

In-situ investigation of deformation mechanisms in various magnesium alloys by X-ray diffraction and acoustic emission

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ABSTRACT: The influence of the LPSO (long period stacking ordered) phase orientation on the deformation mechanisms of magnesium alloys has been investigated by X-ray diffraction (XRD) and acoustic emission (AE) measurements. The adaptive sequential k -means analysis (ASK) method, offering identification of the dominant deformation process (basal, non-basal slip, twinning) in a given time period, has been used for AE data evaluation. The results indicate that the kinking mechanism exhibits a significant dependence on the orientation of the LPSO phase with respect to the loading axis.

1 INTRODUCTION

1.1 Current development in field of high-strength magnesium alloys

Magnesium alloys, having excellent strength to weight ratio, represent a highly interesting material for transportation industry, where the fuel saving achieved through weight reduction belongs to the most important tasks. Nevertheless, their widespread application is limited by several factors, as generally low formability and yield strength at ambient temperature, strong degradation of mechanical properties above 200 °C. There are several ways for improving the mechanical performance of magnesium alloys, including grain size refining and alloying, which leads precipitation formation, which enhance the stability of the microstructure.

The Mg-Zn-RE LPSO (LPSO – long period stacking ordered) structures are fundamentally long-period stacking variants of a hexagonal-closed-packed (hcp) structure of the Mg crystal. The recent investigations shows, that yield strength exceeding 300 MPa can be achieved using technologically simple hot-extrusion process for this class of materials [1]. The high-temperature properties of Mg/LPSO alloys are also very promising – only a slight decrease of strength was found in the temperature range of 20 - 300 °C.

Despite the efforts of the scientific community in the last decades the elucidation of the deformation mechanisms of magnesium alloys with LPSO phase is still a challenging task. There is a general agreement that there are two main processes besides the dislocation slip, which contribute to the plasticity: formation of “*deformation kinks*” and *twinning*.

Deformation kinks form, when a large stress acts parallel to basal slip planes. Owing to the low critical resolved shear stress (CRSS) for basal slip, avalanche-like motion of (0001) basal dislocations takes place, accompanied by elastic buckling of planes. During the ongoing loading, basal dislocations with opposite sign nucleate. They start to move against the dislocations from the first “*wave*”, which leads to the kink formation.

The kink formation significantly depends on materials parameters (e.g. shape and orientation of LPSO phase, grain size, texture) and experimental conditions (loading direction, temperature etc.). The role of the non-basal slip in the kink formation has not yet been clarified. However, the activation of $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ prismatic slip has been reported, but rather at higher testing temperatures [2].

Deformation twinning is the main mechanism accommodating deformation out of the basal plane, particularly at temperatures below 200 °C. In magnesium, the $\{10\bar{1}2\}$ -type extension twinning, associated with extension along the c -axis and reorientation of the lattice by 86.3° and $\{10\bar{1}1\}$ -type compression twinning, resulting in contraction along the c -axis and tilting of the lattice by 56° are the most often reported and studied twinning



mechanisms . In the case of Mg/LPSO alloys, twinning was found to operate for particular sample compositions and texture. However, revealing the conditions for its activation needs further investigation.

1.2 *Experimental methods exploitable for investigation of Mg/LPSO alloys*

Owing to their complex microstructure the experimental study of the deformation mechanisms in Mg/LPSO alloys is rather difficult. The deformation kinks, slip bands and twinning are usually studied by light optical microscopy (LOM). In addition, there are several investigations of the dislocation structure performed by transmission electron microscopy (TEM). However, the study of less populated dislocation types (e.g. prismatic or 2nd order pyramidal ones) by TEM is not an easy task. The SEM experiments are usually limited to documenting of the microstructure by means of secondary and/or back-scattered electrons; the orientation mapping using electron-backscattered diffraction (EBSD) technique is scarce. Generally, the microscopic methods usually examine only a small volume of the material and the results are not always representative for the whole specimens.

