ACOUSTIC EMISSION ANALYSIS OF EXTRUDED AZ31 WITH VARYING GRAIN SIZE

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Acoustic emission was monitored and analysed during tensile tests at room temperature of extruded magnesium alloy AZ31 (Mg-2.9wt.%Al-0.98wt.%Zn-0.29%Mn). Hydrostatically and indirectly extruded rods were water-cooled or air-cooled, respectively, and exhibit different microstructures, e.g. a variation of the grain size, as well as different mechanical properties. The validity of the Hall-Petch relation was proven. The results are explained in terms of dislocations glide and deformation twinning during plastic deformation.

K e y w o r d s: acoustic emission, extrusion, tensile testing, Hall-Petch relation, AZ31

1. Introduction

Magnesium and its alloys belong to the lightest construction metals but show poor strength and formability [1]. Extrusion is a promising technology of processing long and thin-walled profiles with improved mechanical properties [2, 3]. The process parameters (billet temperature, extrusion ratio, profile speed, cooling) significantly influence the micro-structural evolution, e.g. in terms of grain size, grain size distribution and precipitation in the material, and consequently, lead to changes in mechanical properties of the resulting profile [4].

Grain boundaries (GB) are the most important sub-structural defects of the crystalline lattice. Therefore, grain size and grain geometry play a very important role in plastic deformation of polycrystalline materials. The deformation of most polycrystals can be usually described by the Hall-Petch relation

$$\sigma = \sigma_0 + kd^{-1/2}, \tag{1}$$

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where d is the grain size parameter, σ is the deformation stress (e.g. the conventional yield stress σ_{02}), σ_0 is the friction stress necessary for dislocation movement through the lattice and k is a constant which characterizes the strengthening effect of GB [5, 6]. It was shown [7] that the Hall-Petch relation (1) could generally be interpreted considering GB as strong obstacles for the dislocation slip, however, the validity of the relation is found only within a certain range of grain sizes ($d > 10$–100 μm). Beyond such a range a deviation from the Hall-Petch relation can be given by a more complex character of the plastic deformation which may be affected by other mechanisms (GB sliding, diffusion creep, suppression of pile up formation in ultrafine-grained materials etc.) [8].

The acoustic emission (AE) is defined as transient elastic waves generated by sudden release of energy due to a local dynamical change of the material structure like the dislocation slip and twinning [9]. *In-situ* measurements of AE during tensile or compression testing is an appropriate tool to study dynamic processes involved in plastic deformation of magnesium and its alloys.

The present paper uses rods from magnesium alloy AZ31 after indirect and hydrostatic extrusion. The varying extrusion process as well as the cooling condition (water-cooling or air-cooling) directly after extrusion is the reason for different resulting microstructures in terms of the grain size. The influence of this microstructure on the mechanisms of tensile plastic deformation is studied using *in-situ* measurements of the AE parameters.

2. Experimental

Four rod profiles of magnesium alloy AZ31 (Mg-2.9wt.%Al-0.98wt.%Zn-0.29%-Mn) were used for this study. Extrusion trials were carried out at a billet temperature of 300 ℃ using billets after a homogenization heat treatment of 12 h at 350 ℃ followed by air cooling [10]. Three profiles were produced by hydrostatic extrusion. One profile was obtained at an extrusion rate of 9 m/min including water-cooling at the die exit. For a second profile the trial was repeated without water-cooling. A third trial also used water-cooling but the extrusion rate was higher with 37 m/min. One additional profile was produced using indirect extrusion and air-cooling. Products by hydrostatic and by indirect extrusion were used as experimental materials.

During hydrostatic extrusion the pressure is put onto the billet by using a hydrostatic medium. Thus the pressure from a ram is put onto the medium and leads to the hydrostatic pressure from all sides onto the billet to make it flow through the die. During indirect extrusion the billet is put into a container and deformation is realized by pressing a hollow ram with a die in front of it into the billet. More detail on the set-up may be found in [2, 3, 11]. Extrusion process at an initial billet temperature of 300 ℃ and an extrusion ratio 1 : 28 in case of the hydrostatic extrusion and 1 : 23 in case of the indirect extrusion with a profile speed of 8 m/min was chosen. Details about the influence of the processing on the development on the profile microstructure can be found elsewhere [12].

