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Microstructure and mechanical properties of Mg–Al–Zn alloy sheets severely deformed by accumulative roll-bonding

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Abstract The as-rolled AZ31 Mg alloy sheets were subjected to accumulative roll-bonding (ARB) at 300 °C up to three cycles. The microstructure and macro- texture are investigated by means of optical microscopy and X-ray analysis. The mechanical properties are evaluated by micro-hardness and tensile tests. Very fine grain size of 2.4 μm could be achieved after three passes of 50% thickness reduction. The recrystallized structure was already formed after one cycle of ARB. ARB processing resulted in a significant increase of ductility and slight decrease of tensile strength of the AZ31 alloy sheet. Basal texture was notably weakened after ARB processing.

Introduction

Wrought magnesium alloys attract attention since they have more advantageous mechanical properties than cast Mg alloys [1]. However, their low strength and ductility, due to the hexagonal close packed (HCP) structure with limited slip systems, is a major difficulty that must be overcome to enable their widespread consumption in industries. It is well accepted that grain refinement may significantly improve the mechanical behavior of Mg alloys [2].

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In recent years several severe plastic deformation (SPD) techniques have been proposed for deforming metals to very high plastic strains, with the aim of producing bulk submicron-grained materials at a relatively low cost [3]. Notable examples include [3–5]: severe plastic torsion straining (SPTS), multi-axis compression, accumulative roll-bonding (ARB), and equal channel angular extrusion or pressing (ECAE/P). The use of SPD techniques to process Mg alloys could potentially help to overcome the two main disadvantages of these materials, namely their small ductility and low strength, compared to those of competing materials.

The accumulative roll-bonding (ARB) is a relatively new method of severe plastic deformation proposed by Saito et al. [4] to achieve ultra high strains in metallic materials without changing the specimen dimensions. ARB has potential to be adopted by industry to produce fine-grained materials in the form of large sheets, due to its feasibility as a continuous process. The ARB process consists of repeated stages of cutting, stacking and roll-bonding of sheets. The ARB process has been proven to be very effective in enhancing the strength of aluminum, steels and copper [6–8]. However, the information available in the literature about the microstructural and property change of magnesium alloys during the ARB process is still very limited. Pe′rez-Prado et al. [9] used accumulative roll-bonding to produce the AZ31 sheets with a grain size of 3μm. del Valle et al. [10] found that the ultimate grain size was achieved during ARB of AZ61 and the degree of bonding depended on the rolling temperature and on the thickness reduction per pass. However, no mechanical data were provided in their work.

This work aims to investigate whether accumulative roll-bonding is an effective technique to improve the strength and ductility for the AZ31 Magnesium alloy.

Experimental

AZ31 Mg alloys hot-rolled sheet with the thickness of 1.5 mm was chosen for this study. The main alloying additions of these materials are 3% Al and 1% Zn. In performing the accumulative roll-bonding process, two pieces of the strip with dimensions of 200 mm × 75 mm × 1.5 mm were stacked to form a 3 mm thick specimen. Before stacking, the surfaces of the strips were degreased (in acetone) and wire-brushed (stainless steel brush with wire of 0.3 mm in diameter) to achieve good bonding. The stacked sheets were fixed in the corners using stainless wires and subsequently heated for 10 min at 300 °C in an electrical furnace. After heating, the specimen was roll-bonded by rolling using a 50% reduction without lubricant. The roll diameter was 400 mm. The rolled specimen was cut into two halves, and the above-mentioned procedure was repeated two times more to achieve three cycles.

The initial material and the deformed microstructures were examined both by optical microscopy. The grain size was measured on the longitudinal cross-sections of the sheets. Average grain sizes (d) were calculated from the optical micrographs by the linear intercept method.

Macrotexture measurements were performed in the as-rolled and ARBed samples with the aim of understanding the microstructure evolution during ARB processing. X-ray texture analysis was performed in a Philips APD-10 diffractometer. The X-ray radiation used was b-filtered CuKa. The polar angle ranged from 0 to 85° in steps of 3°. Calculated pole figures were obtained with the SIEMENS DIFFRAC/ AT software, using the measured incomplete {10–10}, {0002}, {10–11}, {10–12} pole figures. Sample surface preparation consisted of grinding using progressively smaller grit SiC paper and mechanical polishing with diamond powder of grain sizes 6, 3 and 1 mm. Final polishing was performed using a colloidal silica solution.

