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Microstructure refining and property improvement of ZK60 magnesium alloy by hot rolling

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Abstract: The as-cast ZK60 Mg alloy ingot with mean grain size of 278 μm after solution treatment was subjected to severe plastic deformation by multi-pass hot rolling with decreasing temperatures. The process facilitated steady grain refinement by dynamic recrystallization with increasing rolling deformation, and the final grain size was reduced to 7.2 μm by 6 rolling passes. Tensile experiments demonstrated that the yield and ultimate strength of the finished plates were isotropic, with the former being about 202 MPa and the latter 307 MPa in both the rolling and the transverse direction, respectively. Electron backscattering diffraction revealed that the finished plates presented homogeneous microstructure and texture pattern, which well explains the observed strength isotropy. Furthermore, it is shown that the as-rolled finished plates don’t need the conventional long-time T5 aging treatment, and this can simplify the process flow and help to improve the efficiency for industrial production.

Key words: ZK60 magnesium alloy; hot rolling; aging; microstructure refining, mechanical properties

1 Introduction

Due to their low densities and high specific strength, magnesium alloys have attracted considerable attention as structural materials for applications in aerospace and automobile industries [1]. In order to exploit the application of magnesium alloys for high-performance structural uses, which is impeded by their relatively poor strength and ductility, much effort has been devoted to the study of grain refining, and various techniques, such as equal channel angular pressing (ECAP), differential speed rolling (DSR) and large-strain hot rolling (LSHR) [2−4], have been developed to achieve grain refining and mechanical properties improvement. Indeed, it was reported that, by grain refining, not only can the ductility be enhanced, but also the mechanical strength is improved significantly [5−7]. In this work, LSHR with decreasing temperatures was used to achieve grain refining of a ZK60 Mg alloy, and its effect on mechanical property improvement was investigated.

2 Experimental

The starting material used in the present study was a commercial as-cast ZK60 Mg alloy (Mg-6.63%Zn-0.56%Zr) plate ingot with a thickness of 28 mm (designated as ZK60-C). The ingot was solution treated at 400 °C for 24 h (ZK60-ST), and subsequently hot-rolled to a thickness of 2.0 mm (ZK60-HR) at decreasing temperatures, from 400 to 250 °C, with a thickness reduction ratio of 30%−50% per pass. Finally, aging treatment was conducted at 175 °C in the finished plates for 0.25−32 h.

The microstructures were observed by optical microscopy (OM), and mean linear intercept method was used to measure grain sizes. Tensile specimens with a gauge length of 25 mm were machined out of the as-rolled plates along both rolling (RD) and transverse directions (TD). Tensile tests were performed using an Instron 5569 machine at a constant tensile velocity equal to an initial strain rate of 6.7×10^{-4} s^{-1}. X-ray diffraction (XRD) experiments were performed to examine the effect of solution treatment and the initial crystalline orientation distribution. Electron backscattering diffraction (EBSD) in JEOL 733 electron probe equipped with TSL OIM analysis system was used to identify the texture of the finished plates. Micro-hardness tests were performed on a HVS-5 machine to measure the harnesses of the specimens after aging treatment.

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3 Results and discussion

3.1 Microstructure characteristics

Figure 1 shows the XRD spectra of ZK60-C and ZK60-ST compared with the standard XRD spectrum of magnesium powder (PDF35-0821). It is clearly observed that the intensity of the peaks corresponding to precipitated phases strongly decrease and almost disappear after the solution treatment, and the microstructures are shown in Fig. 2. This means that the single phase solid solution is formed in ZK60-ST and good formability would be obtained in following rolling deformation. On the other hand, the magnitude of the peaks in the present ZK60 alloy is similar to that of the standard magnesium card. Thus, it is suggested that the initial material exhibits no preferred crystalline orientation.

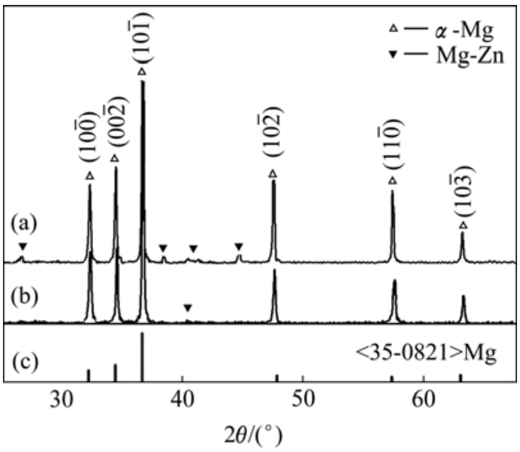

Fig. 1 XRD patterns of ZK60-C (a), ZK60-ST (b) and magnesium standard card (c)

