

Effect of heat treatment on microstructures and mechanical properties of extruded-rolled AZ31 Mg alloys

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Abstract: The deformation behaviors of extruded-rolled (ER) AZ31 Mg alloys with different rolling reduction and heat treatment were investigated. The results show that the accumulation of rolling reduction increases the density of twins, and refines the grain structures, which are in accordance with the enhanced strength and degraded plasticity. Tensile strength and plasticity of the alloy depend mainly on rolling reduction, while heat treatment temperature plays a more important role than heat treatment time at the same rolling reduction. With the increase of rolling reduction, the plasticity becomes more sensitive than strength on heat treatment. Recrystallization of extruded-rolled alloys will occur easily with deformation increasing, which is induced by addition of distortion energy.

Key words: AZ31; orthogonal testing; rolling; mechanical properties; heat treatment

1 Introduction

Magnesium alloy is the lightest metal that can be employed for structural use. However, their applications are severely restricted because of their poor formability inherited by limited slip systems at the room temperature. Wrought Mg alloys, which are mainly obtained by extrusion, rolling and forging, usually present excellent combination of properties. Processing techniques such as equal-channel angular pressing (ECAP)[1–2], high-ratio extrusion[3–4], large strain hot rolling(LSHR)[5–6], accumulative roll bonding(ARB)[7–8] and high-pressure torsion (HPT) provide a capability for achieving fine grain and corresponding ways of enhancing mechanical properties. Among the cited techniques, rolling is the most utilized one for fabricating large bulk sheet or plate samples since it has already been extensively applied to Fe and Al alloys. Lots of studies have been carried out on the mechanical properties, texture evolution and super-plasticity of rolled Mg alloys at room and elevated temperatures[9–17]. The effect of rolling speed, rolling direction and rolling mode are also investigated by some researcher[16, 18–20]. MATSUBARA et al[21] and LIN

et al[22] applied a new processing procedure which involves extrusion and ECAP to Mg alloy. However, reports of processing procedure which involves extrusion and rolling are far from enough. In this work, different rolling reduction, annealing temperature and time are adopted to obtain extruded-rolled AZ31 alloys. The effects of proposed processing technique on mechanical properties and microstructures are investigated in details.

2 Experimental

As-cast AZ31 ingot was extruded at 653 K, and a final product of 18 mm×100 mm sheet was obtained. Then, sheet was rolled in the air after holding for 1 h at 623 K. The rolling reduction in the first pass is 20%, and then it is reduced to 10% in subsequent pass. The holding time is 15 min between every two passes and the holding temperature is 623 K. The thickness of sheet was reduced by rolling from 18 mm to 9 mm, 5 mm and 2.5 mm, and total rolling reduction is 50%, 72% and 86% (which are expressed as ER1 alloy, ER2 alloy and ER3 alloy, respectively). The tensile tests were conducted on a MTS 858 Mini Bionix universal testing machine with a constant gripping head speed of 1 mm/min at room

temperature in laboratory. The tensile direction is rolling direction or extruded direction. The tensile samples had a reduced section of 16 mm×2 mm×2 mm. The grain structure was revealed by mechanical polishing and subsequently etching by means of acetic-picral acid. SEM observations were conducted on XL30-FEG environmental scanning electron microscope.

3 Results and discussion

3.1 Microstructures of alloys

The microstructures of four alloys are shown in Fig.1. It is obvious that recrystallization has taken place after extrusion in ER1–ER3 alloys, which refines grain sizes of alloys. The equiaxial grain size of as-extruded alloy is 50 μm by linear intercept method, while those of ER1–ER3 alloys are 20 μm , 15 μm and 5 μm , respectively. The grain size of ER2 alloy more slightly decreases than that of ER1 alloy but deformation twins increase greatly, and the grain size of ER3 alloy drops to 5 μm with many deformation-induced twins.

