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# Effect of rolling temperature on microstructure and texture of twin roll cast ZK60 magnesium alloy

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**Abstract:** Twin roll cast ZK60 alloy strip/sheet with final thickness of 0.5 mm was prepared, and effect of rolling temperature on microstructure and texture development was investigated using OM and XRD technique, microstructure and texture were measured on specimens subjected to rolling experiment at different rolling temperature, and macrotexture was also evaluated by X-ray diffraction method. In addition, the $(10\bar{1}0)$ and (0002) pole figures were measured, and the tensile test was performed to reveal the influence of rolling temperature on mechanical properties. The results show that the microstructure of ZK60 alloy sheet consisted of fibrous structure with elongated grains, and shear bands along the rolling direction after warm rolling. Dynamic recrystallization could be found during the warm rolling process at rolling temperature 350 °C and above. And many fine recrystallized grain could be observed in the shear bands area. It is a little difficult to see the recrystallized grain in the sheet warm rolled at 300 °C because of higher density of shear bands. The warm rolled ZK60 alloy sheet exhibited strong (0002) pole texture, the intensity of (0002) pole figure decreases with the increasing of rolling temperature and the basal pole tilted slightly to the transverse direction after warm rolling.

**Key words:** warm rolling; microstructure; macrotexture; pole figure; ZK60 alloy

## 1 Introduction

Magnesium alloy is the lightest structural material with high specific strength and specific stiffness, good damping capacity and castability[1−3]. Moreover, because of the ease of recycling of metallic materials, magnesium has received global attention from the standpoint of environmental protection. Unfortunately, the major barrier for magnesium alloy using in cars is still primarily high manufacturing cost. In this regards, the use of continuous twin roll casting (TRC) of Mg alloys has been again attracting increasing attention of researchers. The alloys produced by twin roll casting and rolling technology were found to exhibit homogeneity of microstructure, refined grain size and reduced segregation and a dense distribution of fine particles with Mg/Al solid solution[4−7].

Mg alloys exhibit limited ductility at room temperature because of the presence of limited slip systems and strong fiber texture. The deformation mechanisms of ZK60 alloys were investigated under compression, tension, and rolling at various temperatures and strain rates[8−13]. Dynamic recrystallization (DRX) was also observed during hot deformation of ZK60. Therefore, for industrial application of magnesium sheet metal, the establishment of texture and formability control during rolling process is necessitated. JEONG et al[14] and DEL VALLE et al[15] discussed the effects of rolling conditions on the microstructure and texture development of AZ series alloy. In this study, the average texture of ZK60 Mg alloy produced by twin roll cast and warm rolling with different per pass thickness reduction is evaluated using X-ray diffraction method and its

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correlation with mechanical properties is studied.

## 2 Experimental

The material used in this study was twin roll cast ZK60 Mg alloy strip. The thickness of initial strip was 3.5 mm. Warm rolling was performed in several passes to reduce the sheet thickness to 0.5 mm under various rolling temperature with 50% per pass thickness reduction. The warm rolling was carried out at 300 °C, 350 °C, 400 °C, roller surface temperature of 250 °C and at the roll speed of 5 m/min. Before the first pass, the strip was heated at rolling temperature, and reheated again at the rolling temperature for 5 min before the sequent rolling passes to maintain the sheet temperature at about the rolling temperature. This process was continually carried out several passes to reduce the strip thickness from 3.5 mm to about 0.5 mm. After each rolling experiment, the surface of specimen was examined to check whether the side-cracking occurred.

The specimens used for optical microstructural observation were prepared by mechanical polishing with the polycrystalline diamond suspension glycol based solution. The grain structure was revealed by subsequent etching with a solution of picric acid (3 g), acetic acid (20 mL), distilled water (20 mL), and ethanol (50 mL). All optical microstructures were observed along the transverse direction of the rolling sheet. A JEM−2100F transmission electron microscope (TEM) was used to take TEM micrographs. Thin foils for TEM parallel to the rolling plane were prepared by a twin jet electro-polisher using a solution containing $HClO_4$(5%), butanol(35%) and methanol(60%), and then ion-beam milled. The rolled sheet was machined to the subsize of ASTM E8 tensile specimen, and the specimens were prepared to have orientation parallel to the rolling direction. Tensile test was conducted at an ambient temperature on a standard universal testing machine (Instron 4206). The textures of ZK60 Mg alloy were analyzed on rolling plane using the sheet specimens of 20 mm×20 mm×0.5 mm. The $(10\bar{1}0)$ and (0002) pole figures were measured using X-ray diffraction with $CuK_\alpha$ radiation.