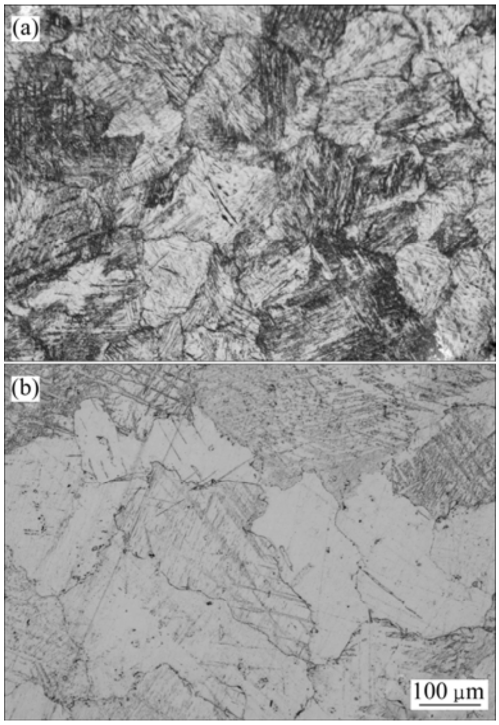

Fig.2 Microstructures of ZK60-C (a) and ZK60-ST (b) alloy billet

After a single pass (Fig. 3(a)) at 400 °C, dynamic recrystallization occurred partially in grain boundaries, resulting in the average grain size of 68.5 μm compared with 278 μm in ZK60-ST. Twinning existed inside coarse grains which have undergone significant deformation but unrefined. The similar situation continued to the third pass as the fraction of fine recrystallized grains steadily increased. When the equivalent strain reached 1.5, as shown in Fig. 3(e), the microstructure consisted of

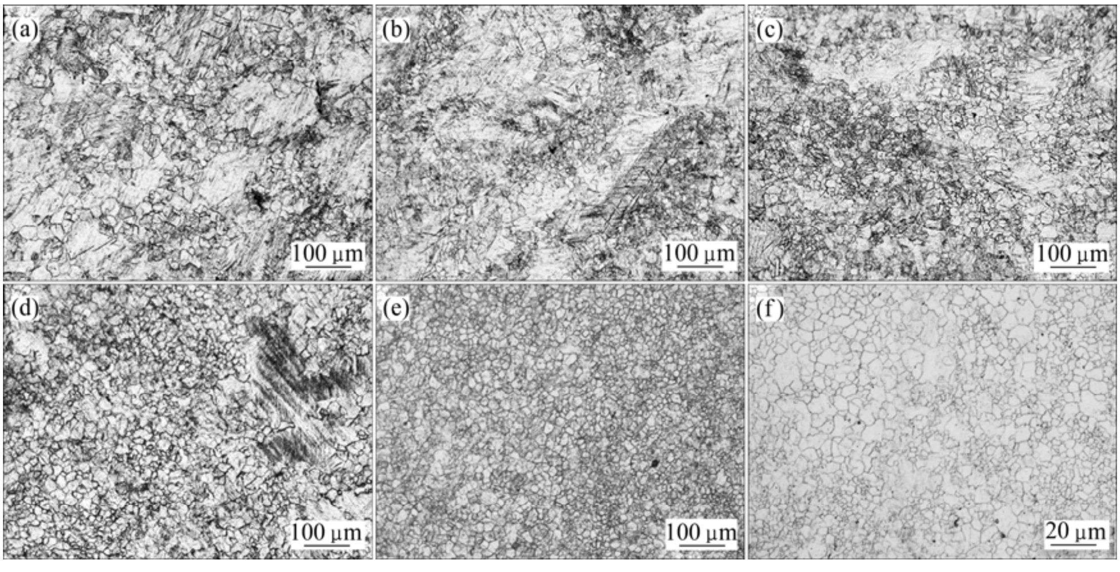

Fig. 3 Microstructures of as-rolled ZK60 Mg alloy after different rolling passes: (a) 1 pass; (b) 2 passes; (c) 3 passes; (d) 4 passes; (e) 5 passes; (f) 6 passes

equiaxed grains with an average grain size of 17.4 μm. Additional hot-rolling at 250 °C with a reduction ratio of 50%, the corresponding grain size further reduced to 7.2 μm with almost a perfect log normal distribution as shown in Fig. 4(b), which indicated that homogeneous microstructure with fine grains was achieved after 6 passes (Fig. 3(f)). Grain refinement was facilitated by the processing with decreasing temperatures as illustrated in Fig. 4(a).