Micro-hardness and tensile tests were performed to evaluate the effect of ARB on the strength and ductility of the AZ31 alloys. Vickers microhardness (HV) was measured on the plane perpendicular to the longitudinal by imposing a load of 500 g for 20 s. The values reported for HV represent the average of seven separate measurements taken at randomly selected points. Tensile specimens with the 25 mm gage length parallel to the longitudinal axis were cut out of the ARBed billets. Tensile testing was carried out at room temperature under a constant cross-head speed condition (2.5 mm/min) on a tensile testing machine (Instron 8516).

Results and discussion

Microstructures

Optical photographs observed at the longitudinal cross-sections of the (a) as-rolled; (b) one-cycle ARBed; (c) two-cycle

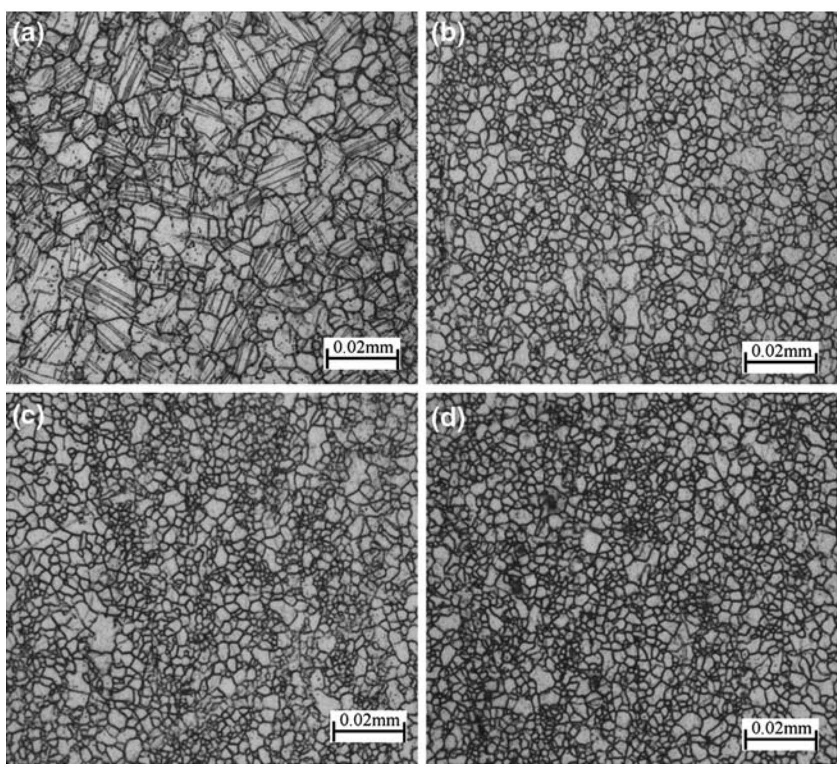

Fig. 1 Longitudinal section optical micrographs of the (**a**) zero-cycled (as-rolled); (**b**) one-cycle ARBed; (**c**) two-cycle ARBed and (**d**) three-cycle ARBed AZ31 alloys after processed at 300 °C

ARBed and (d) three-cycle ARBed AZ31 alloys are shown in Fig. 1. As shown in Fig. 1a, the microstructure of as-rolled AZ31 Mg alloy sheet exhibits a bimodal distribution of very fine recrystallized grains and coarse grains with twins inside. This kind of microstructure was produced by partial dynamical recrystallization during rolling of the coarse-grained ingot. Twinning is often found to accommodate plastic deformation in addition to slip in Mg alloys.

The degree of grain refinement and the extent of homogeneity of final microstructure are most noticeable in the material prepared by the ARB process. ARB processing at 300 °C results in the formation of a fine structure. The final grain size is reached after the first pass, possibly because dynamic recovery and recrystallization occur simultaneously, and a balance is struck between storage and removal of dislocations. However, the homogeneity of the processed microstructure increases with the number of passes. The microstructure became more homogeneous by successive breaking-up of coarse grains with further cycles. After three passes, reasonably homogeneous microstructure was obtained with fine grains of an average size 2.4 μm. As equiaxed grains are observed after each cycle of ARB, it is believed that dynamic recrystallization occurs during ARB. Concurrent dynamic recovery and some amount of recrystallization lead to the formation of nearly uniform fine microstructure. Recent studies of AZ31 suggested that dynamic recrystallization initiated at about 300 °C [11].