## 3 Results and discussion

### 3.1 Microstructure of as-cast ZK60 alloy strip

The surface of the twin roll cast (TRC) strip is smooth and does not react with oxygen in the air, even though no cover gas is used for shielding the cast magnesium at outlet of strip casting process. Fig.1 shows the microstructure of TRC ZK60 alloy strip with a thickness of 3.5 mm. The microstructure of TRC strip is different from the top to the bottom as shown in Fig.1(a), which is mainly attributed to the different cooling rate through the thickness of strip during the TRC process. The molten metal contacts directly with the copper rollers cooled with water inside. Chilled grain structure appears just below the surface of the strip, and dendrite structure is developed from surface to center of cast strip along the thermal gradient. The dendrite structure in the region of the surface shows relatively finer than that in the central zone of the cast strip due to relatively higher cooling effect. The eutectics and intermetallic compounds are seen in the interdendritic region as shown in Fig.1(b). Fig.1(b) also shows the SEM and its corresponding Mg, Zn and Zr dot-mapping images of TRC ZK60 alloy strip. The eutectics and intermetallic compounds in the interdentritic region mainly contain Mg and Zn elements.

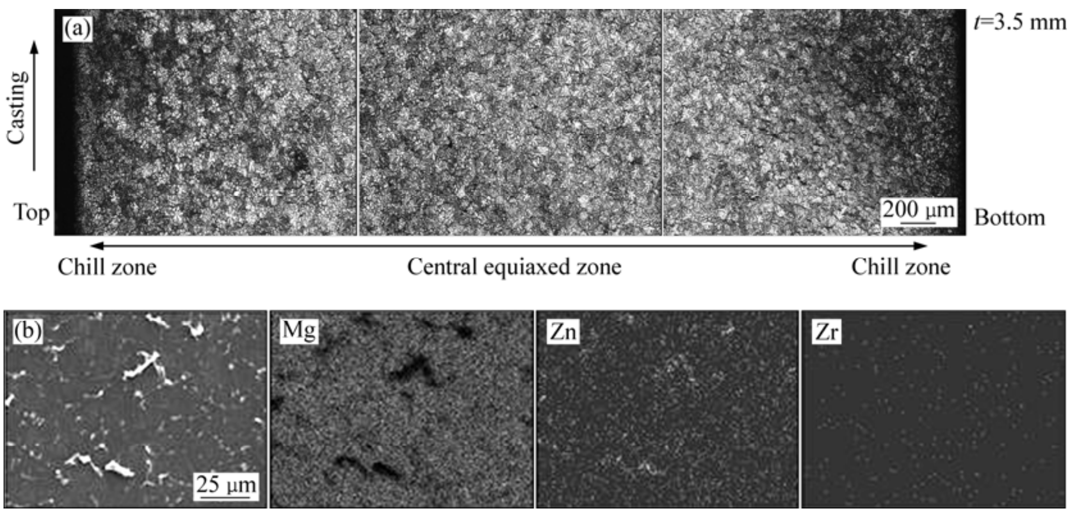


**Fig.1** Microstructures of TRC ZK60 alloy strip: (a) OM of as-cast strip; (b) SEM and its corresponding Mg, Zn and Zr dot-mapping images of as-cast strip

### 3.2 Microstructure of warm rolled ZK60 alloy sheet

Fig.2 shows the microstructure of ZK60 alloy sheet which was warm rolled at 300, 350 and 400 °C, respectively. And the sheet was annealed at the rolling temperature after warm rolling. The microstructure of the rolled ZK60 alloy sheet consists of fibrous structure with elongated grains, and shear bands along the rolling direction, which are changed from dentritic structure (Fig.1(a)). Low temperature warm rolling results in higher density of shear bands (Fig.2(a)), and the density of shear bands decreases with the increasing of the rolling temperature. The dynamic recrystallization could be found during the warm rolling process at higher rolling temperature, and many fine recrystallized grains could be observed in the shear bands area (see Figs.2(c) and (e)). It is a little difficult to find recrystallized grain in the sheet warm rolled at 300 °C because of the higher density of shear bands. Static recrystallization presents in the sheet during annealing heat treatment process (see Figs.2(b), (d) and (f)). Grain refinement is found to occur during the warm rolling and annealing process.

