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Improving mechanical properties of Mg-Al-Zn alloy sheets through accumulative roll-bonding

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Abstract: Experiments were conducted to evaluate the potential for improving the mechanical properties of Mg-Al-Zn alloy at room temperature by subjecting to accumulative roll-bonding(ARB). It is shown that ARB may be applied successfully to Mg-Al-Zn alloy at elevated temperatures and it leads to grain refinement and significant improvements in the ductility. The strength of the as-rolled Mg-Al-Zn alloy sheet after ARB processing is slightly decreased and basal texture is weakened by ARB processing.

Key words: Mg-Al-Zn alloy; accumulative roll-bonding; mechanical property; grain refinement

1 Introduction

Magnesium alloys are attractive materials due to their good specific properties[1]. However, magnesium alloys usually exhibit only limited ductility at ambient temperatures due to a lack of sufficient number of slip systems associated with hexagonal close-packed(HCP) crystal structure. If ductility could be improved to an extent that shape forming becomes possible near ambient temperature, large structural components of magnesium alloys could be produced at substantially lower cost and could be utilized for much wider applications, such as automobile parts.

Important efforts are devoted to improve the ductility of wrought magnesium alloys. Grain refinement could enhance strength and ductility of various metallic alloys at room temperature, and make alloys superplastic at elevated temperatures[2−4]. The process of severe plastic deformation(SPD) has been proved to be an effective approach for manufacturing large quantities of ultrafine grain metals[5]. Developing microstructures capable of undergoing superplastic deformation by severe plastic deformation(SPD) attracts increasing attention. Notable examples include[5−7]: severe plastic torsion straining(SPTS), multi-axis compression, accumulative roll bonding(ARB), and equal channel angular extrusion or pressing(ECAE/P).

The accumulative roll-bonding(ARB) is a relatively new method of severe plastic deformation proposed by SAITO et al[8]. Accumulative roll-bonding(ARB) involves severely deforming sheet metal without changing the original sheet dimensions. The advantage of ARB is that it is applicable to produce large bulk sheet materials. This process consists of repeating of cutting, stacking and rolling of sheets. To date, there has been no attempt to improve the room temperature mechanical properties of Mg and Mg-based alloys through ARB, although there are reports of grain refining after ARB of the Mg-Al-Zn alloys, where the grain size is reduced to about 3 μm[9−10].

The objectives of this work are to examine the potential for grain refinement in AZ31 Mg alloy by ARB and also to investigate the subsequent mechanical properties at room temperature.

2 Experimental

The material used in this study was as-rolled AZ31 sheets. The microstructure was characterized by coarse and equiaxed grains as well as some twins, as shown in Fig.1. In performing the accumulative roll bonding process, the sheets were rolled with a 50% reduction ratio (a von Mises strain of about 0.8 per ARB cycle).

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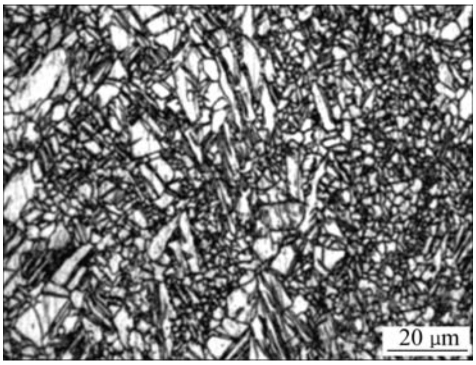

Fig.1 Optical microstructure of as-rolled AZ31

Then, the rolled sheet was cut, stacked to be the initial thickness and the stacked piece was rolled again with the same reduction ratio. Such a procedure was repeated for up to 3 cycles. ARB was attempted at 250, 300 and 400℃ but failed to produce the sound billets without surface or interior cracks at 250 ℃. Before rolling, the sheets were degreased with acetone followed by brushing the surface to remove oxide film. To ensure a firm contact between sheets and to prevent sheets from slipping against each other during rolling, two sheets (200 mm×75 mm×1.5 mm) were bound strongly by wires at the end of the sheets. Rolling was performed at the rolling machine furnished with two 400 mm diameter rolls that rotate at 23 r/min. Before each cycle, the samples were heated at the preset temperature for 10 min. The rolls were neither lubricated nor heated.