3.2 Mechanical properties and deformation behavior of alloys

The room temperature tensile properties of ER alloys are shown in Table 1, and tensile direction is rolling direction. For comparing, mechanical properties of the as-extruded alloy (E0 alloy) were also measured. It is noticed that strength of ER alloys is higher than that of E0 alloy, while the elongation of ER alloys is lower than that of E0 alloy. The strength of ER alloys increases and the plasticity decreases with the addition of rolling reduction. The change of mechanical properties attributes

to alteration of microstructure, which involves grain refining, twins accumulation and formation of basal plane texture. According to Hall-Petch equation, the strength and ductility will increase with decrease of grain size of alloys. At the same time, it is well known that basal plane texture will appear when Mg alloys are rolled or extruded[11, 23], which will worsen the deformation of alloys in a certain orientation because of grains rearrangement. PYZALLA et al[24] found that the basal plane texture that gets more intensive with the increase of rolling reduction will impart more difficulty at rolling. It is induced that more twins appear to accommodate deformation, while the increase of twins will impede dislocations sliding. So, the texture and twins are the main reasons of decline of elongation though the grain sizes of ER alloys are refined obviously.

Table 1 Mechanical properties of E0 and ER1–ER3 alloys at room temperature

Alloy	Tensile strength/ MPa	Yield strength/ MPa	Elongation/%
E0	227	129	18.05
ER1	246	190	12.06
ER2	260	214	11.5
ER3	285	256	6.23

The SEM microscopy shows the surface of deformation to fracture of ER1–ER3 alloy (Fig.2). Figs.2(a) and (b) depict that there are few twins in ER1 alloy, and slip line can go through the whole recrystallized grain and stop at grain boundaries and twins. A few slip lines can cross grain boundary, and the

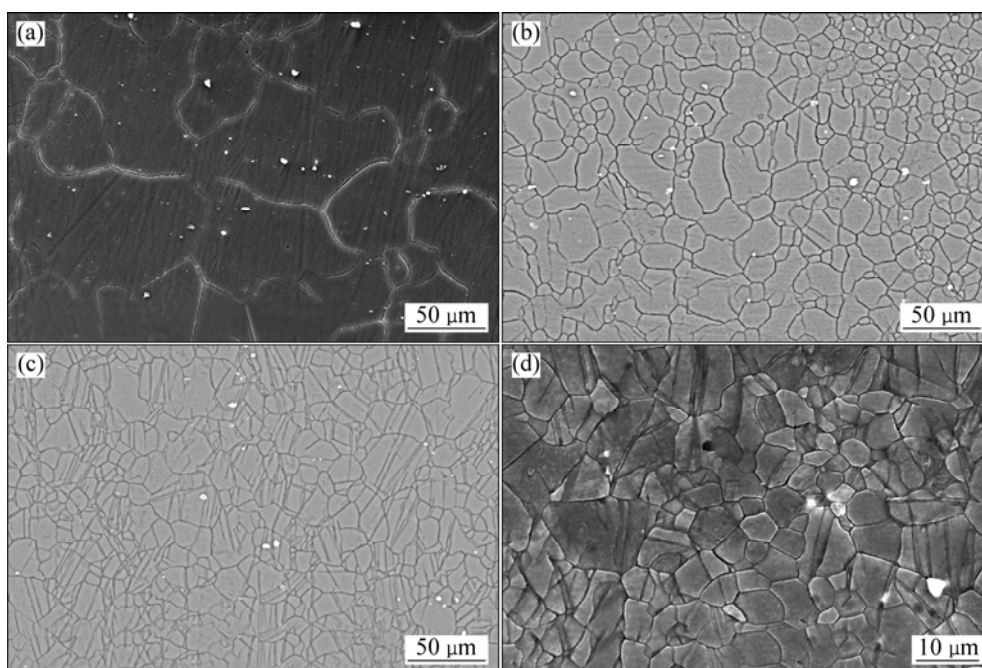


Fig.1 Microstructures of alloys: (a) As-extruded alloy; (b) ER1; (c) ER2; (d) ER3

