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Influence of large cold strain on the microstructural evolution for a magnesium alloy subjected to multi-pass cold drawing

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Cold-drawn AZ31 Mg alloy wires with a wide range of strains up to ~1.2 were prepared by multi-pass cold drawing to investigate the influence of cold strain on microstructural evolution. With the drawing, plastic deformation was initially dominated by twinning, particularly {10–10} twinning, but gradually becoming by slips especially after twinning was exhausted at large strains > 1. More importantly, pronounced refined crystallites with diameter ~100 nm were achieved through profuse intersections across twin bundles starting from strains larger than ~0.6, and contributed to the weakening of {0002} fiber texture. This refinement could be interpreted as twin fragmentation related to dynamic recovery; and to some extent it implied a possibility of fabrication of ultrafine-grained structure by cold severe plastic deformation for magnesium alloys.

1. Introduction

Ultrafine-grained (UFG) microstructure is desirable in achieving significant strengthening without compromising ductility, and it is attracting focus in wrought magnesium alloys owing to their high Hall–Petch slope k_y values but poor cold-formability [1–4]. Of the many techniques for achieving UFG structures, severe plastic deformation (SPD), dealing with grain refinement through dynamic recrystallization (DRX) [5,6], has been considered to the most effective procedure in comparison to other methods such as equal channel angular extrusion (ECAE) of 0.8 μm in a Mg–Zn–Ca alloy [7], friction stir processing (FSP) of less than 0.3 μm in a AZ31 Mg alloy [8], and high-pressure torsion (HPT) of ~1.0 μm in a ZK60 Mg alloy [9]. However, the above-mentioned techniques are not suitable for industrial production, and are still limited to the laboratory scale.

Comparatively, conventional processes, e.g. rolling and drawing, at lower, or even ambient, temperatures, which have larger commercial potential in high quality and productivity than other SPD processes, can also be implemented to achieve a UFG structure by means of plastic strain-induced refinement. This method has been successfully applied in high stacking fault energy materials (such as Cu and Al alloys [10]), and is reasonably expected in low stacking fault energy materials (e.g. Mg alloys [11]) by the refinement stemming from profuse twinning [12]. If it is not viable, the material can also be remarkably refined by static recrystallization under subsequent annealing at low temperatures. Unfortunately, limited studies were presented with the restriction of cold formability of magnesium alloy, which is difficult to achieve cold strains larger than 0.5 [13,14]. Therefore, in order to control microstructure effectively in cold deformed magnesium alloys, it is necessary to understand the mechanisms of cold deformation after large cold strains. With reference to the industrial processes in steel and brass wires, multi-pass cold drawing was used in an attempt to maximize the cumulative cold strain in a fine-grained magnesium alloy. Meanwhile, the fabrication feasibility of UFG structure by cold deformation in Mg alloys was explored.

2. Experimental procedures

Fine-grained AZ31 Mg alloy (Mg–3.28 wt% Al–0.46 wt% Zn) wires with a diameter of 2.0 mm were investigated in this work. The AZ31 Mg wires were processed by successive cold-drawing passes with 9–11% area reduction per pass. A cold-drawn wire of 1.1 mm in diameter with cumulative area reduction of about 71%, corresponding to an equivalent strain of ~1.2 was achieved. Microstructure and texture of as-received AZ31 Mg wires were investigated by Electron backscattering diffraction (EBSD) in JEOL

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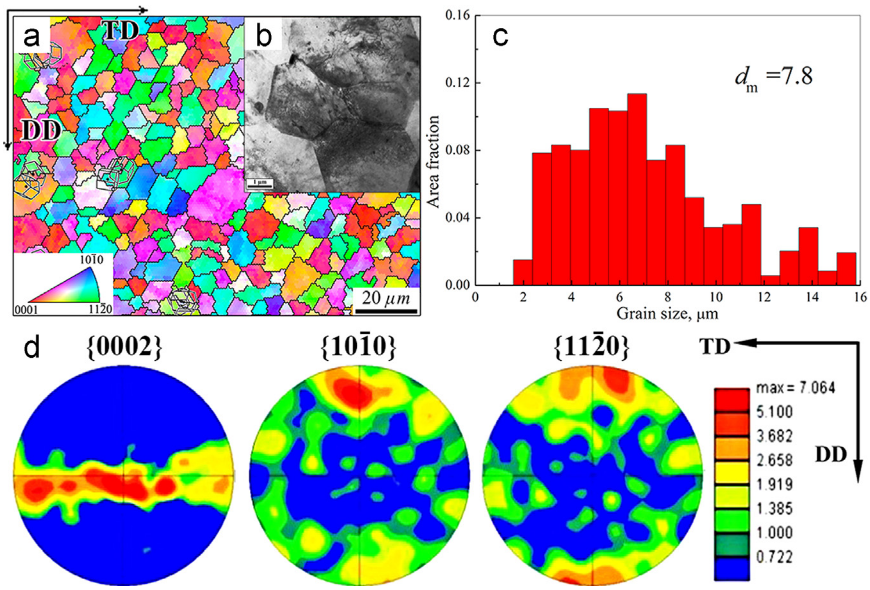

Fig. 1. Microstructural characteristics of as-received AZ31 Mg wires in DD–TD plane: (a) inverse pole figure map with representative crystallographic relationships between parent grains and twins; (b) TEM micrograph of grains; (c) grain size distribution and (d) {0002}, {10–10} and {11–20} pole figures.