Fig.3 shows the TEM images of the warm rolled ZK60 alloy sheet. It can be seen that dislocation has formed during the warm rolling process, and substructure is observed at lower rolling temperature (see Fig.3(a)). The equiaxial grains are discovered in the warm rolled sheet (Figs.3(c) and (e)). Due to the above aspects, it can further explain that dynamic recrystallization process takes place during warm rolling process. In addition, there are many fine particles dispersed in the warm rolled ZK60 alloy sheet. Twin roll casting process is a semi-rapid solidification processing so that the fast solidification rate can result in the supersaturation of Zn in $\alpha$-Mg matrix during the twin roll casting process[16].

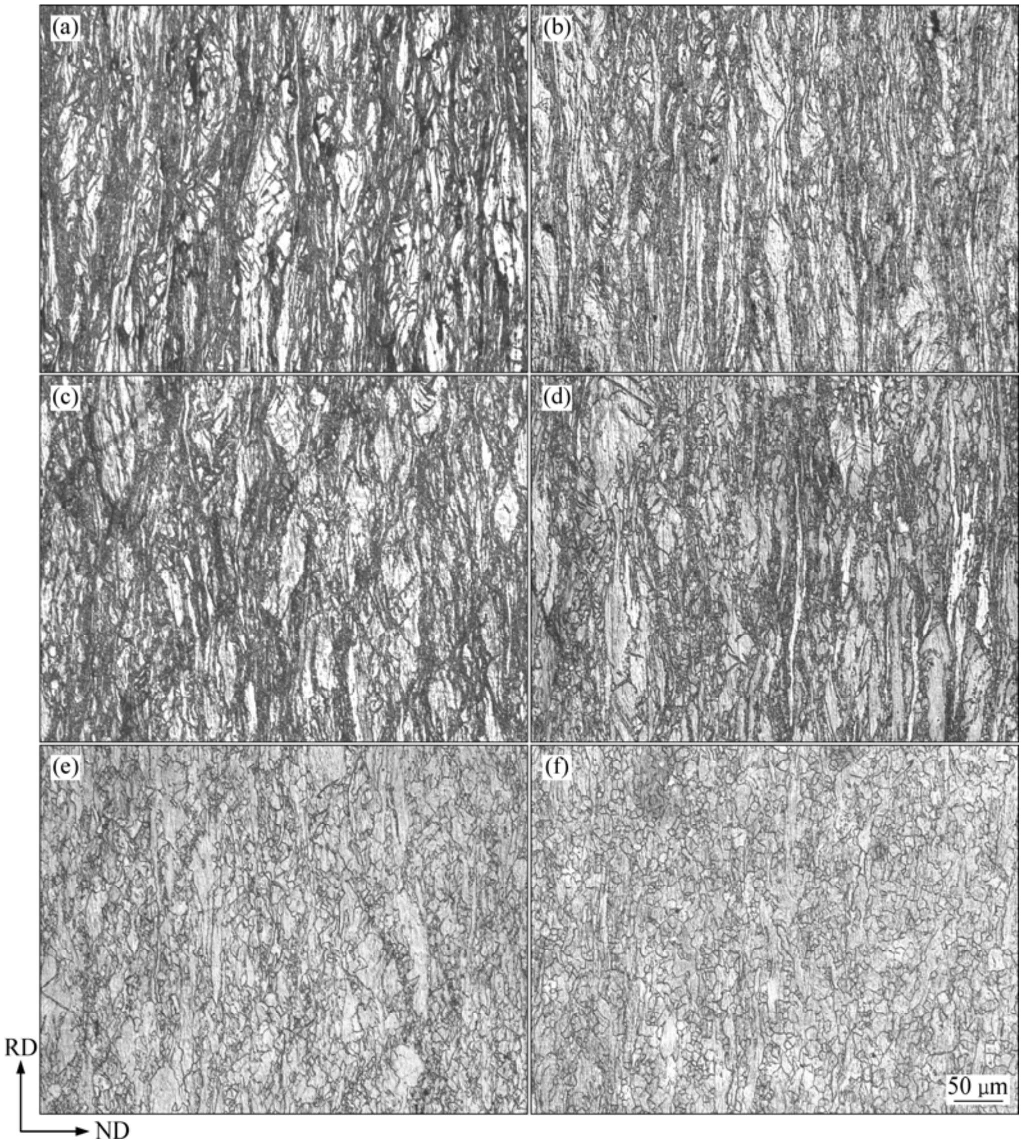


**Fig.2** Microstructures of ZK60 alloy sheet warm rolled and annealed at different temperatures: (a, c, e) As rolled; (b, d, f) As annealed; (a, b) 300 °C; (c, d) 350 °C; (e, f) 400 °C

