientation while effective in improving bendability and stretch formability, has only a limited effect on deep drawability. Magnesium alloy sheets have large anisotropy in mechanical properties, and specific studies based on the deformation mode are required.

In the field of press forming, many studies have used Mg–Al alloys such as AZ31 (Mg–3Al–1Zn–0.3Mn). However, these alloys are not always suitable for examining the properties of magnesium because they have many constituent elements with high concentrations. While research studies using pure magnesium would be ideal, it is difficult to obtain uniform grain size due to the difficulty of rolling and abnormal grain growth. For this reason, the present study focuses on the use of Mg–Zr binary alloy as a research material.

With the Mg–Zr binary alloy the solidification structure can be generally regarded as a single phase. In addition, the solidification structure is grain-refined, uniform in grain size and random in grain orientation due to the effect of Zr. In addition, grain growth during processing and heat treatment is minimal. As it is possible to obtain an ingot without micro-porosity, a sheet cut from the ingot can be used as a random orientation material for the deformation studies.

The purpose of this investigation was to clarify the relationship between the deep drawability and the microstructure of a Mg–Zr alloy, and to obtain basic knowledge of the deep drawability of magnesium alloys from a materials perspective.

2. Experimental Procedure

2.1 Generation of alloy ingots

A Mg–Zr alloy containing 1.5 mass% Zr was prepared using Mg (99.93 mass%) and Mg–33 mass% Zr master alloy. Magnesium was melted in an electric furnace, Zr was added at 750°C and the alloy cast at 700°C in a mold held at room temperature (20–25°C). For this study, two ingots of different shapes were produced. One was a plate-shaped ingot (10 mm thick, 50 mm wide, 80 mm long) used for rolling, and the other was a cylindrical ingot (36 mm diameter, 80 mm long) which was sliced and used for evaluation of deep drawing behavior.

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2.2 Preparation of rolled sheet

The plate-shaped ingot was reduced to 7 mm × 45 mm × 70 mm by milling and homogenized at 400°C for 12 h. It was then rolled at a rolling speed of 5 m/min (0.083 m/s) and a temperature of 400°C using a small two-high rolling mill with a roll diameter of 70 mm and a width of 120 mm. The reduction amount was 0.1 mm/pass in order to obtain a high-quality final sheet 1.0 mm thick and without microscopic cracks. Before each pass, the sample was heated at 400°C for 10 min. The final rolled sheet was subsequently annealed at 400°C for 12 h.

2.3 Preparation of test pieces for deep drawing

Ten millimeter sections were removed from both ends of the cylindrical ingot, and the remaining part was cut with a precision cutting machine. The cross section was polished with emery paper to provide a 1.0 mm-thick sample. Additionally, a specimen processed to a predetermined diameter (D₀) was used as a test piece.

A disc of 36 mm Ø was cut from the rolled sheet by wire-cut electric discharge machining and processed to D₀ in the same manner as the test piece. In this paper, a test piece made from a cylindrical ingot is referred to as a cast blank, and a test piece made from a rolled sheet is referred to as a rolled blank.

Each blank was examined by optical microscopy and EBSD analysis at an acceleration voltage of 15 kV and a measurement interval of 0.75 µm.

2.4 Deep drawing

Figure 1 shows the main parts of the test equipment for deep drawing. This facility consists of a die (hole diameter d_D = 23 mm, shoulder radius r_D = 5 mm), a punch (diameter d_p = 20 mm, shoulder radius r_p = 5 mm) and a blank holder. A blank (1.0 mm thick) is placed in the blank holder together with a stainless steel ring spacer (1.2 mm thick) which positions the blank on the punch. The die slides down to the blank holder by hydraulic pressure and the punch thrusts upward on the blank from below. The clearance between the punch and the die hole was 1.5 mm all around and a gap between the blank and the blank holder was 0.2 mm. The forming temperatures were room temperature (20–25°C) and 150°C, and the limiting drawing ratio (D_max/d_p) was used as an evaluation index for drawability.