733 electron probe equipped with TSL OIM Analysis5 system, and the mean linear intercept method was used to measure mean grain size, with the observation plane parallel to the drawing direction (DD). Microstructures of the specimens during cold drawing were performed on OLYMPUS GX71 optical microscopy (OM) and TECNAI transmission electron microscopy (TEM) parallel to DD. Textures of as-drawn AZ31 Mg wires were measured on a D5000 X-ray diffractometer (SIEMENS Ltd.) using reflection geometry and Cu Ka radiation. Mechanical properties were examined by tensile tests at room temperature with a constant speed of 1 mm/min equal to an initial strain rate of $1.67 \times 10^{-3}\ s^{-1}$. Tensile specimens with a gauge length of 10 mm were cut parallel to DD.

3. Results and discussion

Fig. 1a displays the microstructure characterizations of the initial materials obtained by EBSD in DD–TD (transverse direction) plane with the normal direction (ND) corresponding to the crystal reference system. It is clear that the initial material had relatively equiaxed grains of ~7.8 μm in average and a uniform grain size distribution as a result of the extrusion history of the wires (Fig. 1c), and exhibited a strong {0002}//DD ({0002} basal plane parallel to DD) fiber texture with a maximal pole intensity of about 7(Fig. 1d). Consistent fine equiaxed grains slightly larger than 5 μm with dense dislocations, especially around the boundaries, could be also examined using TEM (Fig. 1b). These results indicate that dynamic recrystallization fully occurred during thermal deformation. Farthing {10–12} tension twins were also observed in large grains whose *c*-axis is inclined from DD with a range of 55–90°, which is beneficial to activation of tension twinning during extrusion [15].

The typical microstructure features in 24% cold-drawn AZ31 Mg wires are evident in Fig. 2. Comparatively, the morphology of grain boundary changed weakly after three cold drawing passes where the cumulative strain was equal to ~0.3, and an average grain size of ~6.3 μm was obtained (Fig. 2a). But inside, lamellar structure mixed mainly with elongated boundaries of ~1.5 μm in width and profusely parallel {10–11} compression twins of ~200 nm in width, often reported as twin bundles [10], was formed (Fig. 2b). In contrast, a small amount of broad, lenticular {10–12} tension twins (~1 μm in width) with merged, and mostly bent, interfaces were also developed as shown in Fig. 2c. Both the features clearly show that twinning, especially of {10–11} compression twins, was one of major deformation modes for small strains during cold drawing. Additionally, high density dislocations, kinked and found in forms of pile-ups not tangles around boundaries, suggest that the interaction between dislocations and boundaries were gradually strengthened, making the activation of non-basal slip systems easier even at ambient temperature [16].

Fig. 3 shows the microstructure of 47% cold-drawn AZ31 Mg wires where the cumulative cold strain reached ~0.6. As drawing proceeded, equiaxed microstructure gradually evolved into fibrous structure mixed with significantly elongated grain boundaries and densely elongated diagonal strips less than 1μm in width was observed, corresponding to the reorientation of grains and twinning evolution (Fig. 3a). As shown in Fig. 3b, more fine lamellar structures enriched with twins were continuously generated and bent. Corrugated slip bands in enlarged view indicated cross slip between basal and non-basal slip planes might be activated referring to Friedel-Escaig mechanism, which is theoretically anticipated at low temperature (≤473 K) in magnesium alloys [16]. Similarly, Koike et al. [17] pointed that cross slip of a/3⟨11–20⟩ dislocations could occur at ambient temperature for fine-grained AZ31 Mg alloy. Interestingly, a considerable number of fragmentations consisting of very small crystallites (having relatively perfect lattice) with ~100 nm in length, appeared first in the narrow regions of twin bundles undergoing significant shear deformation (Fig. 3b and c). Corresponding diffraction patterns taken from selected areas presented arcs with angle of ~50°, and this refers that preferred orientation in the small crystallites were not eliminated despite of new sharp high-angle boundaries formed. This structure is clearly linked to strong slip activity, and is presumably also influenced by dynamic recovery during cold drawing. Meanwhile, some thin lath-like microbands about 100 nm thick were seen in Fig. 3b and d, and therefore was expected to contribute to the formation of fine crystallites.

As drawing continued to large strains ($\varepsilon > 1$), as seen in Fig. 4a where 71% cold-drawn AZ31 Mg wires were achieved, the various lamellar structures and their interfaces could not be clearly identified with interference of increasing dislocations (also mostly