Deformation twins disappear in all ARBed materials as shown in Fig. 1b,c and d. Inhibition of deformation twinning is expected from increased twinning stress with finer grain size [12], and easier accommodation of shear strain on many available grain boundaries. Dislocation process and/or grain boundary sliding or shearing near grain boundaries accommodates plastic deformation.

Figure 2 shows a plot of the average grain size against the ARB cycle number. As shown in Fig. 2, initial grains (2–23 μm) were decreased into fine grains with average size of 2.4 μm after one cycle of ARB. However, the grain size shows no obvious variation during the subsequent cycles. It is noted that the grains of the ARBed AZ31 alloy are not so fine as those of the ARBed Al alloys (0.4–0.8 μm) when compared at the same ARB strain [13]. This is primarily due to the difference in ARB processing temperature. For Al alloys, the ARB temperature can be chosen to be considerably less than 200 °C [13] thanks to its ductile nature of FCC crystal structure. We tried ARB on the AZ31 alloys at 250 °C but failed in obtaining sound samples without surface flaws, being attributable to brittle nature of HCP crystal structure. The present rolling temperature is higher than the typical recrystallization temperature of Mg alloys. As show in Fig. 2, growth of the fine grain occurs during the heating between rolling cycles. The average size 4.58 μm of the heated ARBed sheet is reached

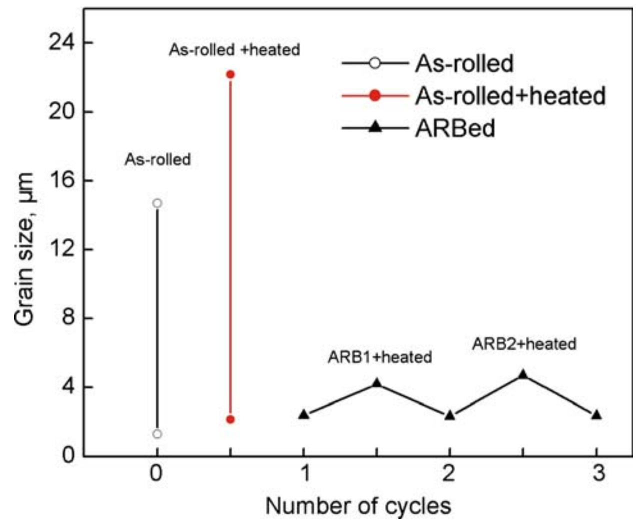

Fig. 2 The variation of the grain size of AZ31 Mg alloy during ARB processing (Heated at 300 °C × 10 min)

before the second ARB cycle. Therefore, the repeated heating between rolling cycles at temperature above the typical recrystallization temperature of Mg alloys would cause recrystallization and grain growth and partially cancel the accumulated strain.

Mechanical properties

The evolution of Vickers microhardness during ARB processing with respect to the number of ARB cycles is plotted in Fig. 3. The hardness of the AZ31 alloy decreased from 83.6 to 72.3 $HV_{0.5}$ after the first cycle, but during subsequent cycles it rose with the ARB cycle slightly to 80. Figure 3 also presents the effect of the annealing at

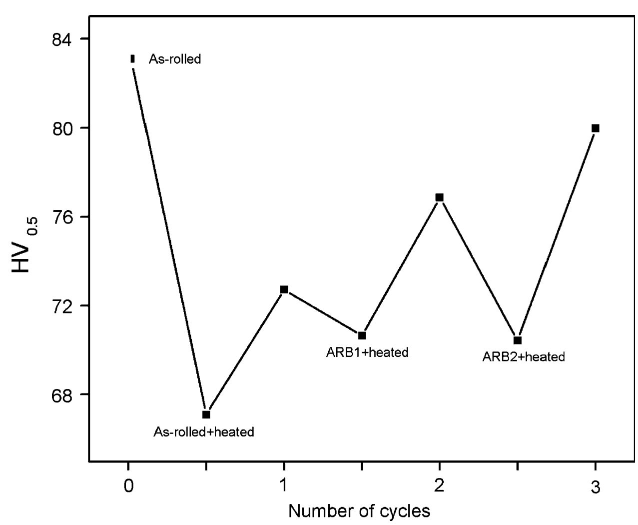

Fig. 3 Evolution of microhardness of AZ31 Mg alloy during ARB processing (Heated at 300 °C × 10 min)

300 °C × 10 min on the microhardness of ARB processed AZ31 alloy. It can be seen that the annealing resulted in a decrease of microhardness in some extent.