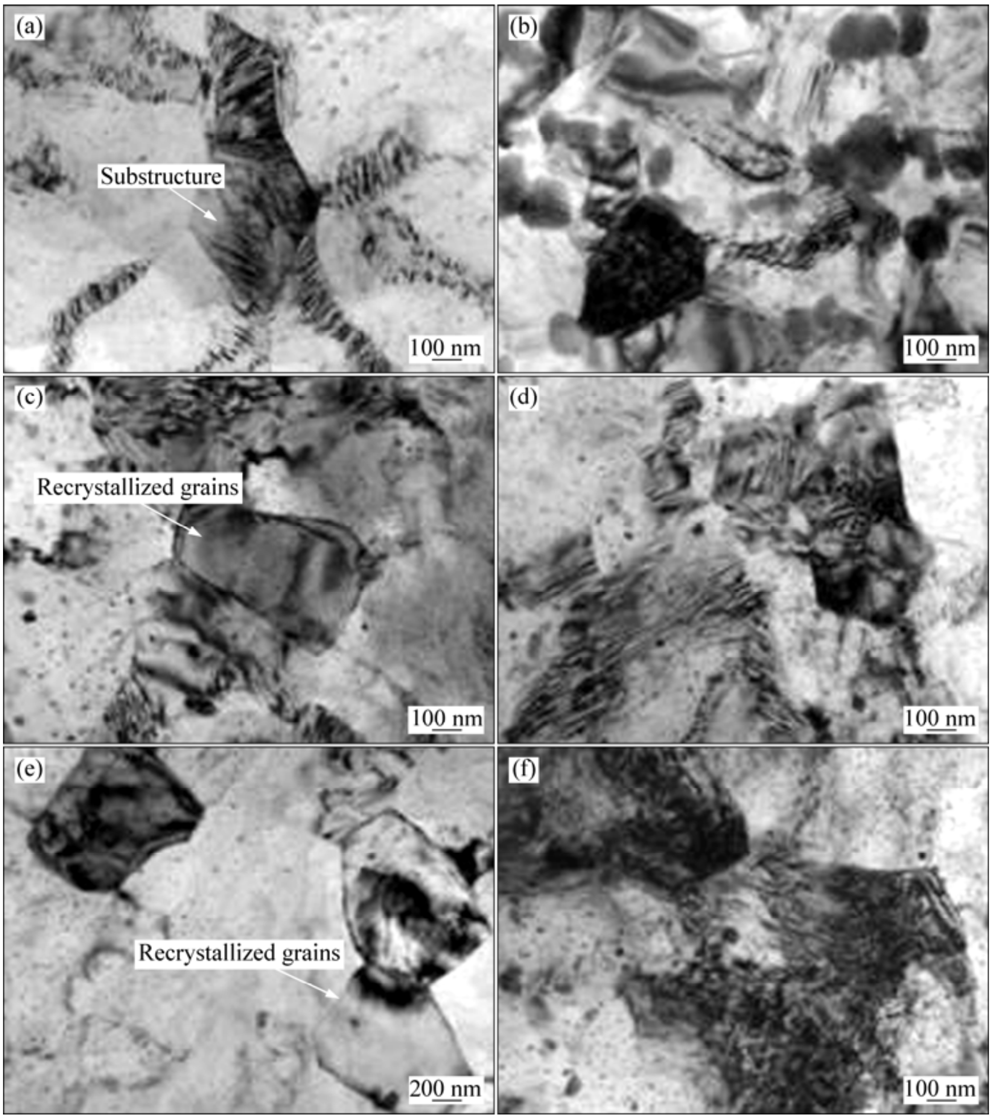


**Fig.3** TEM images of ZK60 alloy sheet warm rolled at different temperatures: (a, b) 300 °C; (c, d) 350 °C; (e, f) 400 °C

This leads to more and finer precipitates present in ZK60 alloy during the sequent warm rolling process.

