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Letter

Preparation of sound ribbons with submicrometer-grained microstructure on a Mg–Zn alloy

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Abstract In this paper, a viable way to fabricate Mg alloy sound ribbons with ultra-fine-grained microstructure was presented. The hot-rolled and annealed Mg–0.4Zn (at%) alloy exhibited excellent rollability to form sound ribbons with submicrometer grains when subjected to one-pass cold rolling process. The more balanced multi-mode dislocation slips originated from the significant decrease of critical resolved shear stress for non-basal slip with the addition of solute Zn and the favorable crystallographic orientation were suggested to be responsible for the excellent cold rollability. The formation of ultra-fine-grained microstructure was attributed to low-temperature dynamic recrystallization occurring during the cold rolling process with large strain.

Keywords Mg–Zn alloys · Cold rolling · Microstructure · Texture

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The ultra-fine-grained materials (UFMs) can achieve a considerable gain in mechanical responses, such as strength and fatigue resistance [1]. The feasible methods to produce UFMs are high-pressure torsion, equal channel angular extrusion, accumulative roll bonding, twist extrusion and multi-axial deformation, of all which impose severe plastic deformation (SPD) at low temperatures [2]. Amount of efforts has been primarily focused on cubic metals due to their excellent formability at low temperatures [3]. The pure magnesium, however, exhibits poor cold formability due to the unbalanced activation of various slip systems at low temperature due to its hexagonal close-packed (HCP) crystal structure, thus restricting the capacity of Mg to achieve UFMs by SPD. Although several attempts have shown that the microstructure of Mg alloys could be refined to ultra-fine down to nanosize by SPD methods [4, 5], the refined regions were highly localized or the refining process was too tedious. Therefore, how to prepare UFMs on Mg alloys in a simple way is still a challenging and interesting issue. For this, the Mg alloy must exhibit excellent formability during SPD process at low ambient temperature. According to Akhtar et al. [6], the critical resolved shear stress (CRSS) for prismatic slip decreases continuously with the addition of solute Zn at low temperatures. It provided a chance to activate more balanced dislocation slips during the deforming process for Mg alloy at low temperatures. In this study, thereby, Mg–0.4Zn (at%) alloy was selected to investigate its rollability as well as the resultant microstructure in one-pass rolling at room temperature.

Mg–0.4Zn alloy samples were produced from 99.98 % pure Mg and Zn. The as-cast materials were homogenized at 500 °C for 4 h and then cut to 4-mm-thick sheets. These sheets were then hot rolled at 400 °C to the final thickness of 1 mm. Hot rolling was performed in several passes with a constant reduction of thickness of less than 20 % per pass. To eliminate the residual stress of the sheets, the 1-mm-thick sheets were annealed at 450 °C for 30 min. Then, the sheets with the size of 100 mm × 10 mm × 1 mm were single-pass rolled to ribbons at room temperature on a two-roll mill with rollers of Φ100 mm × 250 mm. To this end, the gap distance between the two rollers was preset to zero size. To obtain a fully recrystallized microstructure, an annealing treatment of 2 h at 350 °C was performed after the cold rolling process.

The initial microstructure and the texture of the hot-rolled sheets were observed by electron back-scattered diffraction (EBSD) measurements in a JEOL6500F field emission gun scanning electron microscope (SEM, The Japanese Electronics Co. Ltd., Japan) equipped with an automatic orientation acquisition system (HKL Channel 5, Oxford Instruments, UK). The texture of cold-rolled ribbons was measured on a D8 DISCOVER X-ray diffractometer (Bruker AXS Inc., USA) using reflection geometry and Cu Kα radiation. For the detailed microstructure observation, a JEM-2000EX transmission electron microscope (TEM, Hitachi Co., Japan) was used. The foils for TEM observation were thinned at 10 V and −40 °C by the twin-jet polishing technique using an electrolyte consisting of 1 vol% $HClO_4$ and 99 vol% methanol.

Figure S1 (online) shows the ribbons after cold rolling. It was seen that upon the one-pass cold rolling, a uniform, long thin ribbon (about 100 μm thick) with no obvious cracks at the ribbons edges has formed. The ribbon was ductile and could be bent to honeycomb structure. Namely, the Mg–0.4Zn alloy exhibited extensive cold rollability in terms of its thickness reduction over 90 %, which was comparable to some ductile cubic materials, such as Cu and Al.