The initial material and the deformed microstructures were examined by optical microscopy. Average grain size (*d*) was calculated from the optical micrographs by the linear intercept method. Macrotexture measurements were performed in the as-rolled and ARBed samples. X-ray texture analysis was performed in a Philips APD-10 diffractometer. Calculated pole figures were obtained using the measured incomplete $\{10\bar{1}0\}$, {0002}, $\{10\bar{1}1\}$, $\{10\bar{1}2\}$ pole figures.

Microhardness and tensile tests were performed to evaluate the effect of ARB on the strength and ductility of the AZ31 Mg alloys. Vickers microhardness(Hv) was measured on the plane perpendicular to the longitudinal axis by imposing a load of 5 N for 10 s. The values reported for Hv represent the average of seven separate measurements taken at randomly selected points. Tensile specimens with 8.4 mm in gauge length, 3.2 mm in width, 3.2 mm in shoulder radius and the gauge length parallel to the longitudinal axis were cut from the ARBed billets. Tensile testing was carried out at room temperature under a constant cross-head speed (2.5 mm/min) on a tensile testing machine (Instron 8516).

3 Results and discussion

Typical microstructures of the AZ31 Mg alloy after ARB at different temperatures of 300 ℃ and 400 ℃ for three cycles are shown in Fig.2. The microstructures after three cycles exhibit little changes. The grains are reasonably equiaxed and homogeneously distributed for each rolling temperature, which suggests that recrystallization occurs during ARB. Their mean grain sizes, *d*, are determined to be 3.16 μm and 3.45 μm after rolling at 300 ℃ and 400 ℃, respectively. The roll-bonding in ARB process has been usually carried out without lubrication. It has been known that large amount of redundant shear strain is applied at subsurface regions of the sheets in the rolling under the unlubricated conditions[11]. When the 50% rolled sheet is cut and stacked between cycles, one of the surface that has undergone the severe shear deformation comes into the center. As a result, the sheared regions do not localize only at the subsurface layers but complicatedly distribute through thickness of the sheet as the ARB cycle proceeds, which may also contribute to the formation of the fine grains[6].

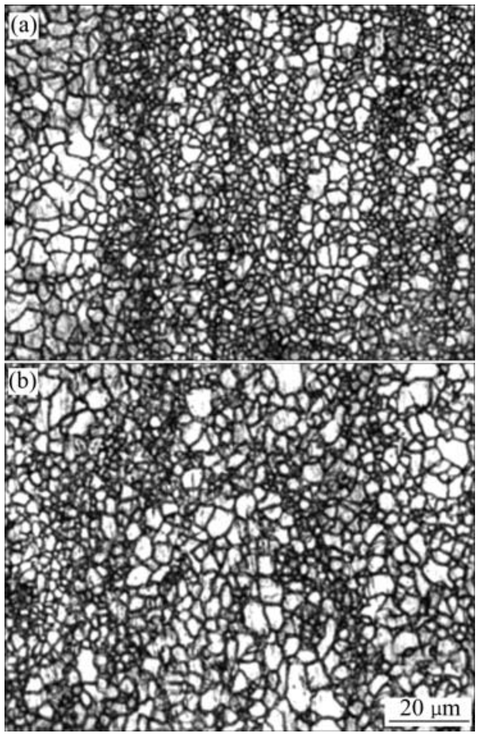

Fig.2 Typical microstructures of AZ31 after ARB for three cycles at different temperatures: (a) 300 ℃; (b) 400 ℃

It is noted that the grains of the ARBed AZ31 alloy are comparable with those of the ECAPed AZ31 alloys (as listed in Table 1)[12]. These measurements show, therefore, that ARB is effective in reducing the grain size of this Mg-based alloy. It is also apparent that ARB is especially effective in reducing the grain size when the rolling is conducted at lower temperatures, where the occurrence of grain growth is limited. The inherent brittleness in magnesium is a consequence of the limited number of slip systems in HCP metals and a failure to fulfill the von Mises criterion of five independent slip systems for homogeneous polycrystalline deformation. Thus, there exists stress concentration at selected points within these materials during rolling and, if these stress concentration is not dissipated through rolling at high temperature, it leads to a brittleness that is especially acute when the grain size is large. In the present investigation, brittleness of the unrolled samples precludes the rolling of AZ31 Mg alloy below a temperature of about 300 ℃.