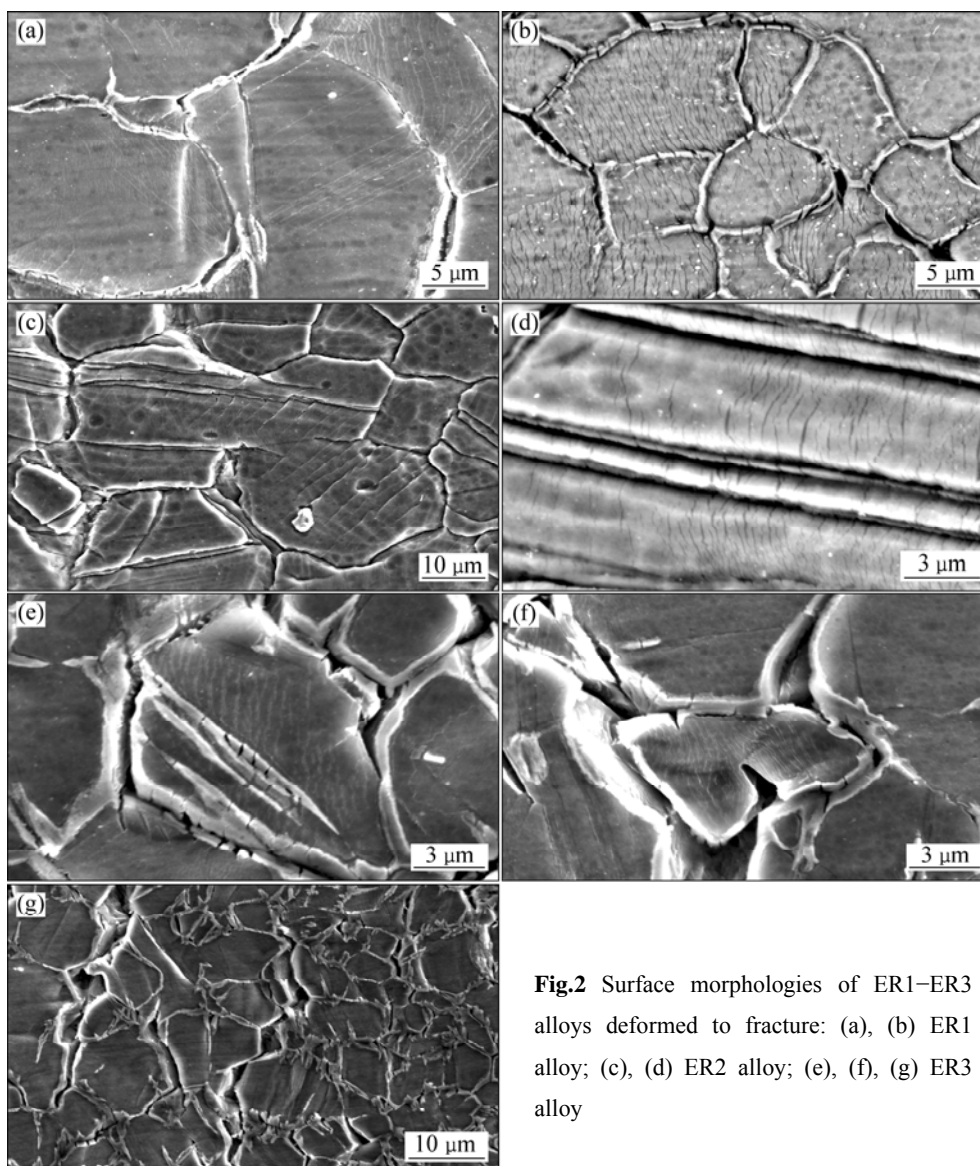


Fig.2 Surface morphologies of ER1–ER3 alloys deformed to fracture: (a), (b) ER1 alloy; (c), (d) ER2 alloy; (e), (f), (g) ER3 alloy

crack initiates at grain boundary. The yield strength is improved obviously because of grain refinement and basal texture. The density of twins rises when rolling reduction is 72%, which will impede slipping and most of slip line cannot cross the twins. Therefore, twin boundary will be the site that crack initiated at (Fig.2(c) and (d)), which will decrease the plasticity of alloy. The grain size gets smaller when rolling reduction is 86%. The basal plane texture will be more intensive with the increase of rolling reduction. Fig.2(e) shows that slipping is impeded by twins inside grain and stops at twin boundaries in ER3 alloy. The crack initiates mainly at grain boundary, while twin boundary may be also crack initiation site as shown in Figs.2(f) and (g).

