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STRENGTHENING MG-AL-ZN ALLOY BY REPETITIVE OBLIQUE SHEAR STRAIN

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Abstract

Grain refinement is one of the possible ways to enhance the strength of magnesium without losing the ductility and/or toughness. In this study, severe plastic working by caliber rolling has been demonstrated to refine the grain structure of a commercial AZ31 Mg-Al-Zn alloy at a commercial processing speed. As a result, ultra-fine-grain structure with sub-grains in a sub-micro-meter scale was obtained. A simultaneous operation of oblique shear strain weakened the basal texture compared to that of the initial as-extruded alloy, and resulted in tensile ductility comparable to that of the commercially extruded alloy, and showed a higher asymmetry ratio of yield stress in compression/tension than that of the as-extruded alloy.

Grain Refinement of Mg for Strengthening

Magnesium alloys are being widely investigated throughout the world for the weight reduction of structural components due to their low density. In structural applications, the strength of magnesium alloys should be increased along with their ductility to improve the relatively low stiffness, so that the alloys can be formed into complex shapes. One of the major microstructural factors for strengthening of metals is grain refinement. It is even more effective in increasing the strength of magnesium alloys because of the relatively large Taylor factor in its HCP structure compared to that of the other lightweight metal, aluminum [1]. The tensile elongation also increases with grain refinement due to the enhanced activity of grain boundary plasticity [2]. Another important factor for enhancing the ductility is the distribution of the basal plane orientation, *i.e.*, the basal texture. The basal texture can be modified by changing the applied shear loading direction during severe plastic working [3], and/or by changing the recrystallization behavior by a combination of distributing nano-scale particles and plastic working [4]. Wrought magnesium alloys are generally known to show yield asymmetry [5]; the yield stress in tension is higher than in compression. The asymmetry of yield stress can be weakened due to the texture modification. Wang *et al.* demonstrated that the asymmetry ratio of yield stress (yield stress in compression / yield stress in tension) increases with grain refinement and approaches 0.9 in the AZ31 alloy [6]. Thus, the combination of grain refinement and the randomization or the weakening of the basal texture may enhance the ductility in high-strength Mg alloys.

There are some reports that the application of repetitive shear loading could refine the grain structure [7-9] and modify the texture of Mg alloys, *i.e.*, repetitive processing of equal-channel-angular-extrusion (or -pressing) (ECAE or ECAP) could incline the basal pole at certain degrees against the extrusion direction, while a strong basal texture is formed almost parallel to the extruded direction by conventional extrusion [10-13]. In our earlier work, when the basal pole was inclined about 45 degrees against the extrusion direction, the tensile yield stress was reduced to just half of that in a directly extruded alloy but the tensile elongation increased to more than double [3]. Koike *et al.* revealed by transmission electron microscopy that an inclined basal pole activates non-basal <a> dislocations as well as basal dislocations even at room temperature [14]. On the other hand, Agnew *et al.* demonstrated by using in-site neutron diffraction that the polycrystalline modeling validated the elastoplastic deformation in the ECAE AZ31 alloy [15]. They pointed out that more than 50 % of the plastic deformation was dominated by the basal slip even after macroscopic yielding, while the prismatic or pyramidal <a> dislocation slip became more active than the basal dislocation in a conventional AZ31 extrusion. Other procedures have been applied to the AZ31 alloy to refine the grain structure and to modify the texture, *e.g.*, Kim *et al.* demonstrated that differential speed rolling (DSR) at a relatively low temperature of 413 K enhanced the strength of AZ31 effectively by means of the ultra-fine grained structure [16]. They also reported that the weakened intensity of the (0002) basal pole could increase the tensile elongation. Xing *et al.* demonstrated that the grain refinement of AZ31 could increase the tensile strength by multi-directional forging by decreasing the processing temperature for each forging [17]. Yang and Ghosh conducted a grain refinement of the AZ31 alloy by weakening the basal texture using an alternate biaxial reverse corrugation pressing [18]. The above mentioned studies suggest that the grain refinement along with the weakened basal texture can activate the basal and/or non-basal dislocations rather than deformation twins and result in enhancing the strength-ductility balance at room temperature.