Figure 1 shows the EBSD band contrast map and the corresponding {0001} pole figure of the annealed sheets (Fig. 1a) and the (00.2) pole figure of the cold-rolled ribbons (Fig. 1b). It could be clearly seen from the {0001} pole figure that a majority of grains oriented with their {0001} pole/*c*-axis distributed in the plane defined by rolling direction (RD)-normal direction (ND), far from transverse direction (TD) of the sheet. However, Fig. 1b shows that the cold-rolled ribbons exhibited a strong ‘‘basal’’ texture. There existed a tendency for the peak intensity to tilt away from ND toward RD. A symmetrical splitting of the {0002} basal texture which amounts about ±20° in RD could be observed. The texture evolution of Mg–0.4Zn alloy during cold rolling was very similar to that of the Mg alloys deformed at room temperature [7].

Figure 2 shows the TEM micrographs of the cold-rolled and annealed ribbons with the thickness reduction above 90 %, respectively. No twinning was observed in the TEM micrographs (Fig. 2a), indicating that dislocation slip was predominant to accommodate the rolling strain. After low-temperature annealing, a fully recrystallized microstructure with the average grain size of 0.3 μm was formed, as

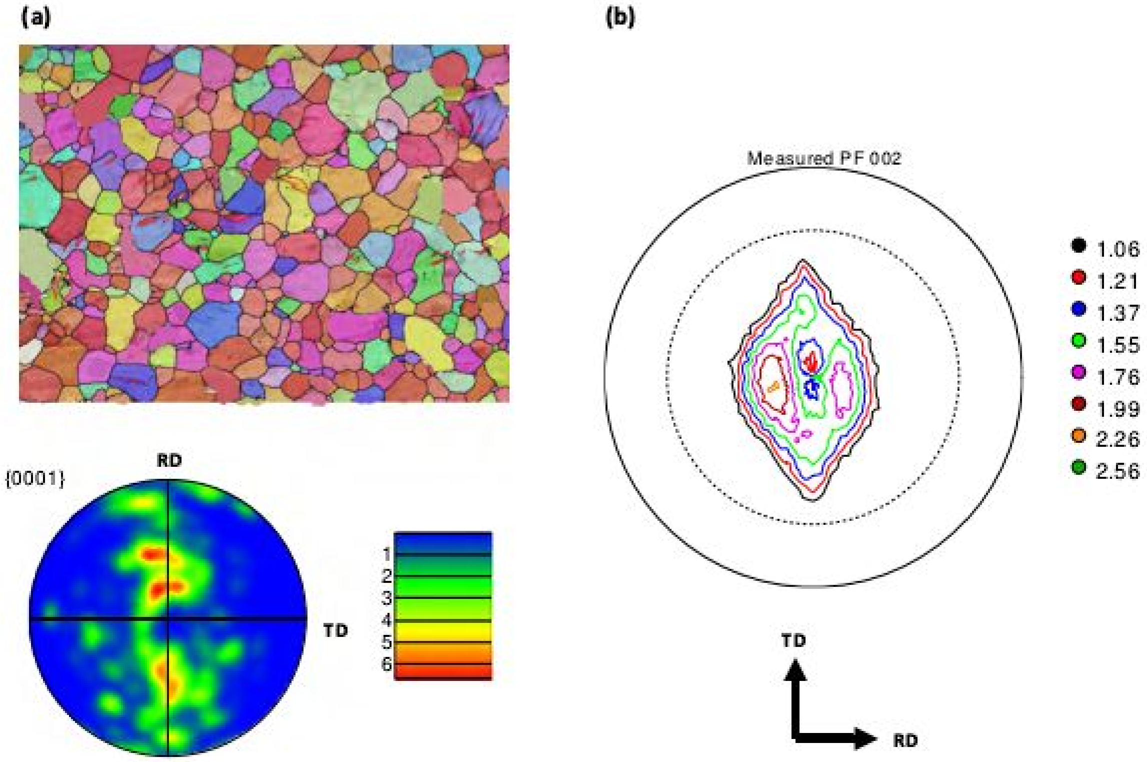

Fig. 1 Evolution of texture during the cold rolling process. **a** EBSD band contrast map and the corresponding {0001} pole figure of the annealed sheets; **b** (00.2) pole figure of the cold-rolled ribbons

