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# Exceptional texture evolution induced by multi-pass cold drawing of magnesium alloy

Liuxia Sun, Jing Bai *, Feng Xue, Li Tao, Chenglin Chu, Jiao Meng

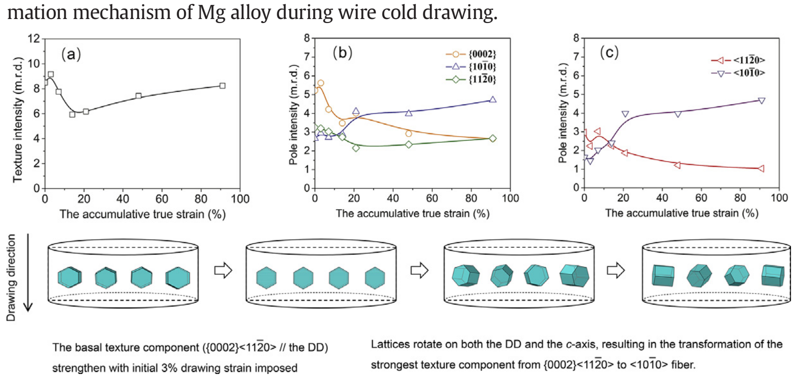
*School of Materials Science and Engineering, Southeast University, Jiangning, Nanjing 211189, Jiangsu, China*
*Jiangsu Key Laboratory for Advanced Metallic Materials, Jiangning, Nanjing 211189, Jiangsu, China*

## HIGHLIGHTS

- Mg-2wt%Zn shows an exceptionally good deformability at room temperature.
- The strongest texture component eventually transformed from as-extruded basal to <10$\bar{1}$0> fiber.
- The rotation processing of Mg lattices explains the cold-drawn deformation mechanism.

## GRAPHICAL ABSTRACT

Mg-2wt%Zn shows an excellent drawing deformability at room temperature. The texture intensity curves, which suggest an exceptional texture evolution, are helpful to understand the transformation of lattices and the deformation mechanism of Mg alloy during wire cold drawing.

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## ABSTRACT

We performed multiple-pass cold drawing for Mg-2wt%Zn alloy until 91% accumulative true strain, indicating an excellent drawing deformability of Mg alloy at room temperature. Based on texture analysis, the strong basal texture, which was formed during hot extrusion, was further strengthened at the initial stage of drawing. Subsequently, under the effect of twining and constrained inclined compressive stress, more slips were activated, leading to weakening of the basal texture and an evident drop in the maximum texture intensity. This process was associated with a rapid rise in microhardness and strength as well as a rapid decline in tensile elongation. At larger strain, non-basal slip systems were gradually activated. As a result, the maximum texture intensity gradually increase again, along with relatively smooth change in mechanical properties. The texture component eventually transformed from as-extruded basal texture to typical ⟨10$\bar{1}$0⟩ fiber texture with ⟨10$\bar{1}$0⟩ parallel to the drawing direction.


## 1. Introduction

For Mg and its alloys, strong texture is apt to be developed because of the significant anisotropy in HCP crystal structure [1–3]. Most texture of Mg alloys induced by cold plastic deformation, such as extrusion and rolling, tends to align with basal plane texture owing to the prior activation of {0002}⟨11$\bar{2}$0⟩ (⟨a⟩) basal slip system with much lower critical resolved shear stress (CRSS) than that of other non-basal slip system as well as twining systems [4]. The strong basal texture, however, results in extremely poor plasticity and formability of Mg alloys at room temperature.

* Corresponding author at: School of Materials Science and Engineering, Southeast University, Jiangning, Nanjing 211189, Jiangsu, China.
*E-mail address:* baijing@seu.edu.cn (J. Bai).

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To improve the formability of Mg alloys, following methods that can weaken basal texture are often adopted: i) Hot deformation: basal slip is only slightly temperature-dependent but non-basal slip systems possess CRSS values that decrease substantially with increasing temperature [5]. ii) Specific alloying addition: many alloying elements, typically like rare earth elements Ce, La, Gd etc., can promote the activation of non-basal slip through decreasing the stacking fault energy (SFE) [6] or reducing the c/a ratio [7], although there is still a controversy arguing the mechanism responsible for the texture effect. iii) Special deformation processes: for example, equal channel angular pressing (ECAP) can generate unique textures with the operating slip planes well oriented for the continuation of the strain by simply shearing at the intersection of two channels [8,9].

Using these methods above, various Mg profiles have been successfully produced. However, for Mg thin wire (usually with a diameter < 0.3 mm), especially precise thin wires, which have shown great application potential in medical materials and instruments (e.g., woven stent for digest tract or blood vessel, suture, staple, and used as reinforcement to prepare the polymer based composite) due to their outstanding biodegration and biological performance [10–13], there are still some difficulties although a few reports on the preparation of Mg thin wire can be found [14–22]. These difficulties main include more complicated processing and annealing technologies in comparison with some common metal (Cu, Al, Fe, etc.) thin wire production, and how to improve the productivity, dimensional accuracy and mechanical properties of thin wire, and so on. Most importantly, the deformation mechanism of Mg alloy during cold drawing is still indistinct, and therefore it is hard to optimize the processing technology through theory.

Cold drawing, as a conventional and the most efficient technique to produce metal thin wire with high dimensional accuracy and surface roughness, was long considered unsuitable to prepare Mg thin wire, because of both the room temperature process and especially the stress characteristics with principle tension stress along the drawing direction, which might accelerate the formation of sharp basal texture of Mg and result in a rapid deterioration in formability of wire. Contrary to the long-standing assumptions, our previous works [23,24] found Mg alloys actually showed quite good cold drawing formability through multiple-pass with the maximum ATS (accumulative true strain) >100%. It was unclear though what is the deformation mechanism.