Repetitive Shear Straining by Caliber Rolling

In this study, grain refinement with a weakened texture is demonstrated by applying a heavy oblique shear strain using caliber rolling, which can operate continuously at a commercial processing speed and commercial scale. Caliber rolling has been used mostly in the research of steels to control the cross sectional shape of products [19] and/or strengthening the material with toughness [20]. Aoki and Yanagimoto reported the effect of caliber shape on the strain distribution in the cross section using the plasticine models. [19]. Inoue *et al.* have demonstrated that the accumulated strain in the cross section could be effectively increased using caliber rolling by numerical simulation [21]. In this study, we have paid attention to the effect of oblique shear strain on the texture evolution. A schematic illustration of the caliber rolling is shown in Fig. 1 [22]. The upper roll has a channel of identical dimensions with the counterpart bottom roll. However, the bottom portion of the caliber has a smaller roll diameter than that of the top portion of the caliber roll. Therefore the outer rolling speed at the bottom (V_{btm}) is slower than that at the top (V_{top}). When a billet is subjected to rolling, the material is deformed plastically during a certain duration. The cross sectional area of the billet is reduced at a fixed ratio, while the discrepancy of V_{top} and V_{btm} creates a certain shear strain simultaneously after passing the roll, as illustrated in Fig. 1(a). After rotating the rolled billet 90 degrees clockwise around the rolling axis, the billet is subjected to rolling in the same way as the former pass. The cross section is reduced again with the same reduction ratio, but the shear direction is the opposite from that of the former pass, as illustrated in Fig. 1(b). Thus the resultant texture after the secondary pass is weakened as compared to that of the formerly rolled billet, although the accumulated strain in the secondary processed billet is much higher than that in the former one. Since the actual deformation may be more complicated due to a lateral extension *etc.* throughout the processing, a detailed numerical simulation by the finite element method has been carried out to understand the deformation sequence [23].

Caliber Rolling for AZ31 alloy

In this experimental work, a commercially extruded AZ31 alloy (Mg-2.9 Al-0.8 Zn-0.42 Mn, by mass %) was used as the starting material. The commercial extrusion was conducted at a temperature of 623 K with an extrusion ratio of 16:1 by Osaka Fuji Kogyo, Japan. The grain structure of the initial material was inspected by optical microscopy, and the average grain size was measured to be ~ 25 μm. A cylindrical billet was machined from the extruded bar with a diameter of 42 mm and a length of 100 mm. The billet was kept at 473 K in a furnace in air for one hour and then subjected to severe plastic working by the caliber rolling facility at room temperature [22]. The outer speed of the roller was fixed at 0.5 m/s. The reduction ratio of the cross sectional area was designated to be -18% for each caliber with an analogy shape as shown in Fig. 1. The billet was rolled repetitively by 18 passes with rotating the rolled bar 90° for each pass. In the final pass, the rolled bar was rotated 90° and rolled with the same caliber as that of the former pass. The cumulative reduction in the area was estimated to be about 95% after 18 passes, corresponding to an equivalent strain of 3.1 estimated from the reduction in area. To avoid abnormal grain growth, reheating was not conducted after the passes. The rolled billet was subjected to subsequent rolling within a few seconds. The surface temperature on the rolled specimen after 18 passes was measured to be ~ 433 K, thus the material was kept in a temperature range of 433 to 473 K during the repetitive rolling.

Effect of Repetitive Shear Straining

The microstructure at the mid-layer of the caliber rolled alloy after 15 passes was inspected by electron backscatter diffraction (EBSD) equipped in a field emission scanning electron microscope (JEOL F7400) to investigate the texture evolution. The orientation map of the grain structure in the caliber rolled alloy after 15 passes was shown in Fig. 2. The grain structure of high angle grain boundaries (> 15 degrees) and low angle grain boundaries are shown by black and gray lines in Fig. 2, respectively. The grain structure was found to be refined to an average grain diameter of 2.5 μm by caliber rolling. (0002) pole figure representing the basal plane was also measured by the Schulz reflection method at α-angles of 20° to 90° [22]. The