Figure 4a records the variation in the mechanical properties with the number of cycles for AZ31 after ARB at a temperature of 300 °C. For comparing purposes, data of the as-rolled AZ31 sheet are also included. This plot of engineering stress versus engineering strain was tested at room temperature at an initial strain rate of 5×10^{-3}/s. Compared with the stress-strain curve of the as-rolled material, the ultimate tensile strength (UTS) of the ARBed material after 1 ARB cycle decreases from 315 MPa to 303 MPa by 3.8%.The UTS of the ARBed AZ31 sheets increases with the ARB cycles. The UTS after three cycles is up to 315 MPa, equal to the UTS of the as-rolled material. It is noted from these curves that there is a significant increase in the ductility of AZ31 after ARB, since failure occurs at an elongation of only 4.27% in the as-rolled condition (labeled 1 in Fig. 4a). The best ductility is obtained after one cycle that is up to 25.1%, while slightly decreasing to 21.43% and 19.6% after two and three cycles, respectively. The results from these tests demonstrate conclusively that ARB is effective in improving the ductility of AZ31 Mg alloy at room temperature. The ductility is increased because the equiaxed and homogeneous grain of dynamic recrystallization contribute to the macroscopic deformation and the stress concentrations are accordingly reduced and spread over a wider area. The same two trends have been observed in the ECAPed AZ31 [14], AZ61 alloys [15] and attributed to the texture modification during ECAP, as shown in Fig. 4b.

The data for yield stress (YS), ultimate tensile strength (UTS), and elongation to failure are summarized in Table 1. There are two important findings. First, the yield stresses of the ARBed alloy are lower than the as-rolled material despite the considerable grain refinement during ARB. Second, tensile ductility is improved markedly after ARB.

The ARB process has been proven to be very effective in enhancing the strength of aluminum, steels and copper [6–8]. Changes in mechanical properties of the ARB processed 6061 and 1100 Al alloy sheets with total equivalent strain were researched by Lee et al [16]. The tensile strength of the ARB processed 6061 alloy increases with the total equivalent strain. The tensile strength of the specimen after eight cycles of the ARB is about three times of the initial value. However, the elongation is much lower than that of the initial value. This characteristic of ARBed materials, however, was not effective in Mg alloys despite the considerable grain refinement, as shown in Fig. 2. Tensile ductility of the ARBed AZ31 Mg alloys, on the other hand, has been remarkably improved after ARB. This outcome is in a sharp contrast with the report from other alloys such as Al, Fe and Cu alloys where considerable reduction in tensile elongation was obtained after ARB processing.

Table 1 Room temperature mechanical properties of as-rolled and ARBed AZ31 Mg alloy sheets

Condition	UTS (MPa)	YS (MPa)	Elongation (%)
As-rolled	315	291.2	4.27
One-cycled	303	225.9	25.1
Two-cycled	308	231.5	21.43
Three-cycled	315	236	19.6

However, there is a difficulty in conducting ARB on Mg alloys to improving the ductility and strength simultaneously. On the one hand, the restricted number of slip systems, and therefore the limited ductility, in HCP metals requires that the ARB is conducted at an elevated temperature. On the other hand, as shown in Fig. 2, grain growth occurs during the heating between rolling cycles when the rolling temperature is higher than the typical recrystallization temperature of Mg alloys so the accumulated strain is partially canceled.

Macrotexture

Figure 5 illustrates the evolution of texture of AZ31 Mg with respect to the number of ARB cycles. As shown in Fig. 5a, a strong basal texture occurred in the as-rolled