3. Results

3.1 Limiting drawing ratio

Figure 2 shows the microstructures of the cast- and rolled-blanks as inverted pole figure (IPF) maps. In optical microscopy, the grain boundaries were unclear and the grain size was occasionally difficult to evaluate due to the etching conditions; thus, the IPF images by EBSD are shown here. Each blank consisted of well-shaped equiaxed crystals with average grain sizes of 40 µm and 15 µm for the cast and rolled blanks, respectively. Figure 3 shows the (0001) pole figure of each blank. The crystal orientation of the cast blank was random, and the development of the basal texture was confirmed in the rolled blank.

Figure 4 shows the appearance of deep drawn cups. The limiting drawing ratio of the cast blank was 1.65 at room temperature and 1.75 at 150°C. The limiting drawing ratio of the rolled blank was 1.45 at room temperature and 1.70 at 150°C. Hereinafter, the cup formed from the cast blank is referred to as "A," and the cup formed from the rolled blank is referred to as "B." "R.T." and "150" are added as subscripts indicating formation at room temperature and 150°C, respectively.
3.2 Hardness and thickness strain of deep drawn cups

As shown in Fig. 5, a cup formed from each blank was cut at three locations in the vertical direction so as to pass through the center axis of the cup, and the hardness was measured at the center of each cross section of ① to ③. A micro-Vickers hardness tester and an optical microscope with measurement software were used. The hardness and dimensional changes in cross-sections were measured in four parts: rim, side wall, bottom corner (R), and bottom. The hardness values were obtained from the rim to the bottom of the cup at an interval of approximately 2 mm. As shown in Fig. 6, each hardness indentation mark was numbered from the bottom to the rim for identification purposes. The wall thickness at each numbered location (measured point for hardness) was also measured and the strain in thickness at each point was calculated in order to obtain the relationship between hardness and strain in thickness. The above measurements were similarly performed for all cross sections. Therefore, each point has three data sets. The thickness of the bottom (measurement point No. 1) was the same as the thickness of the blank in all cups. Therefore, based on the thickness $t_0$ of No. 1, the thickness strain $\varepsilon_t$ was obtained from the relationship: $\varepsilon_t = (t - t_0)/t_0$.

Figure 7 shows the variation in the values of hardness and strain along the cross-section of the deep drawn cups. For cups drawn from the cast blanks (A_{R.T.} and A_{150}), the value of hardness showed the same upward tendency, increasing slightly from the bottom of the cup to the rim and the graphs almost overlapped. The thickness strain distribution of A_{R.T.} and A_{150} showed the same tendency. The thickness decreased at R areas and increased substantially along the side wall toward the rim of the cups, and the graphs also overlapped. For cups drawn from the rolled blanks (B_{R.T.} and B_{150}), the hardness profiles showed the same tendency as A_{R.T.} and A_{150}. However, the graph illustrating the thickness strain distribution showed a clear difference with the forming temperature. The thickness of the cross-section of B_{R.T} only exhibited a positive increase from the bottom to the rim,
while the thickness distribution of B150 was similar to those of AR.T. and A150. However, the reduction in the thickness of the R areas was larger than those of AR.T. and A150, and the increase in the thickness from the side wall to the rim was small.

As shown in Fig. 8, the decrease in thickness of the R regions during deep drawing is caused by the tensile stress in the circumferential and radial directions ($\sigma_\theta$ and $\sigma_r$, respectively), and the increase along the side wall is caused by the compressive stress in the circumferential direction ($\sigma_\theta$). Therefore, it can be concluded that the cast blank with random crystallographic orientation would show similar deformation behavior to the tensile and compressive stresses, regardless of the forming temperature. On the other hand, in the case of rolled blanks, there should be a clear difference in deformation behavior with respect to tensile stress between room temperature forming and warm forming.

4. Discussion

4.1 Cast blank

As shown in Fig. 7, the thickness strain at the rim of AR.T. and A150 exceeded 0.2, but the work hardening was minimal in both cases. This indicates that the deformation depends on the activity of the single slip system, suggesting that basal slip is the main deformation factor. Figure 9 shows IPF images taken from a vertical section of the drawn cups at the rim. In both AR.T. and A150, extremely fine grains were observed locally, as shown in regions 1 and 2 indicated by arrows in the figure.

