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# Evolution of twinning in extruded AZ31 alloy with bimodal grain structure

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ABSTRACT

Twinning in extruded AZ31 alloy with a bimodal grain structure is studied under compression along the extrusion direction. This study has combined in-situ measurements during the compression tests by Synchrotron Radiation Diffraction and Acoustic Emission techniques and the evaluation of the microstructure and texture in post-mortem compression samples deformed at different strains. The microstructure of the alloy is characterized by the coexistence of large areas of fine dynamic recrystallized grains and coarse non-recrystallized grains elongated along extrusion direction. Twinning occurs initially in large elongated grains before the macroscopic yield stress which is controlled by the twinning in equiaxed dynamically recrystallized grains.


## 1. Introduction

Extruded magnesium alloys exhibit a great variety of microstructures depending on the processing and microstructural parameters such as extrusion temperature, extrusion rate, extrusion ratio, composition of the alloy, presence of second-phase particles and/or precipitates in the cast alloy, initial grain size of the cast alloy.… Under particular conditions, the dynamic recrystallization (DRX) process during extrusion is hindered and it is not completed in the whole extruded profile. As a consequence, the coexistence of elongated non-recrystallized grains (non-DRXed) within areas of fully dynamically recrystallized (DRXed) fine grains can be observed in the microstructure [1–9]. Such a bimodal grain structure has a strong influence on the mechanical properties of extruded magnesium alloys. The coarse, non-DRXed grains are preferentially oriented with their basal plane parallel to the extrusion direction. Thus, these grains contributes to the hardening during tensile deformation along the extrusion direction owing to the impossibility of the activation of basal slip and tensile twinning [7,10,11]. At the same time, they significantly reduce the ductility of the extruded alloys [6,9]. Surprisingly, the compression properties of alloys with bimodal grain structures, particularly the occurrence of twinning during ED compression, are barely studied.

It is well-known that magnesium alloys develop a strong texture during the extrusion process. Grains orient their basal planes parallel to the extrusion direction [10,12–14]. During compression along the extrusion axis, a significant part of strain is accommodated by the $\{10\bar{1}2\}$ $\langle 10\bar{1}\bar{1}\rangle$extension twinning. The compression curves exhibit two significant characteristics. First, a yield plateau takes place after yield stress, especially in fine-grained alloys. Barnett et al. [15] have explained that this plateau is formed by a band of twins that propagates progressively over the entire sample, similarly to a Lüders band. The back stress generated by twins in the neighbouring grains enhances the propagation of twinning along the compression sample. This effect becomes more pronounced with decreasing grain size [16,17] or for low($<15^\circ$) grain boundary misorientations [18]. Once the band traverses the entire gauge length, the sample begins to harden. This leads to the second characteristic effect, namely to the convex shape or "S shape" of the deformation curve [16]. During twinning, the basal plane within twins rotates 86° with respect to the initial orientation [19]. The new orientation inhibits the activation of both the basal slip and the extension twinning. This results in significant work hardening of the sample.

Twinning strongly depends on grain size, being Hall–Petch slope higher than that corresponding to deformation controlled by slip [20,21]. Therefore, the twinning process would be completely different in fine DRXed grains or coarse non-DRXed grains. Nevertheless, recent statistical studies [22,23] have revealed that, while the number of twins per grain is strongly related to grain size, the probability of twinning is

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independent of the grain diameter, at least when the latter is larger than a few microns.

The objective of the present paper is to explore the deformation mechanism in an extruded AZ31 alloy with a bimodal grain structure during uniaxial compression tests along the extrusion direction. For this purpose, in-situ measurements by synchrotron radiation diffraction and acoustic emission techniques during the compression tests were carried out. Additionally, the microstructure and texture of the deformed samples were also studied.

## 2. Experimental Procedure

The cast AZ31 alloy was extruded at 250 °C employing an extrusion ratio of 18:1. Microstructural characterization (grain size and volume fraction of DRXed and non-DRXed areas) of the alloy was carried out by optical microscopy. Metallographic preparation consisted of mechanical polishing and etching in a picral solution (5 g of picric acid, 5 ml of water and 25 ml of ethanol.).

Micro-texture analysis of the as-extruded as well as the compressed samples was done by Electron Backscattered Diffraction (EBSD) technique attached to the SEM equipment. Data of EBSD were analyzed using Channel 5 EBSD software. The samples have a cylindrical symmetry where the reference directions are: ED (extrusion direction) and RD (radial direction). High angle boundaries >10° and low angle boundaries >2° are represented by black and white lines, respectively. The specimens for EBSD were ground with 320, 600, 1200 and 2000 grit SiC paper, polished with a solution of colloidal silica in ethanol and finally chemically etched [24].

Transmission electron microscopy was carried out to study the microstructure of the alloy in the extruded condition, before yield stress (225 MPa) and after compression (0.8% of plastic strain). Specimens for TEM observation were prepared by electrolytic polishing using a reactive mixture of 25% nitric acid and 75% methanol at −30 °C and 20 V. Then, ion milling at liquid nitrogen temperature was used to remove the fine oxide film formed on the surface during electrolytic polishing.