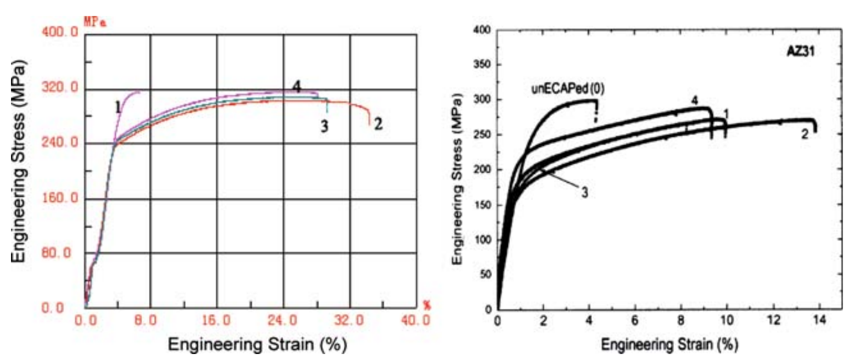

Fig. 4 The engineering stress–engineering strain relations for the (**a**) ARBed AZ31 Mg alloy sheets at different numbers N of ARB cycles:1-N = 0;2-N = 1;3-N = 2;4-N = 3 and (**b**) unECAPed and ECAPed AZ31 alloy (numbers indicate the pressing number)[14]

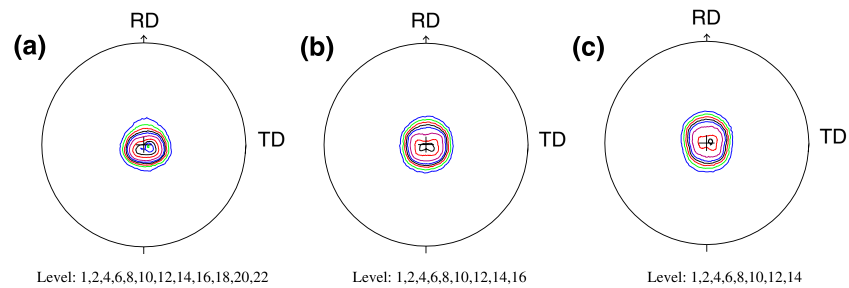

Fig. 5 (0 0 0 2) pole figures corresponding to AZ31 Mg alloy sheets: (**a**) as-rolled, (**b**) one-cycle ARBed, (**c**) three-cycle ARBed

AZ31 sheet. However, basal texture is weakened by ARB processing (Fig. 5b and c).

According to the literature [17, 18], a basal texture usually develops during rolling of HCP metal. With limiting the number of independent slip systems of HCP metal, twinning is an important deformation mechanism and twinning of the type {10–12} reorients the c-axis parallel to the compression axis. According to Ion et al. [18], the HCP material starts deforming by {10–12} twinning, which reorients the basal planes perpendicular to the compression axis. This orientation does not favor basal slip. Simultaneously, new small dynamic recrystallized grains form at the distorsion region in the vicinity of grain boundaries. The new recrystallized grains are formed with orientations more favorable for basal slip development.

As grain size gets finer, the operation of grain boundary sliding or shearing becomes increasingly easier so that plastic deformation in all directions can be accommodated. Grain rotation due to this effect can possibly allow the intensity of basal texture to decrease and the distribution of basal poles to become broader after maximum intensity is reached. Basal texture is weakened when grain size refines, possibly a result of accommodation of deformation by shearing along grain boundaries, and rotation of small recrystallized grains [19, 20].

Plastic deformation induced by a forming method like rolling, in general, significantly increases the strength (yield and maximum stress) owing to the presence of strong texture and obstacles of both dislocation and non-dislocation (e.g. twins, unstable grain boundaries) type. The notable improvement in the strength of AZ91 cast Mg alloys at room temperature after ARB processing has been reported by Perez-Prado et al [21], which was attributed to the simultaneous achievement of a very small grain size and the stabilization of a basal texture during severe rolling. However, the original materials in this study were as-rolled AZ31 alloy sheets with strong basal texture. The decreased strength of AZ31 sheets after ARB processing can be own to the weakened basal texture, the recovery and dynamic recrystallization during ARB and lack of twins.

Conclusions

Accumulative roll-bonding provides a simple and effective procedure for improving the ductility of AZ31 Mg alloy sheets at the ambient temperature. ARB processing refines the grain size even after a single cycle. The reduced grain sizes introduced by ARB in AZ31 Mg alloy are a consequence of dynamic recrystallization during the ARB process. This recrystallization leads to an equiaxed and homogeneous grain structure which contributes to a marked improvement in the room temperature ductility. The slightly decreased strength of AZ31 sheets after ARB processing can be own to the weakened basal texture, the recovery and dynamic recrystallization during ARB and the absence of twins.

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