3.3 Microstructures after heat treatment and orthogonal testing analysis

A set of orthogonal tests were performed by taking the effect of rolling reduction, heat treatment time and

heat treatment temperature on strength and elongation into consideration. Because the recrystallization temperature of Mg alloy is about 423 K, three different heat treatment temperatures are chosen. Room temperature tensile properties of alloys after heat treatment are listed in Table 2, and the orthogonal tests conditions are listed in Table 3.

After heat treatment, it is noticed that the grain size of ER1 alloys maintains around 20 μm and microstructures are almost as same as those after rolling as shown in Fig.3. The microstructure implies that recrystallization process has been finished after rolling, and there is no recrystallization during heat treatment process. The strength and elongation change slightly with the increases of temperature and time. The microstructure of ER2 alloys annealed at 373 K changes slightly. The strength and elongation almost have no change. When ER2 alloy is annealed at 423 K, there are a few recrystallization grains at original grains

Table 2 Room temperature mechanical properties of alloys

Thickness/mm	Rolling reduction/%	Treatment	Tensile strength/MPa	Yield strength/MPa	Elongation/%
18	—	As-extruded	227	129	18.05
9	50	As-rolled	246	190	12.06
		373 K, 0.5 h	248	202	11.65
		423 K, 1 h	246	199	12.02
		473 K, 2 h	245	195	12.20
5	72	As-rolled	260	214	11.50
		423 K, 0.5 h	254	214	9.80
		473 K, 1 h	253	201	13.55
2.5	86	373 K, 2 h	260	216	11.00
		As-rolled	285	256	6.23
		473 K, 0.5 h	265	190	14.90
		373 K, 1 h	288	233	8.60
		423 K, 2 h	283	223	12.05

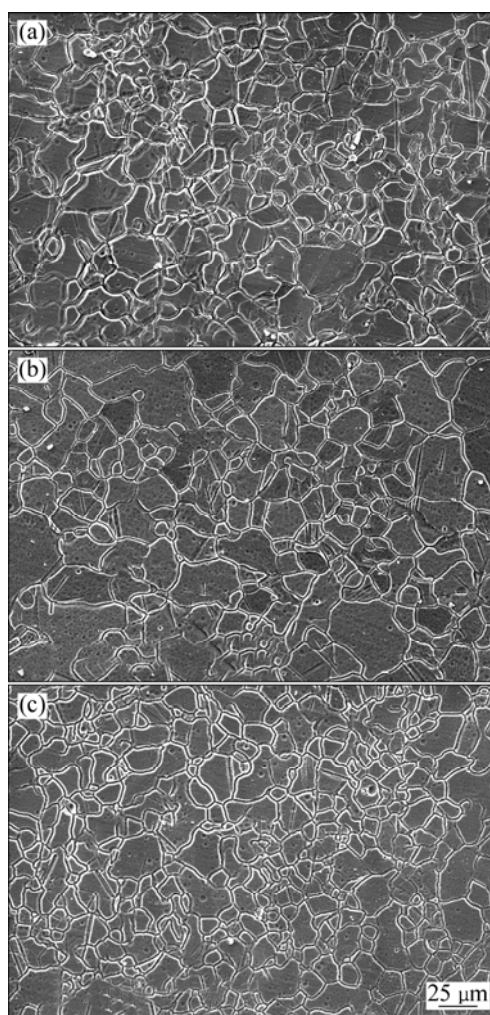
Table 3 Test conditions for orthogonal test