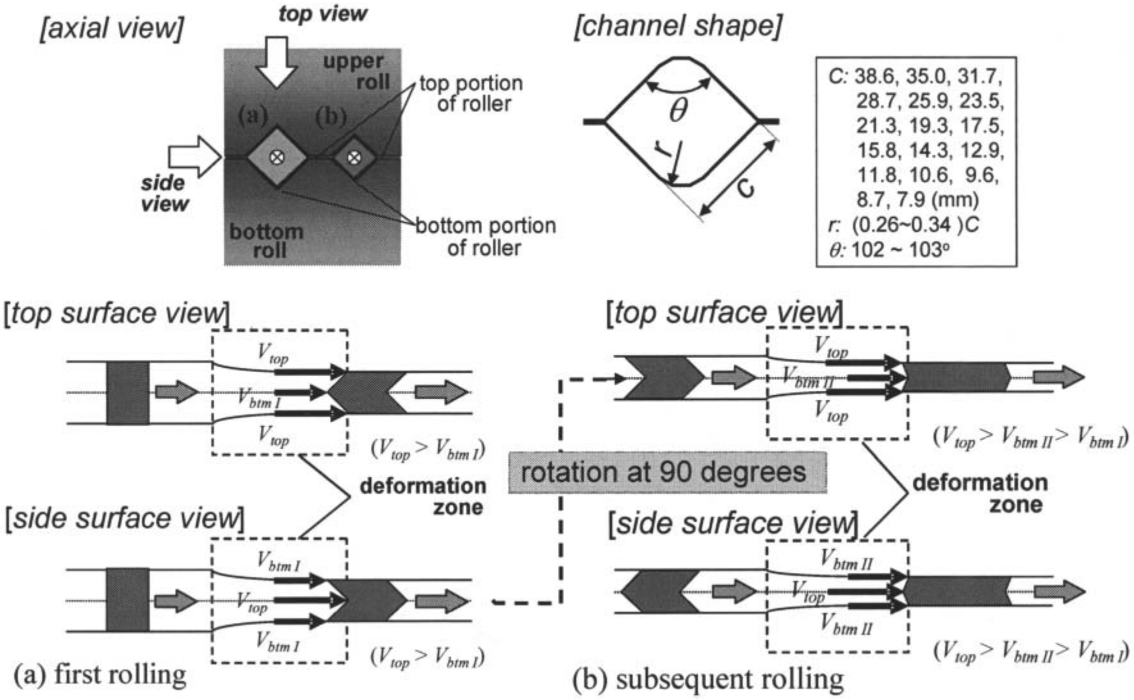

Figure 1 Schematic illustration of the caliber rolling process [22].

XRD measurement revealed that (i) the intensity of the basal pole in the caliber rolled alloy was shifted about 10 degrees from the center toward the transverse direction, (ii) the asymmetrical distribution of the basal plane and the weakening of the maximum intensity of the basal pole were formed in the caliber rolled alloy, although the material was subjected to repetitive severe plastic working.

Mechanical properties were examined using specimens machined from the initial extrusion and rolled bars having a gauge diameter of 3 mm and a gauge length of 15 mm for tensile tests and a diameter of 4 mm and a height of 8 mm for compression tests. The axial direction of each specimen was parallel to the rolling or extrusion direction. The tensile and compression tests were conducted at a strain rate of $1 \times 10^{-3}\ s^{-1}$ at room temperature by an Instron tester. Nominal stress-strain relations in tension and compression for the caliber rolled alloy and the as-extruded alloy are shown in Fig. 3. The tensile yield stress (σ_{tys}) in the caliber rolled alloy being approximately twice as high as that of the as-extruded alloy, although the tensile elongation exhibits a similar value. The curve of caliber rolled alloy in compression exhibit a similar trend with a concave shape similar to that of the as-extruded alloy. The plateau region of the flow stress after yielding increases due to the grain refinement, conforming to the experimental results of the as-extruded AZ31 alloy reported by Barnett *et al.* [24]. Although the yield stress in compression (σ_{cys}) also increases after the caliber rolling, the magnitude of increment in σ_{cys} is noticeably greater than that in σ_{tys}. The asymmetry ratio of yield stress in the caliber rolled alloy is noticeably higher than that in the as-extruded alloy, which suggests the weakened basal texture after caliber rolling. The tensile yield stress-elongation balance of the caliber rolled alloy is shown in Fig. 4. The reference data for the as-extruded alloy and AZ31 wrought alloys using other procedures reported in the literature [13,15,16,25,26] are also shown. The yield stress of the caliber rolled alloy is significantly higher than that of the reference alloys. A direct comparison with the yield stress of a fine-grained AZ31 (d: 1.4 μm) processed by DSR [16] suggests that an additional strengthening factor exists for the high strength caliber rolled alloy, because the caliber rolled alloy has grains of similar sizes (d: 1.5 μm) with high angle boundaries. The EBSD observation in Fig. 2 shows color gradation in several grains which suggests formation of a substructure in the grains. Therefore, the microstructure was inspected by strengthening factor. A typical microstructure of the caliber rolled alloy is shown in Fig. 5. The micrograph shows the formation of fine sub-grains with dense dislocations in the interior. The average size of the sub-grains was measured to be about 0.3 μm in diameter. Kim *et al.* estimated the strengthening effect by grain refinement using the well-known Hall-Petch (H-P) relation in AZ31; the slope (*k*) of the relation was 336 and 394 $MPa/(\mu m)^{-1/2}$ in the conventionally rolled and DSR alloy, respectively. By assuming that the slope of the H-P relation in the caliber rolled alloy is in the range of 336 ~ 394 $MPa/(\mu m)^{-1/2}$, the additional strength from the reduction of the grain size from 1.4 μm to 0.3 μm was estimated to be 330 ~ 387 MPa. The apparent discrepancy between the tensile yield stress of the caliber rolled alloy and the DSR alloy is 92 MPa, which is smaller than the estimated additional strength. The result suggests that since the caliber rolled alloy has a weakened texture and is composed of fine sub-grains with low angle boundaries, which act as a weaker barrier against dislocation motion than high angle boundaries.