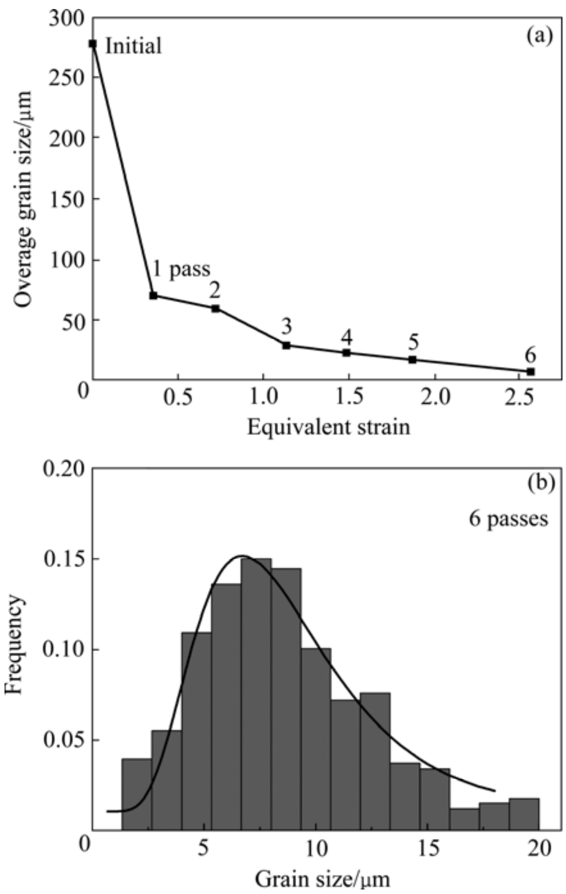

Fig. 4 (a) Average grain size of as-rolled ZK60 Mg alloy after different rolling passes; (b) Grain size distribution of ZK60-HR (6 passes)

3.2 Texture and tensile properties

Figure. 5(a) shows the {0001} and $\{10\bar{1}0\}$ pole figures of ZK60-HR obtained from EBSD data. In contrast to the initial material, the finished plates exhibited a strong basal texture with the *a*-axis randomly rotated in the RD-TD plane, while the {0001} pole intensity was spread more to the RD than to the TD. Basal texture was also beneficial for suppressing twinning deformation during rolling, with the help of elevated temperature and incremental grain refinement [8−9]. Thus the dominant plastic mechanism in hot rolling would be basal slip with participation of pyramidal <*c*+*a*> and prismatic slip [9−11].

The stress—strain curves of ZK60-ST and the finished plates along the RD and TD are presented in Fig. 5(b). As shown, the mechanical properties were significantly improved by hot rolling. Concretely speaking, the enhancement of the strengths, from 142 to 202 MPa in yield strength and 293 to 307 MPa in ultimate strength, was attributed to both grain refinement strengthening [12] and texture hardening [9, 13], meanwhile, the elongation were also strikingly enhanced to 21%−25% by grain refinement [14] with the suppression of twinning. In contrast to the isotropy of strengths, the difference of elongations was correlated with the subtle difference in the distribution of {0001} pole intensity in the RD-TD plane, which mainly manifested as increased elongation if the {0001} pole intensity perpendicular to the tensile direction was more concentrated [15]. This resulted in the uniform elongation of the RD slightly higher than that of the TD, which were determined using the considered construction: $\mathrm{d}\sigma/\mathrm{d}\varepsilon=\sigma$, as shown in Fig. 5(b).

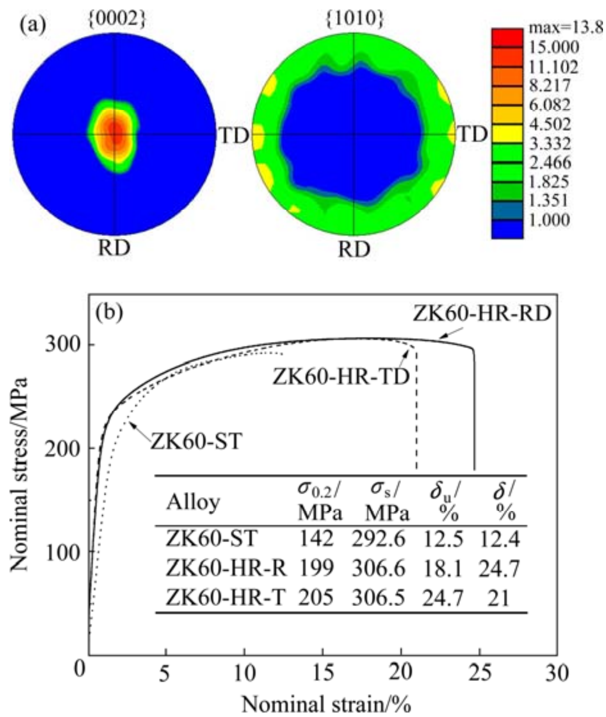

Fig. 5 {0001} and $\{10\bar{1}0\}$ pole figures of ZK60-HR (a) and stress—strain curves of ZK60-ST and ZK60-HR (b)

3.3 Further aging treatment

TEM image of the microstructure of ZK60-HR is shown in Fig. 6(a). The precipitates consisted of numerous rod-shaped and blocky β_1' phases [16−17], which has been considered to be the most effective strengthening phase. In contrast, minute quantity of plated precipitates (β_2') [17] were observed, which are related to over aging phenomenon [16, 18]. This means that a significantly higher number density of precipitates formed during the hot rolling process.