### 3.3 Mechanical properties of warm rolled ZK60 alloy sheet

The room temperature tensile strength, yield strength and elongation of the warm rolled sheet are shown in Fig.4. Tensile test indicates that the warm rolled sheet with high tensile strength, yield strength, but a little low elongation, is obtained as shown in Fig.4(a). The strength decreases and elongation increases with the increasing of rolling temperature. As shown in Fig.2, the density of the shear bands decreases and dynamic recrystallization presents with the increasing of rolling temperature. Therefore, the decrease of strength and increase of elongation could be attributed to the decrease of shear bands density as well as the present of dynamic recrystallization. Annealing heat treatment decreases the strength and increases the elongation as shown in Fig.4(b). This could be attributed to the static recrystallization during annealing process (shown in Fig.2).

### 3.4 Texture analysis of warm rolled ZK60 alloy sheet

The $(10\bar{1}0)$ and $(0002)$ pole figures of the warm rolled ZK60 alloy sheet are shown in Fig.5. The maximum pole intensity of (0002) pole figures is 9.7, 8.3 and 6.2 for the sheet warm rolled at 300, 350 and 400 °C, respectively. The major texture components of all the specimens can be expressed by ND//(0001) fiber texture. The texture of warm rolled ZK60 alloy sheet shows that the basal pole spreads out to transverse direction.

The maximum pole intensity of (0002) pole figure decreases with the increasing of rolling temperature and the basal pole tilts slightly to the transverse direction after warm rolling. In addition to strain, a number of factors may affect the deformation texture, and these are briefly considered[17−18]: rolling geometry, deformation