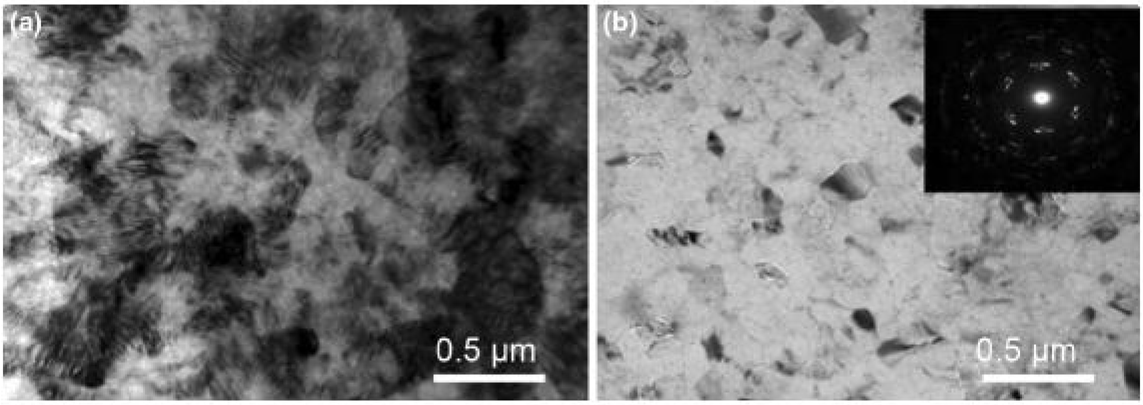

Fig. 2 TEM micrograph and electron diffraction pattern showing the microstructure refinement of the ribbons after cold rolling (**a**) and annealing process (**b**)

shown in Fig. 2b. This indicated that an ultra-fine structure had been achieved in the Mg–0.4Zn alloy after cold rolling process, and this could be further evidenced by the electron diffraction rings on the inset in the right-up corner of the TEM micrograph (Fig. 2b).

It was known that the activation of tension twinning was an important deformation mode to accommodate the strain of pure Mg and AZ31 alloy during the deforming process at low temperature due to its low CRSS. And in such a case, pure Mg and AZ31 alloy easily produced twin dynamic recrystallization (TDRX) [8]. However, in this study, no twinning was observed in the TEM micrographs (Fig. 2a). In order to illustrate the twinning behavior of the Mg–0.4Zn alloy during the cold rolling process, a one-pass rolling process with the strain of 40 % was performed. Figure S2 (online) shows the TEM micrographs of the typical microstructure for the sample with thickness reduction of 40 %. From Fig. S2a (online), although some twins could be seen in the high-strained regions, the "recrystallized" grains did not form in the twinning regions. This was obviously different from the results of Sun et al. [9]. It was suggested that during the surface mechanical attrition treatment of AZ91 alloy, the multiple dislocation slip systems in the twins were activated and subdivided the twin platelets into subgrains. In addition, Fig. S2b (online) shows that the grains boundaries of the recrystallized grains exhibited a specific contrast, which suggested high density of grains boundaries dislocations. All these indicated that the dislocation behavior rather than the twins was the predominant factor to contribute to the excellent rollability and control the recrystallization process for Mg–0.4Zn alloy during the one-pass cold rolling.

Figure 3 presents high resolution transmission electron microscopy (HRTEM) images of the microstructure in the cold-rolled ribbons. In Fig. 3a (electron beam direction was [0001]), prismatic $<a>$ dislocations were identified. The inverse Fourier transform (IFT) image (Fig. 3b) also presented abundant prismatic dislocations in the cold-deformed Mg–0.4Zn alloy ribbons. The TEM specimen was cut with the surface parallel to the rolling plane, so that electron beam direction was most commonly [0001] due to the basal texture. Therefore, it was difficult to observe the pyramidal $<c+a>$ dislocations on the $\{10\bar{1}0\}$ plane under the HRTEM. However, it could not exclude that the pyramidal $<c+a>$ dislocations existed in the cold-rolled microstructure. In fact, the symmetrical splitting of the {0002} basal texture in the RD shown in Fig. 1b has identified that the pyramidal $<c+a>$ slip also had been activated during the rolling process as found also in the study [6]. So, it was sure that the dislocation slips on the non-basal planes were activated and developed as the efficient deformation modes to provide strain in RD and ND directions for Mg–Zn alloys during cold rolling process.

Based on the foregoing TEM observations on the cold-rolled samples, it was concluded that multi-mode dislocation slips were the predominant factor for the hot-rolled and annealed Mg–0.4Zn alloy to exhibit excellent cold rollability. The activation of non-basal especially the prismatic dislocations (Fig. 3) was correlated surely with the significant decrease of CRSS for prismatic slip with the addition of solute Zn at low temperatures, as indicated by the Akhtar et al. [6]. This was the intrinsic factor. The activation of the non-basal dislocation slips could be further interpreted in terms of the texture characteristics. Figure 2a shows that prior to the cold rolling process, the initial texture of the Mg–Zn sheets was characterized by {0001} poles oriented in the RD-ND plane, suggesting that Schmid factor could be very high for the basal $<a>$, prismatic $<a>$ and pyramidal $<c+a>$ slips during the rolling process. So, the basal $<a>$ slip and prismatic $<a>$ contributed the main strain at the initial stage of the rolling deformation. With the increase of the rolling strain, the rapidly formed basal texture (Fig. 2b) could suppress the basal slip of grains to some extent. In such a case, the slip of dislocations on non-basal planes composed of prismatic $<a>$ and pyramidal $<c+a>$ Burgers vectors could be activated due to their more favorable orientations.