No.	Rolling Reduction/%	Heat treatment time/h	Heat treatment temperature/K
1	50	0.5	373
2	50	1.0	423
3	50	2.0	473
4	72	0.5	423
5	72	1.0	473
6	72	2.0	373
7	86	0.5	473
8	86	1.0	373
9	86	2.0	423

boundaries. This shows that recrystallization process starts and the strength and elongation also change slightly. When ER2 alloy is annealed at 473 K, recrystallization process finishes and twins disappear almost completely as shown in Fig.4(c). The strength, especially yield strength decreases and elongation increases with the increase of temperature. ER3 alloy is recovered after holding at 373 K as shown in Fig.5(a), and the yield strength drops and elongation rises while tensile strength almost does not change. It can be found that recrystallization grains appear at the original grain boundaries after annealing at 423 K as shown in Fig.5(b), and the yield strength gets worse but elongation gets better compared with those of alloy annealed at 373 K. The microstructure of ER3 alloy annealed at 473 K shows that static recrystallization process has been finished, so the strength decreases and elongation increases remarkably.

The results of the orthogonal testing are shown in Fig.6 and Table 2. From Fig.6, it can be found that the extent of rolling reduction contributes much to tensile strength, while heat treatment temperature plays a more prominent role in elongation. The posterior and lowest

factor are annealing temperature and annealing time for tensile strength, heat treatment time and rolling reduction for elongation, respectively. Tensile strength and plasticity of the alloy depend mainly on rolling reduction,

**Fig.3** Microstructures of ER1 alloy after annealing: (a) 373 K, 0.5 h; (b) 423 K, 1 h; (c) 473 K, 2 h

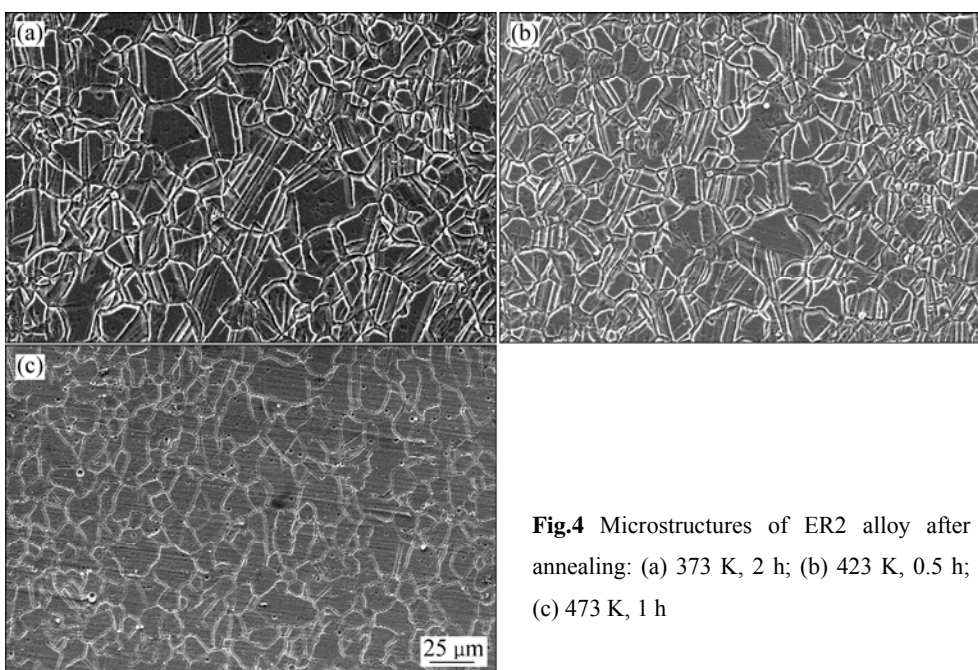


Fig.4 Microstructures of ER2 alloy after annealing: (a) 373 K, 2 h; (b) 423 K, 0.5 h; (c) 473 K, 1 h