On the other hand, the compositions of magnesium alloy wires also need to be considered based on the potential applications of Mg thin wire in medical field. For these researches [14–22] on Mg alloy wires, most of the materials studied were AZ61 or AZ31 alloy based on the Mg-Al series. Although AZ alloys show good mechanical properties and are now the most common commercial Mg alloys, there are a big doubt about the use of Al element in implantable materials due to its neurotoxicity [25]. Therefore, Mg-Zn based alloys with better biosafety have been extensively developed and investigated recently in vitro and in vivo as new biodegradable Mg alloys [26–29]. In addition, as an essential element of magnesium alloys, Zn is also often added to improve the comprehensive mechanical properties due to its strengthening effect by solid solution or aging treatment.

Using macrotexture analysis, in this work, we found some exceptional texture components in cold drawn Z2 (Mg-2wt%Zn) alloy wire, and further investigated the microstructural evolution processing. It is crucial to understand the deformation mechanism of Mg alloy during wire cold drawing, thus explaining our previous experimental results and providing a guide to produce Mg thin wire for medical or similar applications.

## 2. Experimental procedure

The materials employed in the present study were Z2 prepared from Mg (99.99%), Zn (99.99%) and the analyzed composition of the Z2 used here was Mg-2.13wt%Zn. Alloy ingots were prepared in a resistance heating furnace under protective gas atmosphere of $SF_6$ and $CO_2$ mixture at the ratio of 1:99, and then cast into a cylindrical water-cooling copper mold with 60 mm in diameter and 250 mm in height. Cylindrical samples with a diameter of 60 mm and a height of 90 mm were machined from one homogenized billet along the casting direction for preparation of annealing. Homogenization annealing of as-cast Z2 alloy billet was carried out in an electric furnace at the temperature of 655 K for 24 h. These billets then were hot extruded at 680 K with an extrusion ratio of ~20. After that, as-extruded Z2 wire with a thicker initial diameter of 3.0 mm was obtained followed by the successive cold-drawn passes.

During the cold drawing, the Z2 alloy wires are subjected to the external forces mainly containing tensile stress ($P$), compressive stress ($N$), friction ($T$). It is to reduce friction that the diamond die was used inside the wire drawing die and the lubricating oil was applied to the wire surface during cold drawing. The metal in deformation zone is subjected to radial compressive stress ($\sigma_r$), circumferential shear stress ($\sigma_\theta$), axial tensile stress ($\sigma_l$). The state of stress is schematically shown in Fig. 1(a). The microstructures and mechanical properties of Mg alloy wires are closely related to their state of stress. Here, they are affected by semi-cone angle ($\alpha$) of die, strap length ($l$) and the drawing speed ($v$). We simulated and analyzed the drawing process by finite element simulation (FEM) using Deform-3D software. Considering the least tensile stress as optimal standard, selecting 1/4 symmetry model of the wire with a diameter of 3.0 mm, setting two symmetrical planes and all particles moving in axial direction as well as only imposing displacement load on wire front profile, the optimized parameters and the distribution of the effective stress in wire during cold drawing was put forward. These parameters of die $\alpha = 4°$, $l = 0.4\,D$ ($D$ represents the diameter of the die) and $v = 10$ mm/s as well as the die height $h = 19.3$ mm were directly used in this work. The combined action of above external forces led to the uneven distribution of the effective stress of alloy wires, as shown in Fig. 1(b).

As-extruded Z2 wire with an initial diameter of 3.0 mm was drawn step by step at room temperature. After the first two passes with true strain ~3% each pass, the diameter of the wire was reduced to 2.949 mm and 2.899 mm, respectively. And then the drawing was performed step by step with true strain ~7% each pass, until ~91% ATS where fracture occurred frequently followed by the wire of 1.907 mm in diameter.

For the experimental wires with different strain, microstructure was observed and analyzed using an Olympus BHM optical microscope (OM) and a Tecnai G2 transmission electron microscope (TEM). For metallography, the samples were etched with acetic picral (0.84 g picric acid, 2 ml acetic acid, 7 ml $H_2O$, 14 ml ethanol) for 3–5 s. To obtain TEM image, the specimens were thinned by twin-jet electro polishing in a solution of 5 ml perchloric acid and 95 ml ethanol. In addition, the misorientation angle distributions of the Z2 wire with lower strain was also examined using electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM) with a field emission gun operated at 20 kV. The samples preparation for EBSD included carefully grinding with a series of SiC sand papers and diamond polishing, followed by electrochemical polishing in a solution of 5% nital acid in ethanol at 15–20 V for approx. 30 s and temperatures ranging from −20 to 0 °C depending on the alloy state.

Texture examinations were conducted on the sections aligned along the drawing direction by X'pert-PRD X-ray diffractometer (XRD) in the back-reflection mode with Cu $K_\alpha$ radiation. The specimens were prepared by ranging several wires to forming the dimension of 20 mm × 20 mm in the same direction, and then, the specimens were ground into a plane. We adopt here the usual convention of extrusion or drawing direction (ED or DD), transverse direction (TD) and normal direction (ND) to describe the orientation of samples deformed. The tensile tests were performed using CMT5105 electronic universal testing machine for 3–5 samples. Vickers microhardness (HV) was measured on the polished surfaces under the load of 300 g for 10 s, 5 points per sample for three samples.