Table 1 Average grain size of ECAPed[13] and ARBed AZ31 alloys

Condition	Pass	Processing temperature/℃	Strain	Grain size/μm
ECAPed	1	320	1	8.1
	2	320	2	6.3
	3	250	3	4.3
	4	200	4	2.5
ARBed	3	300	2.4	3.16
	3	400	2.4	3.45

The degree of grain refinement and the extent of homogeneity of final microstructure are two aspects that are most noticeable in the material prepared by the ARB process. As shown in Fig.2, grain size after ARB is fine and homogeneous when deformation twinning is inhibited, possibly because dynamic recovery and recrystallization occur simultaneously, and a balance is struck between storage and removal of dislocations. Absence of twins in the ARBed AZ31 alloy may be also owing to its relatively high ARB temperature. It is also noted that the grains of ARBed AZ31 alloy are almost equiaxed but elongated and lamellar grains are produced in ARB of Al and IF steel[13−14]. This may be owing to the fact that the recrystallization temperature of magnesium alloys is lower than the present deformation temperature. Thus the repeated heating between rolling cycle at temperature above the typical recrystallization temperature of Mg alloys would cause recrystallization and partially cancel the accumulated strain, resulting in a constant grain size after two cycles.

Fig.3 shows the variation in the mechanical properties at room temperature of AZ31 Mg alloy ARBed at temperature of 300 ℃ and 400 ℃, respectively. For comparing purposes, data of the as-rolled AZ31 sheet are also included. The data for yield stress(σ_y), ultimate tensile strength(σ_b), and elongation to failure are summarized in Table 2.

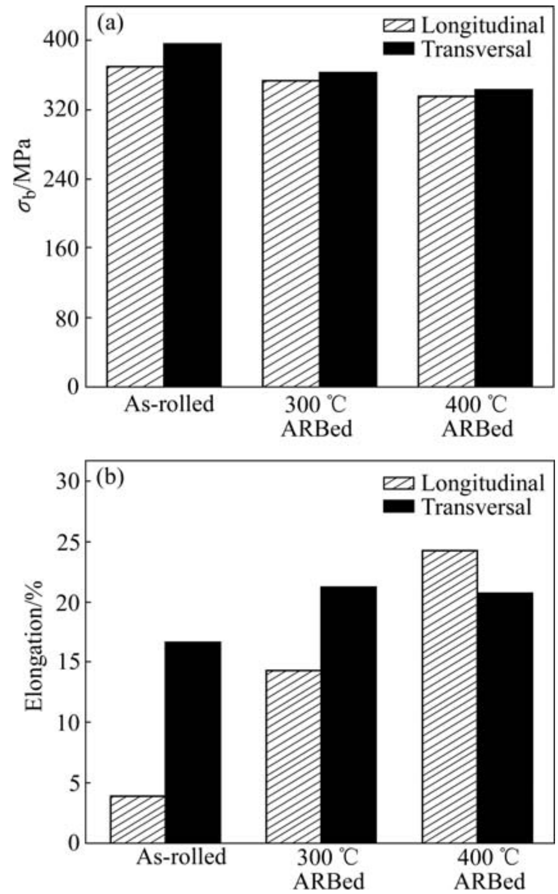

Fig.3 Mechanical properties of AZ31 after being ARBed at 300℃ and 400 ℃: (a) UTS; (b) Elongation

Table 2 Mechanical properties of unARBed and ARBed AZ31 Mg alloy sheets at room temperature