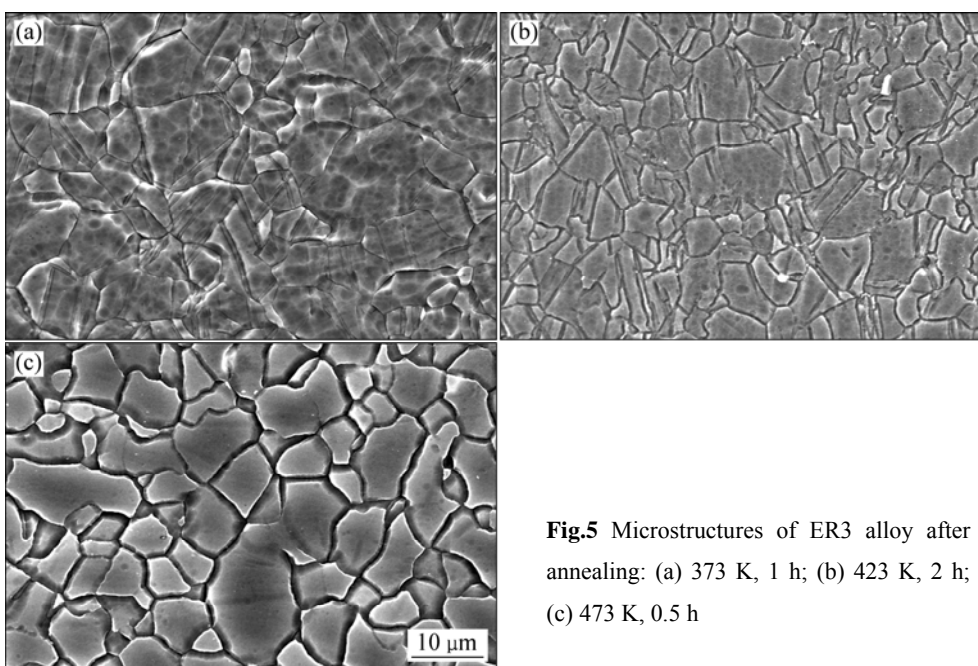


Fig.5 Microstructures of ER3 alloy after annealing: (a) 373 K, 1 h; (b) 423 K, 2 h; (c) 473 K, 0.5 h

while heat treatment temperature plays more important role than heat treatment time at the same rolling reduction. With the increase of rolling reduction, the plasticity becomes more sensitive than strength on heat treatment. Dynamic recrystallization occurs when Mg alloys deform at high temperature. Distortion energy accumulated in rolling process is the driving force for static recrystallization. It can be seen that the static recrystallization process of ER1 alloy has finished after rolling cooled in air. So, there is not enough unreleased distortion energy to drive recrystallization when ER1 alloy is annealed. However, there is still enough unreleased distortion energy in ER2 and ER3 alloys

because they are too thin to radiate when they are cooled in air. More distortion energy is accumulated in the alloys by increasing of rolling reduction. With the addition of distortion energy, lower holding temperature will result in recrystallization as shown in Fig.5. ER2 alloy appears recrystallization grains annealed at recrystallization temperature and recrystallization process finishes soon at higher temperature. Because of more distortion energy, ER3 alloy even begins to recrystallize at 373 K. Similar results that recrystallization has taken place at warm temperature, even at room temperature, have been reported[9, 25]. The mechanical properties data of orthogonal testing