The specimens (diameter 6 mm, length 60 mm, following DIN 50 125) were deformed in a universal testing machine Zwick® Z50 in tension at room temperature (RT) at a constant strain rate of 10^{-3} s^{-1}. The microstructure of materials was investigated by an optical microscope. For sample preparation, an etchant based on picric acid was used [13].

The computer controlled DAKEL-XEDO-3 AE system was used to perform monitoring (two-threshold-level detection recommended by an ASTM standard [14]). More information about the XEDO system may be found in the web [15]. A miniaturized MST8S piezoelectric transducer (diameter 3 mm, almost point AE detection, a flat response in a frequency band from 100 to 600 kHz, sensitivity 55 dB ref. 1 V_{ef}) was attached on the specimen surface with the help of silicon grease and a spring. The total gain was 94 dB. A comprehensive set of AE parameters involving count rates at two threshold levels (simple amplitude discrimination), AE rms voltage, event count, event amplitude and signal waveforms were evaluated. The AE signal sampling rate was 4 MHz, the threshold voltage for the total AE count N_{C1} and for the burst AE count N_{C2} were 730 and 1450 mV, respectively. The full scale of the A/D convector was ± 2.4 V. The threshold voltage for the AE event start and the AE event end were 1450 and 965 mV, respectively. The dead-time was 1000 μs [16, 17].

3. Results

Table 1 gives an overview on the average grain size which is obtained for rods from different extrusion trials. Typical micrographs of the extruded AZ31 are shown in Fig. 1. The vertical direction in all micrographs represents the direction of the extrusion and of the applied stress. In all cases, the microstructure after extrusion (Figs. 1a,b) exhibits a limited number of inhomogeneities and the grain size distribution shows a wide range. The grains are elongated in the extrusion direction. The microstructure after the tensile test (Figs. 1c,d) reveals twins which are visible as thin bands in the grains and the angle between twins is about 60° (Fig. 1d), in accordance with the hexagonal structure.

Table 1. Conditions of the preparation and average grain size in alloy AZ31

Extrusion	Cooling	Grain size [μm]	Extrusion rate [m/min]
Hydrostatic	Water	4 ± 1	9
Hydrostatic	Air	6 ± 1	9
Hydrostatic	Water	8 ± 1	37
Indirect	Air	22 ± 7	9

