

# Effects of ECAE temperature and billet orientation on the microstructure, texture evolution and mechanical properties of a Mg–Zn–Y–Zr alloy

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## ABSTRACT

Single-pass equal channel angular extrusion (ECAE) experiments of an extruded Mg–Zn–Y–Zr alloy with an intense initial basal texture were performed in two inter-perpendicular billet orientations and at 473 and 623 K. The study was aimed to determine the effects of ECAE temperature and billet orientation on the microstructure, texture evolution and mechanical properties of the ECAEed alloy. It was found that the grain refinement achieved through the single-pass ECAE in the Orient-I billet orientation (the normal direction (ND) of the extruded plate parallel with the ECAE exit direction) was more effective than that in the Orient-II billet orientation (the ND of the extruded plate perpendicular to the ECAE exit direction). The average grain sizes after ECAE at 473 K were much smaller than those after ECAE at 623 K. The pole figures of the alloy ECAEed at 473 K showed that most of the basal planes in the Orient-I and Orient-II samples were inclined about 40° and 35°, respectively, with respect to the longitudinal direction of the ECAE extrudate. However, for the alloy ECAEed at 623 K, most of the basal planes were parallel with the longitudinal direction of the ECAE extrudate. It was remarkable that the yield strengths of the alloy ECAEed at 473 K were lower than those at 623 K. The peculiar relationship between ECAE temperature and the mechanical properties of the alloy was ascribed to the texture evolution during ECAE.

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## 1. Introduction

Low density, high specific strength and good damping characteristics are attractive attributes of magnesium alloys. In recent years, these attributes have been increasingly utilized in transport, electronic and consumer products. Although wrought magnesium alloys are known for possessing better mechanical properties than the cast counterparts, the structural applications of extruded, forged and rolled magnesium alloys are yet quite limited, mainly because of their poor deformability at room and moderately elevated temperatures—an intrinsic characteristic of a metallic material with a hexagonal close packed (HCP) crystal structure [1,2].

The limited number of activated slip systems in an HCP-structured alloy results in the formation of a strong crystallographic texture during thermomechanical processing [2]. In the cases of pure magnesium and its alloys with an initial texture, a number of studies [1–4] were conducted to investigate their deformation characteristics under the conditions where the tensile or compressive

stress axis relative to the initial texture was specially arranged at various angles. Discrepancies in mechanical behaviour were found and explained in terms of the orientation relationship between the loading axis and the texture in the deformed materials.

The application of equal channel angular extrusion (ECAE) as an effective method for grain refining [5,6] has been extended from aluminium alloys to magnesium alloys to improve their strength and ductility [7–9]. Liu et al. [7] found significant improvements in the yield strength and ductility of the conventionally extruded Mg–3.3%Li alloy after 4-pass ECAE at 523 K with Route A and Route Bc (Route A is defined as that when the billet is extruded without rotation between passes; route Bc is defined as a rotation of 90° in the same direction between passes), and the improvements with Route A were greater than those with Route Bc. In contrast to these findings, Mukai et al. [8] found that, for the AZ31 alloy after 8-pass ECAE at 473 K with Route Bc, its tensile yield strength was slightly lower than the conventionally extruded counterpart, although its elongation to failure was twice as large as that of the conventionally extruded alloy. Kim et al. [9] obtained similar results from the tensile tests of the AZ61 alloy after the conventional extrusion and then ECAE at 548 K with Route Bc. The peculiar mechanical behaviour of these magnesium alloys was attributed to the strong texture developed during the ECAE process.

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In recent years, the interest in Mg–Zn–Y(–Zr) alloys with an icosahedral quasicrystal phase, *i.e.* the I-phase formed upon solidification, has been growing, because these alloys, after going through thermomechanical processing such as hot extrusion, possess desired yield and ultimate tensile strengths both at room temperature and in a low temperature range, typically up to 473 K [10]. To improve the strength and ductility of Mg–Zn–Y(–Zr) alloys further, ECAE has been tried [11,12]. Zheng et al. [11] reported that, while the conventionally extruded Mg–11Zn–0.9Y (wt%) alloy did not exhibit a marked improvement in ductility after ECAE with Route Bc at 523–473 K, the yield strength of the ECAEed alloy was significantly higher than that of the conventionally extruded alloy, and the yield strength increased as the number of ECAE passes increased. The authors [12] also reported that, after ECAE under the same condition, the ductility of the Mg–5.9Zn–0.9Y–0.2Zr (wt%) alloy increased with increasing ECAE passes, while its yield strength was lower than that of the conventionally extruded alloy and decreased significantly as the number of ECAE passes increased.

It appears that the mechanical properties of extruded magnesium alloys, especially those of extruded Mg–Zn–Y–Zr alloys, after multi-pass ECAE, do not necessarily exhibit improved strength and ductility as expected. It is likely that ECAE process parameters, such as temperature, and the initial crystal orientation of the billet with respect to the shear stresses imposed during ECAE are influential on the evolution of texture and thus on the mechanical properties of the alloys tested uni-axially under tensile or compressive loading [1–4]. In the open literature, there are few reports on the relationship between ECAE process parameters, billet orientation and the resultant mechanical properties of magnesium alloys.