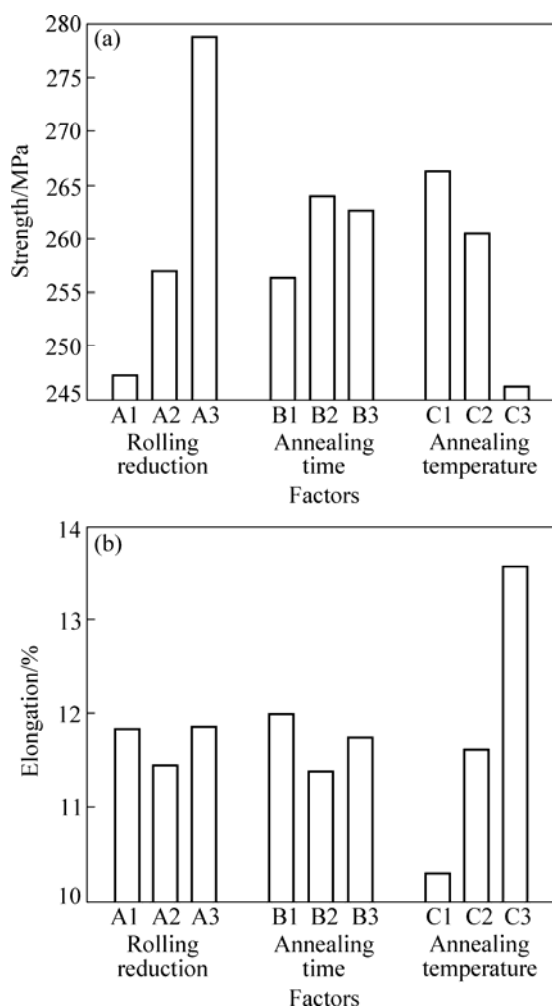


Fig.6 Variation of tensile strength (a) and elongation (b) with rolling reduction, heat treatment time and heat treatment temperature

coincide with the change of microstructures of alloys.

4 Conclusion

1) The heat treatment of extruded-rolled AZ31 Mg alloy is carried out at different temperatures for different time. The strength of ER alloys is higher than that of as-extruded alloy, while the elongation of ER alloy is lower than that of as-extruded alloy. The addition of rolling reduction increases the amount of twins and refines the grain, which are in accordance with the strength increasing and the plasticity decreasing.

2) The orthogonal testing results show that the largest factor of affecting tensile strength is rolling reduction and the largest one on elongation is heat treatment temperature. Recrystallization of ER alloys will become easy with the increase of deformation, which is the result of addition of distortion energy. When there is enough distortion energy, the ER3 alloys begin to recrystallize at 373 K which is lower than

recrystallization temperature.