Condition	σ_b/MPa	σ_y/MPa	Elongation/%
As-rolled, longitudinal	369.3	320.3	4.1
As-rolled, transversal	395.2	337.5	17.2
300℃ ARBed, longitudinal	350.8	287.5	15.5
300℃ ARBed, transversal	362.0	315.1	24.8
400℃ ARBed, longitudinal	332.0	275.0	25.0
400℃ ARBed, transversal	342.0	282.4	26.2

There are three important findings. First, the yield stresses of the ARBed alloy are lower than those of the as-rolled material despite the considerable grain refinement during ARB. Second, there is a significant increase in the ductility of AZ31 after ARB. Third, the ultimate tensile strength of the ARBed AZ31 sheets decreases and the ductility increases with the rolling temperature.

Fig.4 illustrates the evolution of texture of AZ31 Mg after ARB processing. As shown in Fig.4(a), a strong basal texture occurs in the as-rolled AZ31 sheet. However, basal texture is weakened by ARB processing (Fig.4(b)). According to the Ref.[15], a basal texture usually develops during rolling of HCP metal by twinning of the type $\{10\bar{1}2\}$ which reorients the basal planes perpendicular to the compression axis.

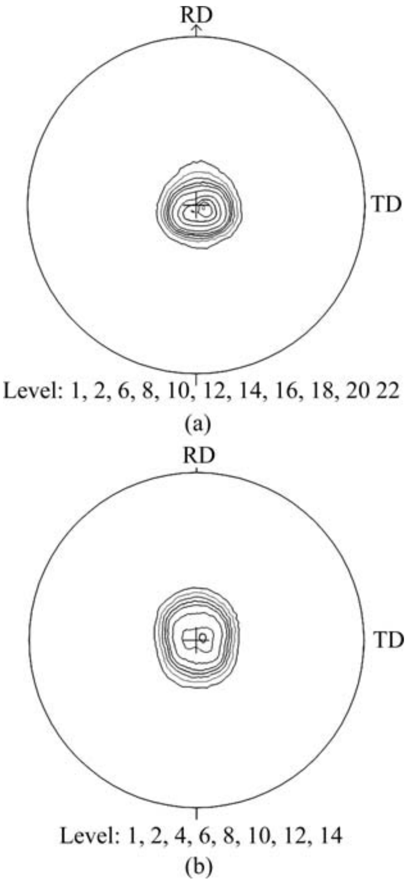

Fig.4 (0002) pole figures corresponding to AZ31 Mg alloy sheets: (a) As-rolled; (b) Three-cycle ARBed at 300 ℃

As shown in Fig.2, new small dynamic recrystallized grains form at the distortion region in the vicinity of grain boundaries during ARB processing due to the severe deformation. 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. Basal texture is weakened when grain size is refined, possibly as a result of accommodation of deformation by shearing along grain boundaries, and rotation of small recrystallized grains[16].

Therefore, the decreased strength of AZ31 sheets after ARB processing can be owing to the weakened basal texture, the recovery and dynamic recrystallization during ARB and lack of twins. At the same time, the new fine and recrystallized grains with orientations more favorable for basal slip contribute to the improvement of ductility.

Fig.5 shows a plot of the average grain size, *d*, against the annealing time of the ARBed AZ31 sheets. Relatively small grain growth is observed below 250 ℃ even for 240 min, while marked grain growth is observed at 400 ℃ even for only 5 min. This result indicates that the fine-grained microstructure produced by the current ARB technique is stable below 250 ℃ while becomes unstable above 300 ℃ even for 10 min. The microstructures of the annealed materials are shown in Fig.6. The average grain size measured in the AZ31 Mg sheet ARBed at 300 ℃ after the three cycles is 3.16 μm, while the average grain sizes are 2.96 μm, 4.58 μm and 4.91 μm after annealing at 250 ℃ for 240 min, 300 ℃ for 10 min and 400 ℃ for 5 min, respectively. It is noted that the average grain size after annealing at 250 ℃ for 240 min is smaller slightly than that of the as-ARBed one. Such decrease in grain size can be due to the formation of fine grains as a result of static recrystallization during annealing.