In order to examine the potential of further aging hardening, further aging treatments were carried out on the finished plates. The micro-hardness curve of the ZK60 alloy at 175 °C is shown in Fig. 6. The kinetic of aging reached its maximal hardness of HV 77.6 in 0.5 h, in contrast to 10−24 h in T6 treatment at the similar temperatures [16, 18−19]. In addition, compared with the initial hardness, the effect of further age hardening resulted in merely 3HV increasing. Therefore, the results revealed that there was no need for further long-time aging treatment and the finished plates could be availably heat treated by stress relief annealing at 175 °C for 0.5 h. This high efficiency is very favorable for industrial production.

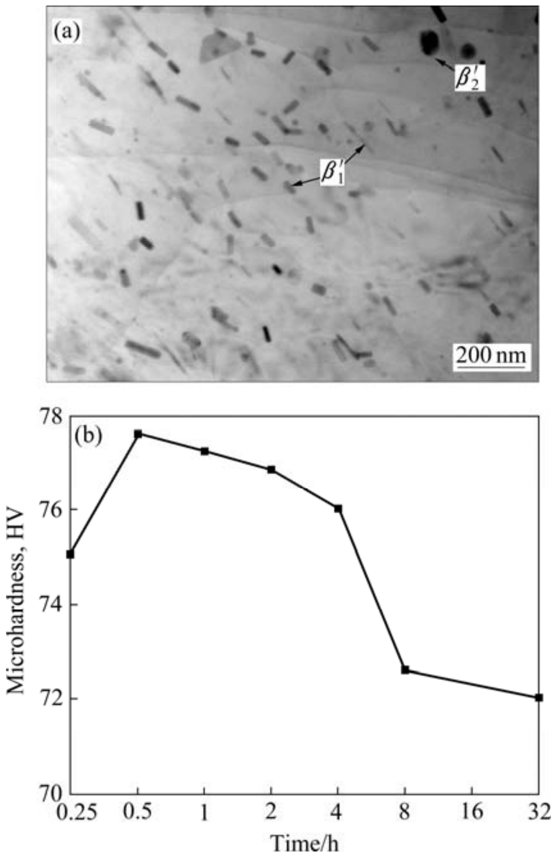

Fig. 6 TEM image (a) and micro-hardness (b) curve of ZK60-HR for aging at 175 °C

4 Conclusions

1) Significant grain refinement was achieved by multi-pass hot rolling with decreasing temperatures. The grain size with 278 μm in ingot was reduced to 7.2 μm by only 6 rolling passes.

2) Enhancement of strengths, from 142 to 202 MPa in yield and 293 to 307 MPa in ultimate, is most attributed to both grain refinement and basal texture hardening. In contrast, the effect of grain refinement on the ductility is also beneficial, but the concentration of basal texture goes against ductility improvement. Total elongations along the RD and the TD are both more than 20%.

3) A significantly higher number density of (β_1') strengthening phase precipitated during the hot rolling process. Further aging treatments reveal that stress relief annealing at 175 °C for 0.5 h, rather than long-time aging treatment, is availably used as the finial heat treatment process for the finished plates.

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ZK60 镁合金在热轧过程中的组织细化及性能改善

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摘 要：采用多道次降温热轧工艺对 ZK60 镁合金铸锭进行累积大塑性变形。此工艺方法可促进动态再结晶细化晶粒的程度，经 6 道次轧制后，晶粒尺寸由铸态时的 278 μm 细化至终轧板时的 7.2 μm。拉伸试验结果表明终轧板材在板平面内具有很好的强度各向同性，其屈服强度约为 202 MPa，抗拉强度约为 307 MPa，这与电子背散射衍射测试结果所揭示的良好组织均匀性和板平面晶粒取向分布均匀性相一致。进一步的研究表明，终轧板材不需要做进一步的时效处理，因此，本工艺方法在优化组织性能的同时，可以简化工艺流程和提高生产效率。

关键词：ZK60 镁合金；热轧变形；时效处理；组织细化；力学性能

(Edited by CHEN Ai-hua)