References

- [1] YAMASHITA A, HORITA Z, LANGDON T G. Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation [J]. *Mater Sci Eng A*, 2001, 300(1/2): 142–147.
- [2] FENG X M, AI T T. Microstructure evolution and mechanical behavior of AZ31 Mg alloy processed by equal-channel angular pressing [J]. *Trans Nonferrous Met Soc China*, 2009, 19(2): 293–298.
- [3] LIN H K, HUANG J C. High strain rate and/or low temperature super plasticity in AZ31 Mg alloys processed by simple high-ratio extrusion methods [J]. *Mater Trans*, 2002, 43(10): 2424–2432.
- [4] WATANABE H, MUKAI T, KUHZU M, TANABE S, HIGASHI K. Low temperature super plasticity in a ZK60 magnesium alloy [J]. *Mater Trans*, 1999, 40(8): 809–814.
- [5] PEREZ-PRADO M T, DEL VALLE J A, CONTRERAS J M, RUANO O A. Microstructural evolution during large strain hot rolling of an AM60 Mg alloy [J]. *Scr Mater*, 2004, 50(5): 661–665.
- [6] PEREZ-PRADO M T, DEL VALLE J A, RUANO O A. Achieving high strength in commercial Mg cast alloys through large strain rolling [J]. *Mater Lett*, 2005, 59(26): 3299–3303.
- [7] DEL VALLE J A, PEREZ-PRADO M T, RUANO O A. Accumulative roll bonding of a Mg-based AZ61 alloy [J]. *Mater Sci Eng A*, 2005, 410/411: 353–357.
- [8] ZHAN M Y, LI Y Y, CHEN W P. Improving mechanical properties of Mg-Al-Zn alloy sheets through accumulative roll-bonding [J]. *Trans Nonferrous Met Soc China*, 2008, 18(2): 309–314.
- [9] YIN D L, ZHANG K F, WANG G F, HAN W B. Warm deformation behavior of hot-rolled AZ31 Mg alloy [J]. *Mater Sci Eng A*, 2005, 392(1/2): 320–325.
- [10] JÄGER A, LUKÁČ P, GÄRTNEROVÁ V, BOHLEN J, KAINER K U. Tensile properties of hot rolled AZ31 Mg alloy sheets at elevated temperatures [J]. *J Alloys Comp*, 2004, 378(1/2): 184–187.
- [11] CHANG T C, WANG J Y, O C M, LEE S Y. Grain refining of magnesium alloy AZ31 by rolling [J]. *J Mater Pro Tech*, 2003, 140: 588–591.
- [12] BOHLEN J, CHMEL'Y K F, DOBROŃ P, KAISER F, LETZIG D, LUKÁČ P, KAINER K U. Orientation effects on acoustic emission during tensile deformation of hot rolled magnesium alloy AZ31 [J]. *J Alloys Comp*, 2004, 378(1/2): 207–213.
- [13] BARNETT M R, NAVE M D, BETTLES C J. Deformation microstructures and textures of some cold rolled Mg alloys [J]. *Mater Sci Eng*, 2004, 386(1/2): 205–211.
- [14] MOHRI T, MABUCHI M, NAKAMURA M, ASAHINA T, IWASAKI H, AIZAWA T, HIGASHI K. Microstructural evolution and super plasticity of rolled Mg-9Al-1Zn [J]. *Mater Sci Eng*, 2000, 290(1/2): 139–144.
- [15] GALIYEV A, KAIBYSHEV R. Superplasticity in a magnesium alloy subjected to isothermal rolling [J]. *Scripta Mater*, 2004, 51(2): 89–93.
- [16] CHINO Y, LEE J S, SASSA K, KAMIYA A, MABUCHI M. Press formability of a rolled AZ31 Mg alloy sheet with controlled texture [J]. *Mater Lett*, 2006, 60(2): 173–176.
- [17] ZHANG B P, TU Y F, CHEN J Y, ZHANG H L, KANG Y L, SUZUKI H G. Preparation and characterization of as-rolled AZ31 magnesium alloy sheets [J]. *J Mater Pro Tech*, 2007, 184(3): 102–107.
- [18] WATANABE H, MUKAI T, ISHIKAWA K. Differential speed rolling of an AZ31 magnesium alloy and the resulting mechanical properties [J]. *J Mater Sci*, 2004, 39(4): 1477–1480.

- [19] WATARI H, DAVEY K, ALONSO M T. Effects of rolling condition on warm deep drawability of Mg alloy sheets produced by twin-roll strip casting [J]. Mater Sci Forum, 2005, 475/479: 489–492.
- [20] CHINO Y, MABUCHI M, KISHIHARA R, HOSOKAWA H, YAMADA Y, WEN C, SHIMOJIMA K, IWASAKI H. Mechanical properties and press formability at room temperature of AZ31 Mg alloy processed by single roller drive rolling [J]. Mater Trans, 2002, 43(10): 2554–2560.
- [21] 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(11): 3073–3084.
- [22] LIN H K, HUANG J C, LANGDON T G. Relationship between texture and low temperature superplasticity in an extruded AZ31 Mg alloy processed by ECAP [J]. Mater Sci Eng A, 2005, 402(1/2): 250–257.
- [23] KLEINER S, UGGOWITZER P J. Mechanical anisotropy of extruded Mg-6% Al-1% Zn alloy [J]. Mater Sci Eng A, 2004, A379(1/2): 258–263.
- [24] PYZALLA A, BRODMANN M, LEE P L, HAEFFNER D. Microstructure, texture and residual microstrains in MgAl₃Zn deformed at very high strain rates [C]//Magnesium Alloys and the Applications, Munich, 2000: 125.
- [25] KLIMANEK P, PÖTZSCH A. Microstructure evolution under compressive plastic deformation of magnesium at different temperatures and strain rates [J]. Mater Sci Eng A, 2002, A324(1/2): 145–150.

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