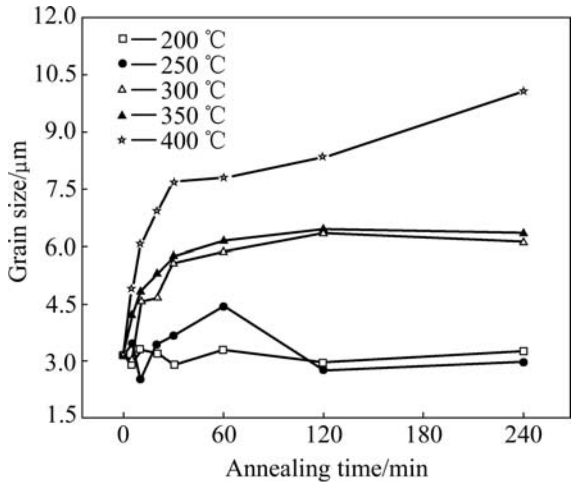

Fig.5 Grain size versus annealing time at different annealing temperatures of AZ31 after ARB at 300 ℃

Fig.7 shows the variation in the microhardness with the annealing time at different temperatures for AZ31 after ARB. The Hv value continues to decrease with increasing temperature. This trend is consistent with the grain-size increase with increasing temperature (Fig.5). It is concluded from Fig.7 that the hardness decreases sharply during the early stage at all annealing temperature. This effect diminishes with increasing annealing time and the decrease in hardness is not significant by further prolonging the duration after annealing for 30 min at all temperatures.

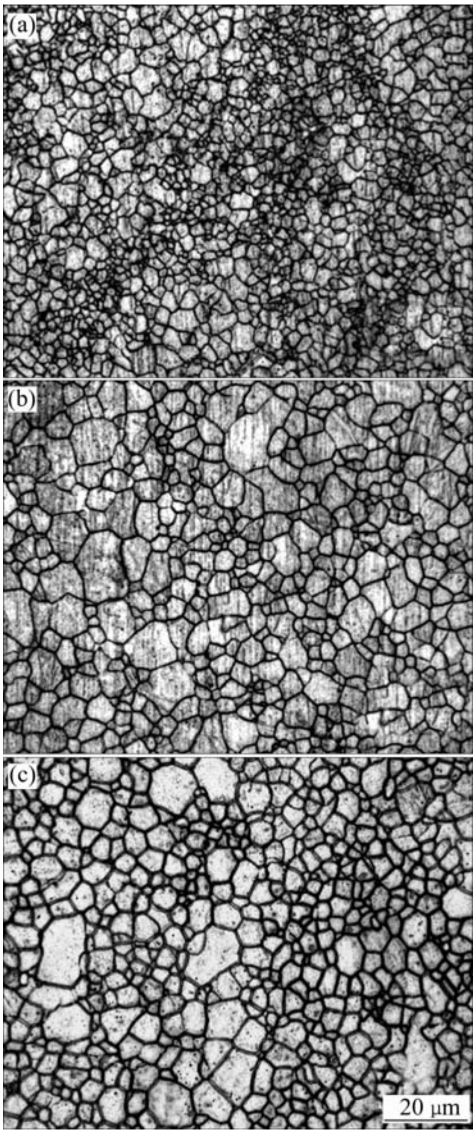

Fig.6 Microstructures of ARBed AZ31 sheets after annealing under different conditions: (a) 250 ℃, 240 min; (b) 300 ℃, 10 min; (c) 400 ℃, 5 min