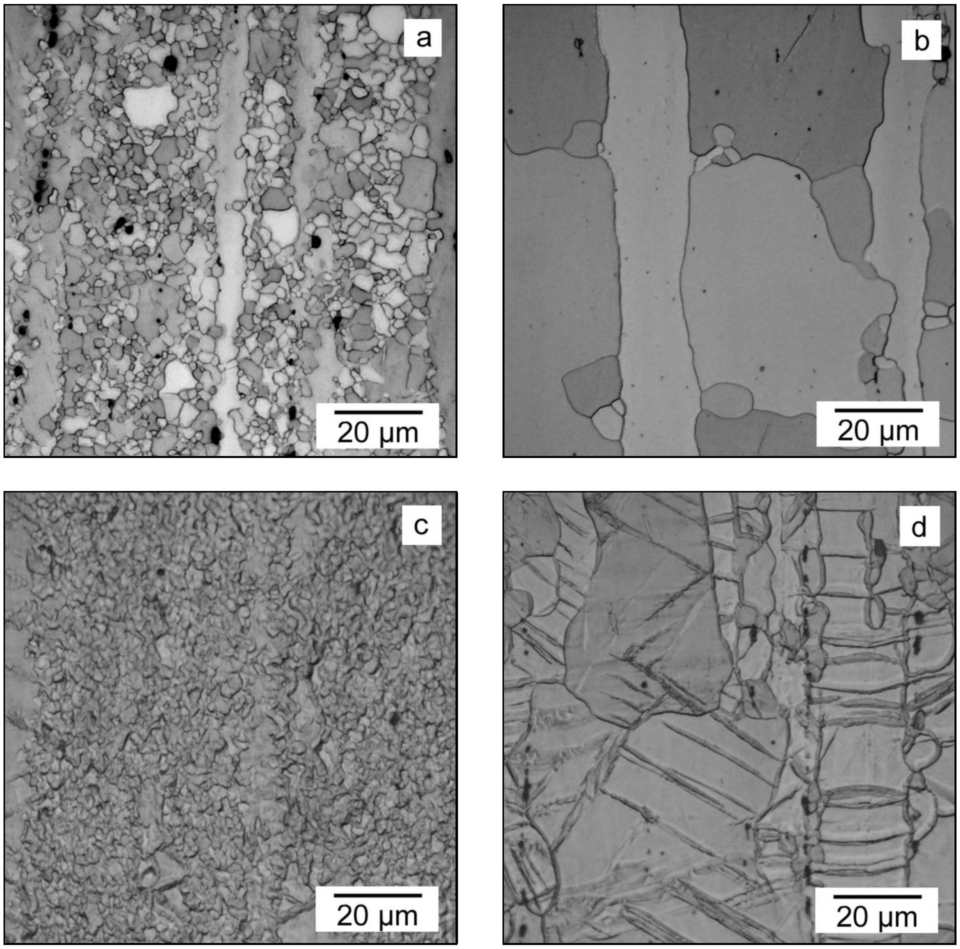

Fig. 1. Micrographs of extruded AZ31 alloy: (a) hydrostatic, grain size 4 μm, (b) indirect, grain size 22 μm, (c) hydrostatic, grain size 4 μm, after tensile testing, (d) indirect, grain size 22 μm, after tensile testing.

The dependence of the yield stress $\sigma_{0.2}$ on the $d^{-1/2}$ is depicted in Fig. 2. The friction stress σ_0 and the constant k, derived from the Hall-Petch relation (1), are equal to 172 MPa and 5.1 MPa/mm$^{-1/2}$, respectively.

The stress-strain curves correlated with the time dependences of the AE count rates at the both threshold levels ($\dot{N}_{C1}$ and $\dot{N}_{C2}$) are shown in Figs. 3–6. The correlation between the testing time and the engineering strain is given by the applied strain rate. The deformation curves show a fairly large elongation to fracture of about 0.22 and the yield stress $\sigma_{0.2}$ decreases with increasing grain size. The AE

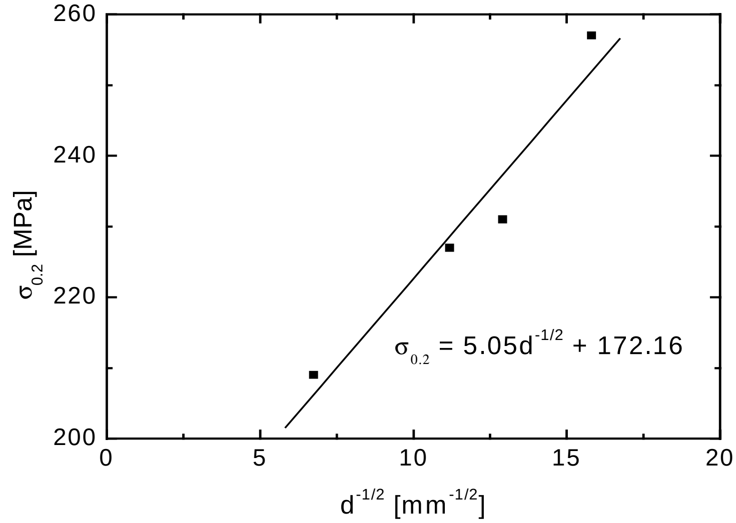

Fig. 2. The dependence of the yield strength $\sigma_{0.2}$ on the $d^{-1/2}$.