Dynamic recrystallization of pure magnesium has been known to occur over a wide temperature range (20–500°C). Due to the intersection of twins, subdivision of the crystal occurs in the region between the twin planes, and fine grains with low dislocation density are generated during deformation. Such twin dynamic recrystallization (TDRX) occurs in the low-temperature range (20–200°C). In this case, fine recrystallized grains are formed as chains between twins, and their intragranular dislocation density is approximately three orders of magnitude lower than that of other parts, causing work softening. The ultrafine structure obtained in this study agrees well with TDRX at both room temperature and 150°C. Therefore, it is considered that similar dynamic recrystallization occurred.

Figure 10 shows an image of the fractured cups. The cups cracked at the rim because the limiting drawing ratio was exceeded. The cracks formed approximately 45° to the circumferential direction of the cups. This suggests that the cracks were caused by compressive stress in the circumferential direction. Figure 11 shows the (0001) pole figures of the vertical cross-sections of the rim regions of the cups drawn from cast blanks at room temperature (AR.T.) and 150°C (A150). For both room temperature forming and warm forming, it was confirmed that circumferential compressive stress causes a pronounced basal texture in the cross-section of the rim areas.

The basal plane rotates during forming, due to the compressive stress, so that it becomes parallel to the vertical cross-section of the cup. Therefore, even if the crystal orientation is random as in the cast blank, the shear stress component on the basal plane approaches zero as the forming
progresses. Consequently, the compressive stress generated on the basal plane does not activate the tension twin \{1012\}(1011); thus, the fracture stress is considered to have been reached before the second order pyramidal slip \{1122\}(1123), i.e., (c + a) slip, and the compression twin \{1011\}(1120) were sufficiently activated. The limiting drawing ratio for warm forming was larger than that for room temperature forming because of the dynamic recrystallization promoted by heating.

### 4.2 Rolled blank

In the case of a rolled blank, both the radial stress \(\sigma_r\) and the circumferential stress \(\sigma_\theta\) act parallel to the rolling surface. Therefore, it is difficult to obtain the thickness strain by basal slip. The (c + a) slip activity is necessary to develop thickness strain by tension. However, the (c + a) slip has a critical resolved shear stress (CRSS) as high as 40 MPa (room temperature), and it is unlikely to be a major deformation mechanism, especially at room temperature or 150°C. On the other hand, in the case of compression, tension twins can occur. Tension twinning is caused by tension in the c-axis direction or compression in the normal direction of the prism planes. Its CRSS is as low as 3 MPa and is minimally dependent on temperature. In the case of a rolled blank, compression from the circumferential direction is equivalent to compression from the normal to the prism planes.

The shear strain associated with tension twins is as low as 0.13, but the formation reduces the stress concentration. In addition, since the c-axis is tilted to 86°, it even in a sheet with a basal texture, shear stress acts on the tilted basal plane. Based on the above, tension twins can be cited as a factor in increasing the thickness due to compressive stress.

Figure 12 shows the optical microstructures of BR.T. and B150 in the R. Due to the lateral load from the punch to the sheet surface, the R is bent at the beginning of forming. At that time, compressive and tensile stresses are generated inside and outside the neutral plane, respectively. In the case of a rolled blank, the compressive stress due to bending acts from the direction normal to the prism plane. Therefore, twins were prominently observed inside the neutral plane of the R areas in BR.T. On the other hand they were minimally observed outside the neutral plane of the R, where tensile stress due to bending occurred. In this case, the tensile stress occurs parallel to the basal plane. Therefore, it is expected that twin and basal slip are unlikely to occur outside the R. However, even at room temperature, forming was possible without breaking to the extent that the limiting drawing ratio could be derived. Despite the relatively high limiting drawing ratio of 1.70, almost no twins were observed in the optical microstructures inside and outside the R of B150, as shown in Fig. 12.