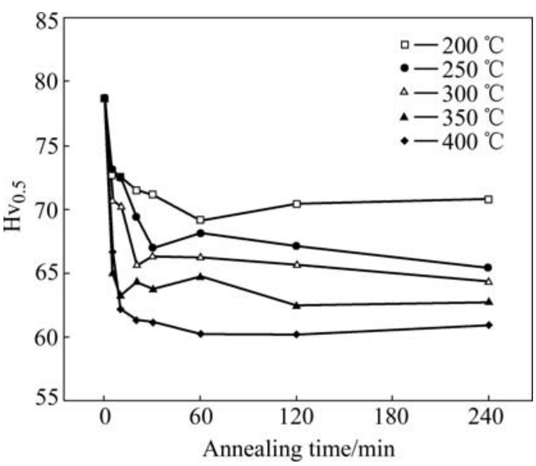

Fig.7 Variation in microhardness with annealing time at different temperatures of AZ31 after being ARBed at 300 ℃

Significant hardening during intensive plastic deformation is characterized by accumulation of energy in the form of defects that can be released by recovery and recrystallization during deforming and subsequent annealing[17−18]. The stored strain energy can be lowered, in general, by recovery and/or recrystallization during annealing, which results in the decrease of hardness.

Fig.8 shows the relation of $Hv_{0.5}$ against $d^{-1/2}$ of the annealed ARBed AZ31 alloys after being annealed under different conditions.

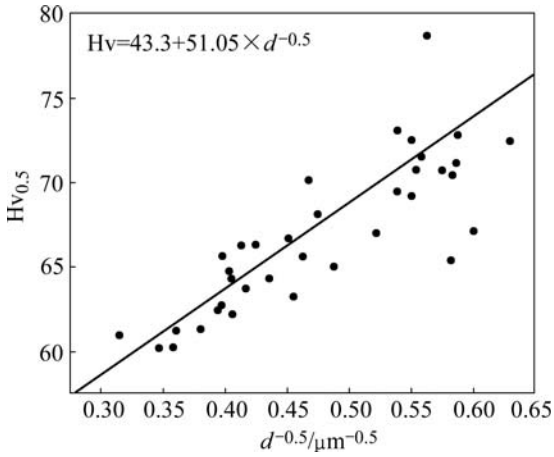

Fig.8 Values of $Hv_{0.5}$ versus reciprocal of square root of grain size for all annealing conditions

It can be concluded that the relationship between hardness and grain size can be expressed as the following Hall-Petch relationship:

$$Hv=H_0+K_H d^{-1/2} \tag{1}$$

where H_0 and K_H are the material constants. Most of the datum points lie on a single straight line depicted by Eqn.(1) with H_0=43.3 and K_H=51.05.

The ductility of the ARBed AZ31 alloy could be enhanced after annealing, which was accompanied by a decrease in the yield strength and ultimate tensile strength(UTS). As for the material ARBed at 300 ℃ for three cycles, the average ultimate strength decreases to 303 MPa, but the average elongation to failure increases to 29.3% when being annealed at 300 ℃ for 120 min. Compared with a commercially available AZ31H sheet (hard rolled, σ_y=220 MPa, σ_s=290 MPa, δ=15%)[19], it is recognized that the application of ARB plus subsequent annealing makes it possible to enhance ductility significantly while keeping the strength similar to that of the commercial AZ31H sheet.

4 Conclusions

1) ARB is a simple and effective procedure for improving the ductility of AZ31 Mg alloy sheet at

ambient temperatures.

2) Grain sizes of the as-rolled sheet are refined to 3.16 μm and 3.45 μm after ARB at 300 ℃ and 400 ℃ for three cycles, respectively, due to the occurrence of recrystallization during the rolling process, which leads to an equiaxed and homogeneous grain structure.

3) Slight decrease of strength of AZ31 sheets after ARB processing can be ascribed to the weakened basal texture, the recovery and dynamic recrystallization during ARB and lack of twins.

4) The best compromise between strength and ductility is obtained after annealing at 300 ℃ for 120 min. In this case, the average ultimate tensile strength is 303 MPa and average tensile elongation is 29.3%.

References

[1] MORDIKE B L, EBERT T. Magnesium properties—applications—potential [J]. Mater Sci Eng, 2001, A302: 37−45.