Global texture analysis was also performed in AZ31 alloy in the as-extruded state as well as after compression deformation using neutron diffraction. Five complete pole figures were measured at STRESS-SPEC located at FRM II (Garching, Germany) with a wavelength of 1.74 Å from Ge (311) monochromator using the robot system [25].The reference system was selected with the extrusion direction parallel to the Z axis.

Cylinders (5 mm in diameter and 10 mm length) for compression tests were machined from the extruded bar with its longer dimension parallel to the extrusion direction. Compressive tests were performed at room temperature in a universal tensile machine under a constant cross-head speed corresponding to an initial strain rate of $10^{-3}$ $s^{-1}$.

The in-situ synchrotron diffraction experiments were carried out on the P07 beamline of PETRA III, at the DeutschesElektronen-Synchrotron (DESY) [26]. The compression sample where deformed in a Bähr 805A/D dilatometer modified for synchrotron experiments. The compression test was performed in argon flow. The diffraction patterns were recorded using an exposure time of 0.5 s by a Perkin-Elmer XRD 1622 flatpanel detector with an array of $2048^2$ pixel, with an effective pixel size of 200 × 200 $\mu m^2$. The beam energy was 100 keV, corresponding to a wavelength of 0.0124 nm. $LaB_6$ was used as a reference to calibrate the acquired diffraction spectra. The detector-to-sample distance was 1620 mm. Conventional line profiles were obtained by azimuthal integration of the Debye-Scherrer rings. Cylinders of 5 mm in diameter and 10 mm of length were deformed in compression at strain rate of $10^{-3}$ $s^{-1}$. The synchrotron radiation beam was positioned at the centre of the sample with the gauge volume defined approximately by the beam section (1 × 1 $mm^2$) and the cylinder diameter.

The elastic strain for each orientation can be calculated by the shift in the position of the diffraction peak, as:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} \quad (1)$$

where $d_{hkl}$ and $d_{0,hkl}$ are the planar spacing of the hkl plane in the stressed and stress-free crystal. The lattice spacing and the diffraction angle θ are related through Bragg's law.

The AE activity during deformation tests was monitored by a computer-controlled MICRO-II system developed by Physical Acoustic Corporation (PAC). A piezoelectric MIDI-410-61 sensor (ZD RPETY-DAKEL) with a diameter of 6 mm was used. The AE sensor was attached on the sample with a help of a clamp. High signal/noise ratio was ensured by using a 60 dB preamplifier. A threshold-level detection of the recorded AE signal was performed to achieve a comprehensive set of AE parameters. The threshold level was set as 27 dB.

## 3. Results

Fig. 1(a,b) shows the microstructure of the AZ31 alloy along the extrusion direction. The microstructure exhibit a bimodal grain structure of fine equiaxed DRXed grains and coarse non-DRXed grains elongated along the extrusion direction. The volume fraction of elongated grains is 12%. The grain size of DRXed grains, evaluated using the mean intercept length method, is 3 μm. Fig. 1(c–e) shows the Orientation Image Mappings (OIM) of the magnesium grains and the (0002) pole figures obtained from the OIM image. The alloy exhibits a intense fiber texture with the basal plane parallel to the extrusion direction as it is expected for extruded magnesium alloy bars. Therefore, (0002) planes are located along the RD axis in Fig. 1d. However, while the coarse non-DRXed grains are oriented with their basal plane parallel to the extrusion direction (see grain A and B in Fig. 1c), the DRXed grains can be found slightly tilted from this orientation. The non-DRXed grains contribute mainly to generate the intense basal texture as it is shown in Fig. 1e where it is represented the average texture. The macroscopic texture was also measured using neutron diffraction and $\{10\bar{1}0\}$ and (0002) pole figures are shown in Fig. 2a. It is interesting to point out that the reference system used in this case is different from the microtexture and the extrusion direction is parallel to the Z direction. The macrostructure measured with neutron diffraction is naturally in agreement with microtexture measured by EBSD with a strong fiber texture with the prismatic and basal planes perpendicular and parallel to the extrusion direction.

Fig. 1f and g show the TEM bright field images of the DRXed and non-DRXed grains, respectively. DRXed grains are equiaxed, with a low density of dislocations and separated by high angle grain boundaries. On the other hand, non-DRXed grains are elongated along the extrusion direction as it was observed by optical microscopy. They are highly deformed with a high density of dislocations and subgrains. The small rotation between subgrains is observed in the selected area electron diffraction (SAED) pattern from the $B = [11\bar{2}0]$ zone axis where diffracted spots of each family of planes are slightly tilted between each subgrains.

Fig. 3 shows the true stress-true plastic strain curve for the extruded AZ31 alloy compressed along the extrusion direction. The compressive strain is similar to those obtained by Barnett et al. [16] in the same material with similar grain size in the DRXed grains. The alloy shows an elastic behaviour up to 250 MPa. After yielding, the stress is almost constant during further 4% of strain. Beyond this value, the stress and the work-hardening increase again. The shape (S-shape) of the compression curve is typical for the twinning-controlled deformation.

Fig. 4a shows the Debye-Scherrer rings obtained before the compression test. After integration along the axial and radial directions, diffraction patterns as a function of 2θ are obtained (Fig. 4b). In the axial