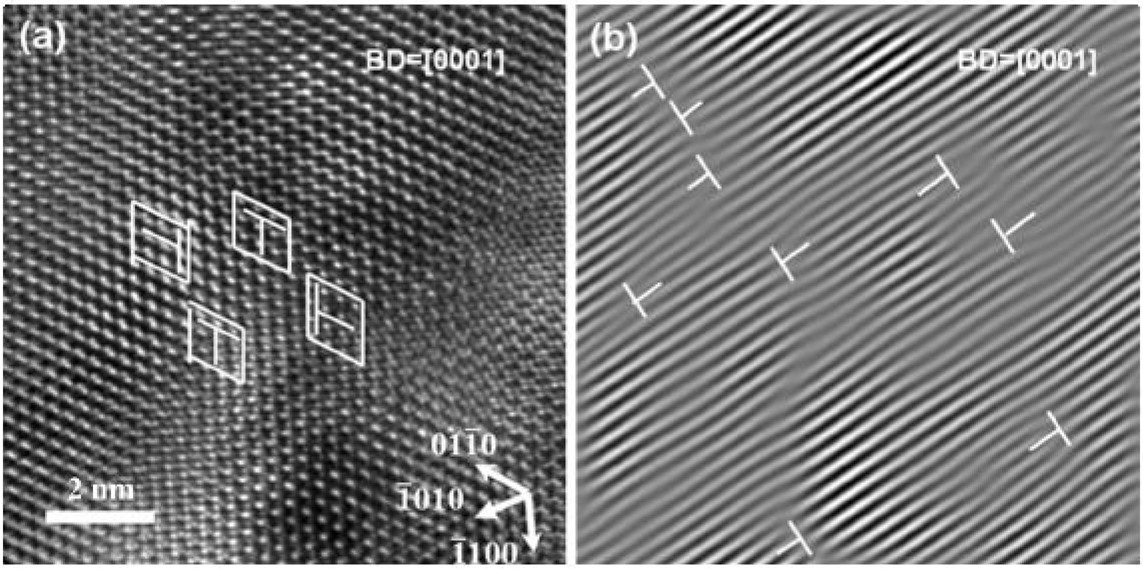

Fig. 3 HRTEM image (**a**) and IFT image, (**b**) showing$\{10\bar{1}0\}$ <*a*> dislocations in the deformed matrix in the cold-rolled ribbons

Nonetheless, although the basal slip was suppressed in the basal grains, it would be easy to be activated in grains deviating slightly from the strict basal orientation because of its low CRSS. Namely, the Mg–0.4Zn alloy has exhibited a more balanced basal and non-basal dislocation slip during the cold rolling process. This means that in order to realize the homogeneous deformation, the key point was that the equivalent resolved shear stress, which was determined mainly by CRSS and Schmid factor (orientation), could reach similar levels for all basal and non-basal dislocation slips.

As an accommodation mechanism of non-basal dislocation slips to the rolling strain, the contribution of non-basal slip to total rolling deformation was modest as presented in the study of Agnew et al. [10].

Therefore, the dynamic recrystallization (DRX) behavior of Mg–0.4Zn occured in the cold rolling process was similar to the pure Mg, in which a high rate of DRX with strain was available. From Fig. 3b, the high density of grains boundaries dislocations could be observed during the cold rolling process, meaning that low-temperature DRX (LTDRX) had occured, as suggested by Sitdikov et al. [8].

Thus, the high-rate LTDRX during cold rolling process was suggested to be the key factor to form the ultra-fine microstructure in the cold-rolled ribbons in this study. At low ambient temperature, the strains imposed in conventional processing were not sufficient to introduce DRX grains with size less than 1 μm because of the low workability of magnesium and its alloys at these temperatures. However, in this case, due to the favorable crystallographic orientation and the effect of Zn addition, the as-rolled and annealed Mg–1Zn alloy could be rolled to the final strain of 3. In such case, the LTDRX process happened during the cold rolling process with intensive strain produced the UFG structure.

In this study, the rollability and the microstructure of hot-rolled and annealed Mg–0.4Zn sheets under single-pass cold rolling were investigated. The annealed sheets exhibited excellent rollability with reduction of 90 % when subjected to one-pass cold rolling. The excellent rollability stemmed mainly from the balanced dislocation slips from both basal and non-basal planes. The low-temperature dynamic recrystallization process could effectively refine the microstructure of the cold-rolled ribbons to the ultra-fine scale.

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Conflict of interest The authors declare that they have no conflict of interest.

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