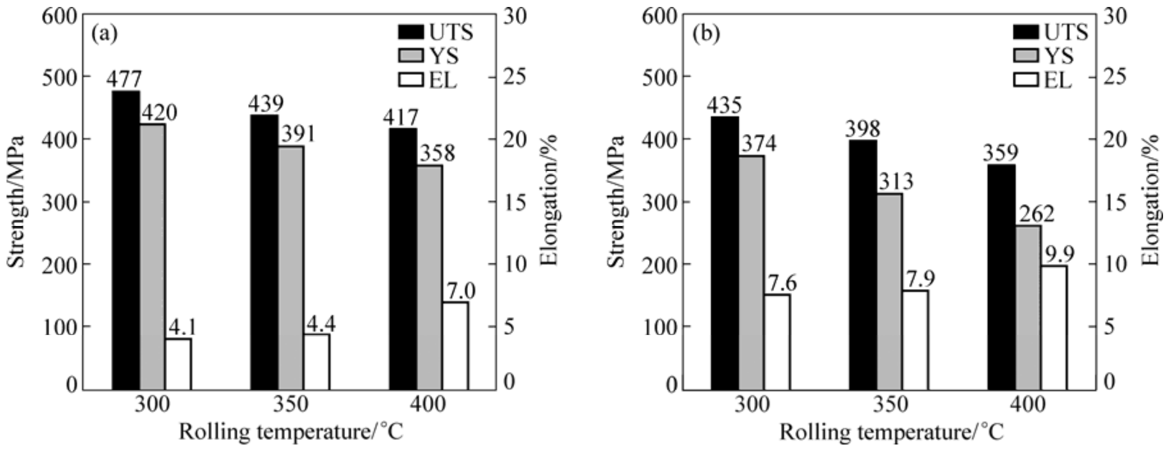


**Fig.4** Room temperature tensile properties of ZK60 alloy sheet: (a) as rolled; (b) as annealed

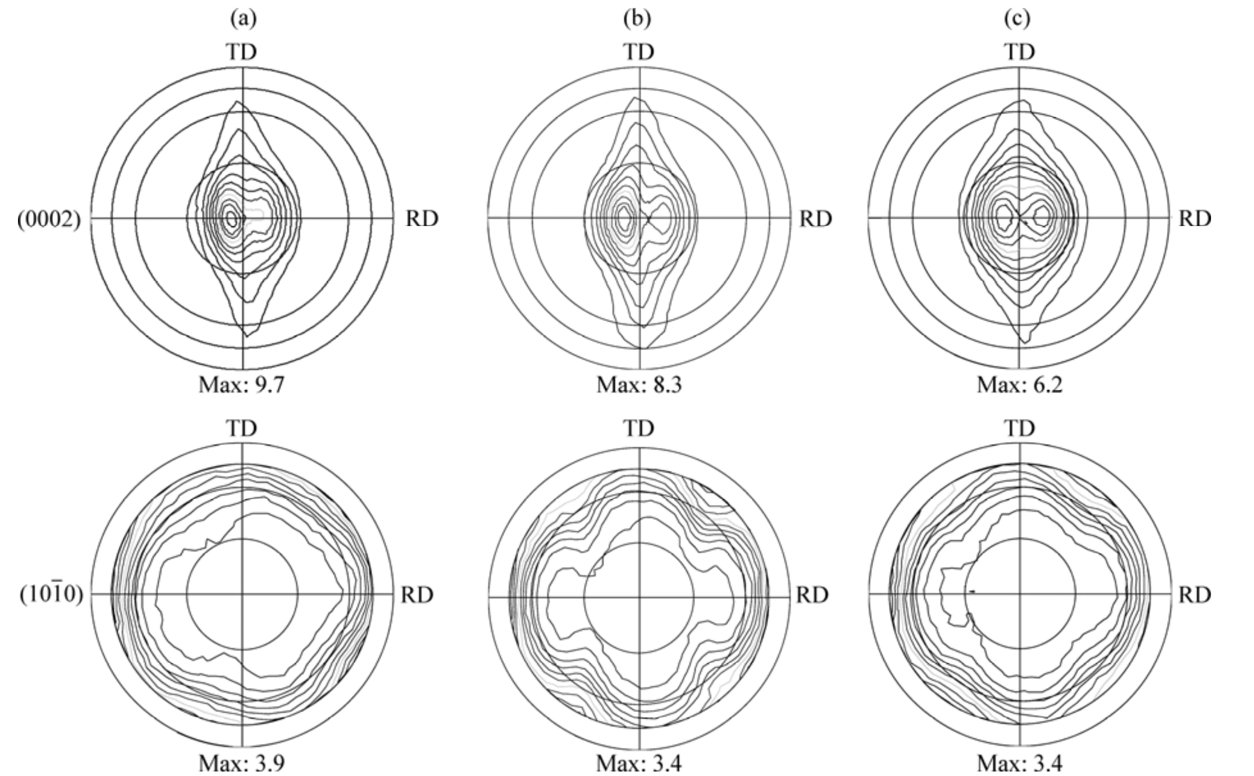


**Fig.5** (0002) and (10$\bar{1}$0) pole figures of ZK60 alloy sheet with 0.5 mm thickness warm rolled at: (a) 300 °C; (b) 350 °C; (c) 400 °C

temperature, grain size (here is the initial grain size), shear bands and second-phase particles, etc. In this study, the three warm rolled specimens are all rolled with the same rolling equipment and with the same initial grain size, so the factors that may cause difference of deformation texture of the warm rolled ZK60 alloy sheet are rolling temperature, shear bands and second-phase particles. As shown in Figs.2(c) and (e), the density of shear bands decreases with the increase of the rolling temperature. The formation of the shear bands tends to cause rotation of the material about the transverse direction, and this may lead to changes in intensity of the texture and an intensity redistribution along the fibre. Some materials could be weakened (such as copper) and some materials could be strengthened (such as brass) by shear band.

## 4 Conclusions

1) The microstructure of ZK60 alloy sheet consists of fibrous structure with elongated grains, and shear bands along the rolling direction after warm rolled at 300, 350, 400 °C with 50% per pass thickness reduction.

2) Dynamic recrystallization could be found during the warm rolling process at 350 °C and above. And many fine recrystallized grain could be observed in the shear

bands area. It is a little difficult to observe the recrystallized grain in the sheet warm rolled at 300 °C because of the higher density of shear bands.

3) The warm rolled ZK60 alloy sheet exhibits strong (0002) pole texture, the intensity of (0002) pole figure decreases with the increasing of rolling temperature and the basal pole tilts slightly to the transverse direction after warm rolling.

## References


[1] MORDIKE B L, EBERT T. Magnesium properties—applications—potential [J]. Mater Sci Eng A, 2001, 302(1): 37−45.