The present study was aimed at determining the effects of ECAE temperature and the orientation of the billet with an initial texture on the microstructure, texture evolution and resultant mechanical properties of magnesium alloys. To reach this aim, a conventionally extruded Mg–Zn–Y–Zr plate with an intense initial (0002) basal texture was subjected to single-pass ECAE experiments at 473 and 623 K and in two inter-perpendicular billet orientations. Microstructure and texture analyses were performed and the results were used to explain the mechanical behaviour of the alloy exhibited during tensile testing.

## 2. Experimental details

The alloy with a chemical composition of Mg–6.43%Zn–1.0%Y–0.48%Zr (wt%) was prepared from pure magnesium (99.9%), pure zinc (99.99%), Mg–25%Y and Mg–33%Zr master alloys using an electric resistance heating furnace in an SF<sub>6</sub> and CO<sub>2</sub> atmosphere. The molten alloy was poured into a cylindrical metal mould with a diameter of 100 mm. The as-cast ingot was machined into extrusion blocks and extruded at 663 K in the conventional manner into plates with a rectangular cross-section of 14 mm × 60 mm. The extrusion ratio applied was about 10:1.

The die used in the ECAE experiments had two equal channels with a square cross-section of 12 mm × 12 mm and an intersecting angle of 90°, as illustrated in Fig. 1. With such an ECAE die setup, an equivalent strain of 1.05 per pass could be applied to the billet [5]. The ECAE billets with a length of 100 mm and a square cross-section of 12 mm × 12 mm were cut from the middle part of the extruded plate with the cross-section of 14 mm × 60 mm, using electro-discharge machining. The orientations of the billets for the ECAE experiments with respect to the plate were divided into two groups, one with the normal direction (ND) of the extruded plate parallel with the X direction of ECAE (designated as Orient-I) and another with the ND of the plate parallel with the Y direction of

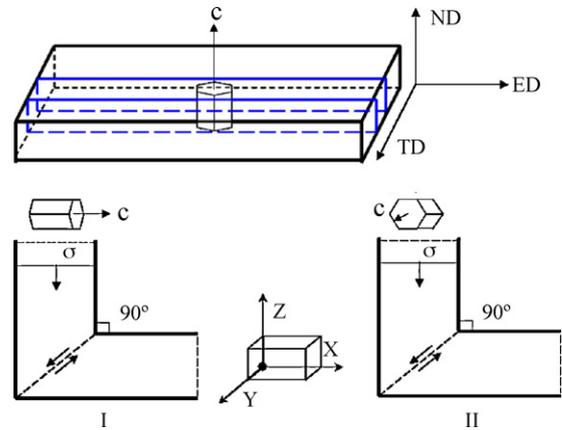


Fig. 1. Schematic of the ECAE die setup used for the ECAE experiments on the conventionally extruded Mg–Zn–Y–Zr alloy in the Orient-I and Orient-II billet orientations. The ECAE billets were machined from the extruded plate along the extrusion direction (ED).

ECAE (designated Orient-II), as shown in Fig. 1. Before a billet was inserted into the ECAE entry channel, lubrication was applied to the billet to decrease its friction with the channel inner wall. The billet was held in the entry channel at test temperature for 15 min before ECAE started. Single-pass ECAE experiments were performed at 473 and 623 K and at a constant ram speed of 5 mm/min. After ECAE, the extrudate was taken out from the exit die and quenched in water immediately.

The ECAEed extrudate was sectioned on the X–Z plane (see Fig. 1) at the center for metallographic examination. Metallographic samples were polished to a mirror finish, etched in a glycol-diluted nitric acid solution, and examined using an optical microscope. The grain size  $d$  was estimated using the linear intercept method. Crystallographic texture measurements were made on the ED–TD plane in the conventionally extruded plate, and on the X–Z plane (not given in the paper) and the X–Y plane of the ECAE extrudate. The pole figures of {0002} were measured up to a reflection angle of 70° using an X-ray diffractometer.

Tensile specimens with a gauge length of 5 mm and a rectangular cross-section of 2 mm × 3 mm were machined from the extrudate with their longitudinal axes in parallel with the X direction of ECAE samples. Tensile tests were performed at room temperature and at an initial strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$ .

## 3. Results

### 3.1. Microstructural characteristics

#### 3.1.1. Initial microstructure and texture

The original microstructure of the as-cast ingots before conventional extrusion was shown in Fig. 2a, with a small number of secondary I-phases distributed in the matrix [10–12]. The initial microstructure of the conventionally extruded plate before ECAE is shown in Fig. 2b. It can be seen that the microstructure is inhomogeneous with some extrusion strips and small grains. The strips, which are some elongated grains oriented in the extrusion direction (ED as shown in Fig. 1), are dispersed in the matrix with small grains of about 10 μm. In addition, the broken second-phase particles (*i.e.* the black particles in Fig. 2b) scatter in the matrix. The pole figures of the extruded plate on the ED–TD plane are given in Fig. 2c, showing a typical texture of an HCP-structured metal after extrusion deformation, *i.e.* the c-axes of most crystals being approximately parallel with the normal direction (ND) of the extruded plate.

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