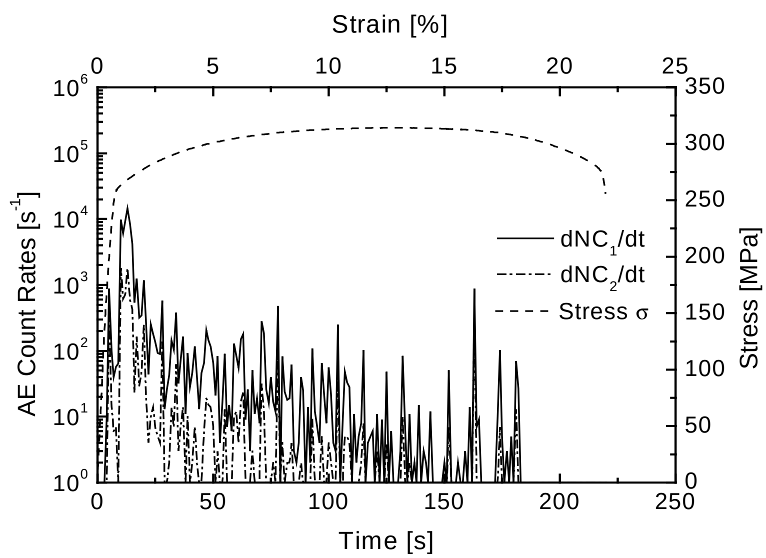

Fig. 3. Stress-strain and AE count rates-time curves for extruded AZ31 alloy with grain size 4 μm.

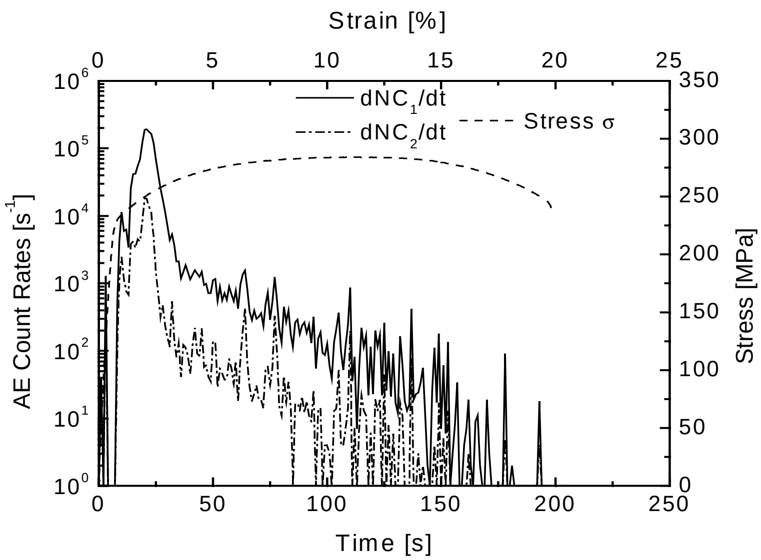

Fig. 4. Stress-strain and AE count rates-time curves for extruded AZ31 alloy with grain size 6 μm.

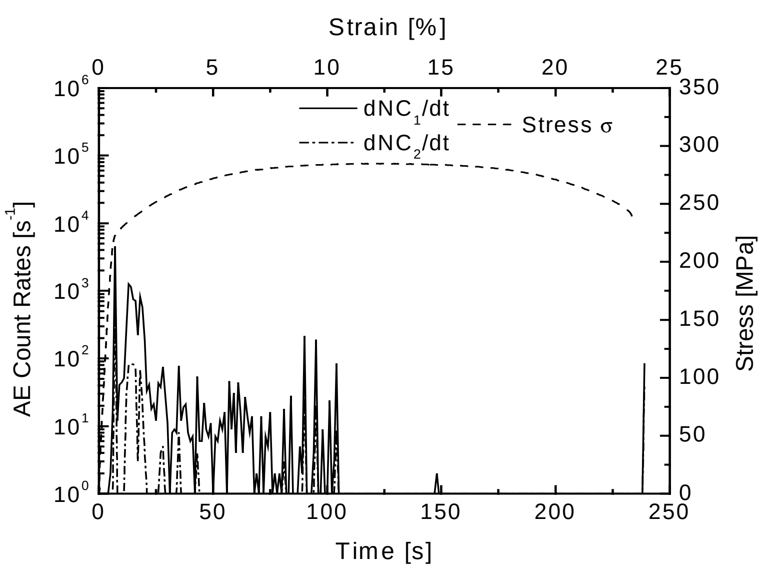

Fig. 5. Stress-strain and AE count rates-time curves for extruded AZ31 alloy with grain size 8 μm.