[2] STANFORD N, BARNETT M R. The origin of “rare earth” texture development in extruded Mg-based alloys and its effect on tensile ductility [J]. Mater Sci Eng A, 2008, 496: 399−408.

[3] LUO Shou-jing, SHAN Wei-wei. Steady state rheological behavior of semi-solid ZK60-RE magnesium alloy during compression [J]. Transactions of Nonferrous Metals Society of China, 2007, 17(5): 974−980.

[4] CHEN H M, KANG S B, YU H S, KIM H W, MIN G H. Microstructure and mechanical properties of Mg-4.5Al-1.0Zn alloy sheets produced by twin roll casting and sequential warm rolling[J]. Mater Sci Eng A, 2008, 492(1/2): 317−326.

[5] KANG S B, CHEN H M, KIM H W, CHO J H. Effect of reheating and warm rolling on microstructure and mechanical properties of twin roll strip cast Mg-4.5Al-1.0Zn-0.4Mn-0.3Ca alloy sheet [C]// Magnesium Technology 2008. Charlotte, USA: TMS, 2008: 147−152.

[6] WATARI H, DAVEY K, RASGADO M T, HAGA T, IZAW S. Semi-solid manufacturing process of magnesium alloys by twin-roll casting [J]. Journal of Materials Processing Technology, 2004, 155/156: 1662−1667.

[7] ZHAO Hu, LI Pei-jie, HE Liang-ju. Deformation microstructure and characteristics of cast-rolling and normal rolling AZ31 magnesium alloy sheets [J]. The Chinese Journal of Nonferrous Metals, 2009, 19(11): 1887−1893. (in Chinese).

[8] YANG Xu-yue, JI Ze-sheng, MIURA H, SAKAI T. Dynamic recrystallization and texture development during hot deformation of magnesium alloy AZ31 [J]. Transactions of Nonferrous Metals Society of China, 2009, 19(1): 55−60.

[9] KIM W J, HONG S I, KIM Y S, MIN S H, JEONG H T, LEE J D. Texture development and its effect on mechanical properties of an AZ61 Mg alloy fabricated by equal channel angular pressing [J]. Acta Mater, 2003, 51(11): 3293−3307.

[10] GALIYEV A, KAIBYSHEV R, GOTTSTEIN G. TEM and 3DAP characterization of an age-hardened Mg-Ca-Zn alloy [J]. Scripta Mater, 2005, 53(6): 675−679.

[11] WANG C Y, WANG X J, CHANG H, WU K, ZHENG M Y. Processing maps for hot working of ZK60 magnesium alloy [J]. Mater Sci Eng A, 2007, 464(1/2): 52−58.

[12] YANG Y Q, LI B C, ZHANG Z M. Flow stress of wrought magnesium alloys during hot compression deformation at medium and high temperatures [J]. Mater Sci Eng A, 2009, 499(1/2): 238−241.

[13] LIU Z Y, BAI S, KANG S B. Low-temperature dynamic recrystallization occurring at a high deformation temperature during hot compression of twin-roll-cast Mg-5.51Zn-0.49Zr alloy [J]. Scripta Mater, 2009, 60(6): 403−406.

[14] JEONG H T, HA T K. Texture development in a warm rolled AZ31 magnesium alloy [J]. J Mater Process Tech, 2007, 187/188: 559−561.

[15] DEL VALLE J A, PEREZ-PRADO M T, RUANO O A. Texture evolution during large-strain hot rolling of the Mg AZ61 alloy [J]. Mater Sci Eng A, 2003, 355(1/2): 68−78.

[16] PARK S S, OH Y S, KANG D H, KIM N J. Microstructural evolution in twin-roll strip cast Mg-Zn-Mn-Al alloy [J]. Mater Sci Eng A, 2007, 449/450/451: 352−355.

[17] HUMPHREYS F J, HATHERLY M. Recrystallization and related annealing phenomena [M]. 2nd. Kidlington, UK: Elsevier Ltd, 2004.

[18] TANG Wei-qin, ZHANG Shao-rui, FAN Xiao-hui, LI Da-yong, PENG Ying-hong. Texture and its effect on mechanical properties of AZ31 magnesium alloy [J]. The Chinese Journal of Nonferrous Metals, 2010, 20(3): 371−377. (in Chinese)


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