[2] KUBOTA K, MABUCHI M, HIGASHI K. Processing and mechanical properties of fine-grained magnesium alloys [J]. J Mater Sci, 1999, 34: 2255−2262.

[3] SERGUEEVA AV, STOLYAROV V V, VALIEV R Z, MUKHERJEE A K. Superplastic behaviour of ultrafine-grained Ti-6A1-4V alloys [J]. Materials Science and Engineering, 2002, A323: 318−325.

[4] MATSUBARA K, MIYAHARA Y, HORITA Z, LANGDON T G. Developing superplasticity in a magnesium alloy through a combination of extrusion and ECAP [J]. Acta Mater, 2003, 51: 3073−3084.

[5] VALIEV R Z, ISLAMGALIEV R K, ALEXANDROV IV. Bulk nanostructured materials from severe plastic deformation [J]. Progress in Materials Science, 2000, 45: 103−189.

[6] TSUJI N, SAITO Y, LEE S H, MINAMINO Y. ARB (Accumulative Roll-Bonding) and other new techniques to produce bulk ultrafine grained materials [J]. Advanced Engineering Materials, 2003, 5(5): 338−344.

[7] YAMASHITA A, HORITA Z, TERENCE G L. Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation [J]. Materials Science and Engineering, 2001, A300: 142−147.

[8] SAITO Y, UTSUNOMIYA H, TSUJI N, SAKAI T. Novel ultra-high straining process for bulk materials development of the accumulative roll-bonding(ARB) process [J]. Acta Mater, 1999, 47(2): 579−583.

[9] PE′REZ-PRADO M T, DEL VALLE J A, RUANO O A. Grain refinement of Mg-Al-Zn alloys via accumulative roll bonding [J]. Scripta Materialia, 2004, 51: 1093−1097.

[10] DEL VALLE J A, PE′REZ-PRADO M T, RUANO O A J A. Accumulative roll bonding of a Mg-based AZ61 alloy [J]. Materials Science and Engineering, 2005, A 410-411: 353.357.

[11] LEE S H, SAITO Y, TSUJI N, UTSUNOMIYA H, SAKAI T. Role of shear strain in ultragrain refinement by accumulative roll-bonding (ARB) process [J]. Scripta Materialia, 2002, 46: 281−285.

[12] KIM H K, KIM W J. Microstructural instability and strength of an AZ31 Mg alloy after severe plastic deformation [J]. Materials Science and Engineering, 2004, A385: 300−308.

[13] TSUJI N, UEJI R, MINAMINO Y. Nanoscale crystallographic analysis of ultrafine grained IF steel fabricated by ARB process [J]. Scripta Materialia, 2002, 47: 69−76.

[14] LEE S H, SAITO Y, SAKAI T, UTSUNOMIYA H. Microstructures and mechanical properties of 6061 aluminum alloy processed by accumulative roll-bonding [J]. Materials Science and Engineering, 2002, A325: 228−235.

[15] AGNEW S R, YOO M H, TOME C N. Application of texture simulation to understanding mechanical behavior of Mg and solid solution alloys containing Li or Y [J]. Acta Mater, 2001, 49: 4277−4289.

[16] YANG Q, GHOSH A K. Production of ultrafine-grain microstructure in Mg alloy by alternate biaxial reverse corrugation [J]. Acta Materialia, 2006, 54: 5147−5158.

[17] MYSHLYAEV M M, MCQUEEN H J, MWEMBELA A, KONOPLEVA E. Twinning, dynamic recovery and recrystallization in hot worked Mg-Al-Zn alloy [J]. Materials Science and Engineering, 2002, A 337: 121−133.

[18] PE′REZ-PRADO M T, RUANO O A. Texture evolution during annealing of magnesium AZ31 alloy [J]. Scripta Mater, 2002, 46: 149−155.

[19] KIM W J, LEE J B, KIM W Y, JEONG H T, JEONG H G. Microstructure and mechanical properties of Mg-Al-Zn alloy sheets severely deformed by asymmetrical rolling [J]. Scripta Materialia, 2007, 56(4): 309−312.

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