Figure 13 shows that when the punch touches the blank, a certain area between the punch and the die generates a shear stress \(\tau_r\) in the cross section during forming. At the same time, conjugate shear stress \(\tau_\theta\) acts on the basal plane perpendicular to the cross section. For example, \(\tau_\theta\) works on the bottom of the range indicated by the double arrow in the figure. Thus, when tensile and compressive stresses caused by bending acts on the rolled sheet, basal slip may occur due to \(\tau_r\), respectively. As shown in Fig. 8, biaxial tension is applied to the R, and a force is applied to the sheet surface from the lateral direction. Therefore, a very severe condition is imposed on the material. However, in the case of magnesium alloys with a basal texture, the lateral load induces a basal slip and reduces the stress concentration. Basal slip activity due to conjugate shear stress has been confirmed in single crystals.

Figure 14 is a (0001) pole figure near the central axis of the R of BR.T. and B150 (area of 300 \(\mu m\) × 300 \(\mu m\)). In the case of BR.T., the high (0001) pole density region is separated into two portions. The angle between regions of extremely high density on the great circle, coincided with the c-axis tilt angle of 86° associated with the tension twin. On the other hand, in the (0001) pole figure of B150, twin relations such
as those occurring in BR.T were not detected, and the basal planes were widely distributed around the central axis (\(a_1\)) of the cup longitudinal section. Figure 15 shows an enlarged view of the observation area as an image quality map (IQ map) and IPF map. The tension twins are indicated by red lines on the IQ map of BR.T and confirm the occurrence of many tension twins aligned in one direction. On the other hand, the IQ map of B150 showed a characteristic contrast as if one grain was divided into multiple grains. In the IPF map, sub-grain boundaries were formed in the grains, as in the region surrounded by the dashed line in the figure, and the parts that transformed to new grains were widely observed. The sub-boundaries or part of the grain boundaries dividing the emerged grains had a twinning relationship with the matrix grains. This suggests that multiple twins are formed in one grain, and the grains intersect with each other, causing them to split and rotate, leading to dynamic recrystallization. When grain refinement, softening, and rotation occur in the process described above, the wall thickness of the cup may decrease by activating the basal (a) slip or the prism (a) slip due to tensile stress. This is one of the reasons why the critical drawing ratio of the sheet having the basal texture was improved by warm forming.

As shown in Fig. 16, when the deformation exceeded the limiting drawing ratio, the BR.T. cracked at the R and B150 cracked at the rim. When the stress in the R is relaxed, cracks start to occur at the rim. However, unlike the case with cast blanks, the rolled blanks cracked when the thickness strain at the rim was relatively minor. This needs to be studied further.

5. Conclusion

In this study, the following conclusions were obtained.

1. The cast blanks exhibited similar strain profiles along the cross-section of the deep drawn cups under both room temperature forming and warm forming, indicating similar deformation processes occurring under
tensile and compressive stresses in both forming cases. The limiting drawing ratios were also relatively close. As the deformation progresses, the basal plane rotates due to the compressive stress in the circumferential direction and approaches parallel to the longitudinal section of the cup. Therefore, there is no deformation mechanism to relieve compressive stress, and cracks occur at the rim. As a result, no significant improvement in deep drawability was observed, although the cast blank had a random crystal orientation.

(2) In the rolled blank, a difference was found in the deformation behavior with respect to tensile stress when comparing room temperature forming and warm forming. In the case of warm forming, the evidence of sub-grain boundaries and dynamic recrystallization was observed in the R regions of the drawn cup. These occurrences, probably caused by twin boundary crossings, lead to grain refining, softening, and grain rotation. The resulting effects open the possibility to decrease the thickness under tensile stress conditions and improve the limiting drawing ratio during warm forming.

In the case of warm forming, the R of the cup had evidence of sub-grain boundaries and dynamic recrystallization, probably caused by twin crossing. The resulting refinement, softening, and rotation of the grains can decrease the thickness under tensile stress conditions and improve the limiting drawing ratio during warm forming.

(3) In the case of a rolled blank having a basal texture, the lateral load on the sheet surface induces basal slip due to conjugate shear stress and reduces stress concentration.

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