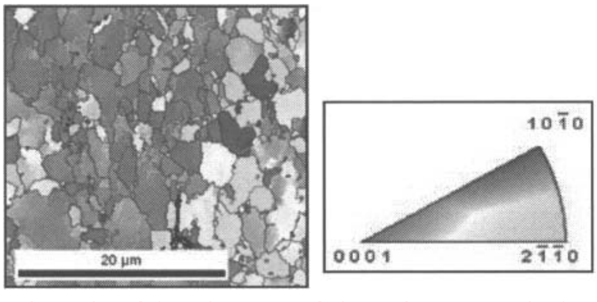

Figure 2: Orientation map of the grain structure in 15 passed alloy observed by EBSD. The grain structure of high angle grain boundaries (> 15 degrees) and low angle grain boundaries are shown by black and gray lines, respectively.

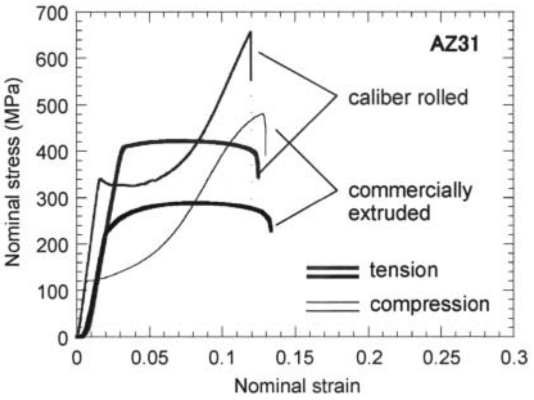

Figure 3: Nominal stress-strain relations in tension and compression.

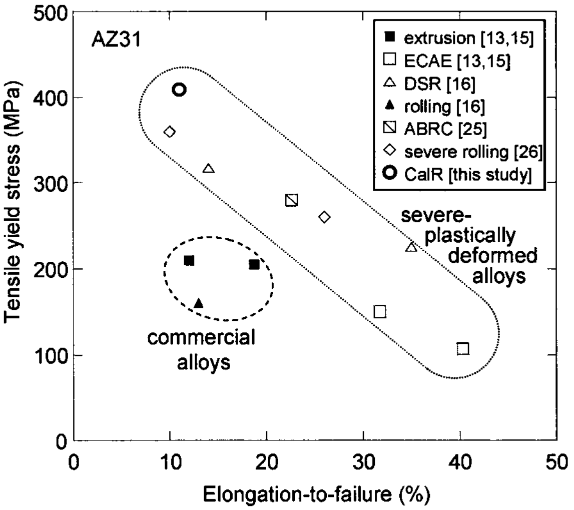

Figure 4: Tensile yield stress-elongation balance in the present alloy exhibits a superior combination [13, 15, 16, 25, 26].

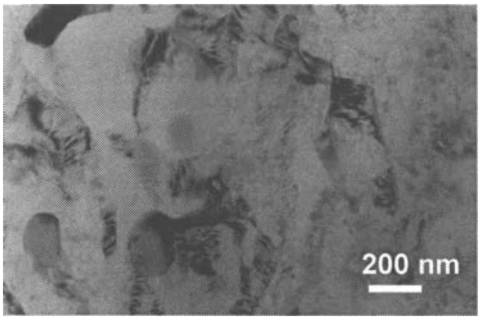

Figure 5: Typical microstructure after caliber oblique shear rolling for 18 passes.

Summary

Severe plastic working by caliber rolling refined the grain structure effectively at a commercial processing speed with the formation of fine sub-grains in sub-micro-meter scale and resulted in a high yield stress of over 400 MPa. A simultaneous operation of oblique shear strain weakened the basal texture from that of the initial as-extruded alloy and resulted in a certain tensile ductility comparable to the commercially extruded alloy and a higher asymmetry ratio of yield stress in compression/tension than that of the as-extruded alloy. It was summarized that the combination of refining grain structure and weakening texture was a possible procedure to improve the mechanical properties in Mg alloys.

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