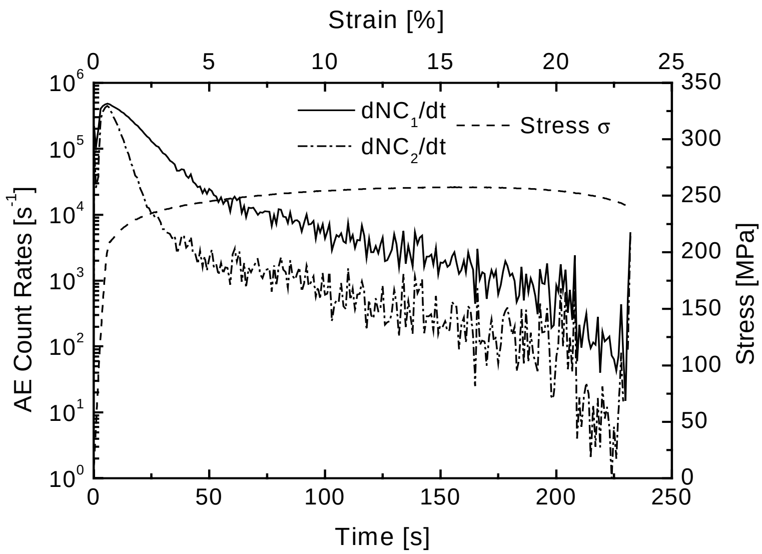

Fig. 6. Stress-strain and AE count rates-time curves for extruded AZ31 alloy with grain size 22 μm.

count rates exhibit a characteristic peak related to the macroscopic yield point, which is followed by a decrease of the AE activity during further strain hardening. The AE response on the applied stress is significantly higher for the air-cooled rods with a grain size of 6 and 22 μm than for the water-cooled rods with a grain size of 4 and 8 μm, respectively.

4. Discussion

The extruded AZ31 alloy exhibits an increase of the $\sigma_{0.2}$ with decreasing grain size in accordance with the Hall-Petch relation (1). The AE activity is easily correlated with the plastic behaviour of the AZ31 alloy, shown in Figs. 3–6. The characteristic peak of AE is usually explained by a massive dislocation multiplication which is an excellent source of AE. The following decrease of the AE count rates may be ascribed to an increasing density of forest dislocations which reduces the flight distance and the free length of moving dislocations [9]. A very strong AE in comparison to e.g. cubic materials like Al and its alloys [18] is caused by twinning as the second important deformation mechanism.

The higher AE activity in coarse-grained structure (Fig. 6) than in fine-grained structure (Fig. 3) is connected with a lower density of grain boundaries and with large twins which can release higher energy in form of AE. The local change of the lattice orientation by deformation twinning leads to additional dislocation glide in regions favorably re-oriented for the basal slip [19, 20].

Non-monotonic dependence of AE response on the grain size (AE is higher for grain size 6, 22 μm than for 4, 8 μm) is probably given by cooling conditions after extrusion of AZ31 alloy. Water-cooled samples (4, 8 μm) may contain more dislocations and have higher stacking fault energy than air-cooled samples due to more rapid cooling. High density of dislocation and high stacking fault energy are necessary conditions for deformation in non-basal slip systems which reduce AE signals. This result is very interesting for further investigations because the elongation to fracture for all samples was approximately the same, about 0.22.

5. Conclusions

Acoustic emission has been measured and analysed during tensile test of hydrostatic and indirect extruded AZ31 alloy with different grain size at room temperature. The average grain sizes were 4, 6, 8 and 22 μm. In all samples the yield stress $\sigma_{0.2}$ increases with decreasing grain size in accordance with the Hall-Petch relation. The AE count rates exhibit a characteristic peak close to the macroscopic yield point followed by a decrease of the AE activity. Dependence of the AE response on the grain size has non-monotonic character (with respect to the magnitude of AE count rates). This result is explained by different cooling conditions after extrusion which influenced the density of dislocations and stacking fault energy. Dislocation glide can locally reorient the crystal lattice and leads to activating fresh slip systems and to the increase of AE activity.

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