Statistically representative data about the dislocation structure and twinning in magnesium alloys can be obtained from both *diffraction experiments* and *acoustic emission* (AE) tests [3]. The total twinned volume is determined from the intensity variations of particular peaks, caused by the crystal lattice reorientation during the twinning [4]. The diffraction measurements can take place both in *ex-situ* and *in-situ* regime. In the latter case, the combination of the diffraction experiments with the AE testing can result in a complex characterization of the plasticity [5].

The *acoustic emission* (AE) is an efficient experimental tool for *in-situ* testing of deformation mechanisms in magnesium alloys . The main advantages of the method are a fine time resolution and sensitivity to twin nucleation and collective dislocation motion. Nevertheless, the differentiation between the particular signal types is a difficult task due to their simultaneous appearance. Various statistical methods (*k*-means, fuzzy *c*-means, neuron analysis) have been worked out to analyse the AE data. The recent one, the so called adaptive sequential *k*-means analysis (ASK) by Pomponi and Vinogradov [6] appears very promising for determination of the dominant deformation mechanisms at the various stages of deformation in magnesium alloys.

2 EXPERIMENTAL

2.1 *Material*

The Mg₉₇Y₂Zn₁(at.%) alloy was fabricated by melting in an electric resistance furnace using high purity Mg and Zn elements and a Mg-Y master alloy. Ingots were cast by pouring the liquid metal into a cylindrical steel mould of diameter 42 mm. Cast billets were extruded to flat bar at 400 °C with a reduction ratio of 18:1. This rectangular bar was sectioned to 6x4x4 mm³ sized specimen, which were used for compression tests.

2.2 *Microstructural characterization*

Prior the optical microscopy, the specimens were sequentially ground on SiC grinding papers (800, 1200, 2000, 4000) with water. Then coarse polishing was performed using 3- μ m and 1- μ m diamond abrasive. In the next step the specimens were etched with nital 5% and electrolytically polished in a solution of picric acid (100 ml ethanol, 18 ml H₂O, 6 ml glacial acetic acid, 12 g picric acid 98%) for 7s. Texture analysis was performed by the back-Schulz reflection method, using a SIEMENS TM Kristalloflex D5000 x-ray diffractometer equipped with an Eulerian cradle.

2.3 *In-situ acoustic emission (AE) and synchrotron diffraction (SD)*

The deformation test has been performed in compression at room temperature, using a strain rate of 10⁻³ s⁻¹. In the case of ED the test was stopped after reaching 20% of strain in order to avoiding the destroying of the AE sensor. A miniature (2 mm in diameter) broadband AE sensor (flat frequency response in the range 100 – 500 kHz) from Dakel company was mounted on the specimen using vacuum grease and a clamp. The AE was amplified by 40 dB in the frequency range 100 – 1200 kHz using a 2/4/6-type preamplifier manufactured by Mistras Corporation. The AE acquisition (PCI-2 board from Mistras Corp.) took place in a so-called data streaming regime, where the data were recorded continuously with 18 bits amplitude resolution and 2 MHz sampling rate.

In-situ synchrotron diffraction experiments were carried out during separate compression test on the beamline EDDI at BESSY, Berlin, Germany. The energy range of the synchrotron white beam was from 10 to 135 keV. The diffraction angle was around $2\theta = 9.74^\circ$. The resulting gauge, determined by slit setting, was a rhomboid prism of $0.5 \times 0.5 \times 5.6$ mm. The gauge volume was always positioned in the centre of the cylinder. The sample was tilted within the scattering plane between $\psi = 0^\circ$ (axial direction) and $\psi = 90^\circ$ (radial direction), where ψ is defined as the angle between the scattering vector and the extrusion axis. The use of a white beam allowed the entire diffraction pattern to be collected for each ψ angle. Individual diffraction peaks were obtained for each diffraction pattern and fitted with a Gaussian curve to determine the peak position and peak intensity.

3 RESULTS AND DISCUSSION

3.1 Initial microstructure

Figure 1 shows the microstructure in the extrusion direction (ED) and transverse direction (TD). The LPSO phase, having long fiber shape, has deformed during the extrusion process. Its volume fraction was estimated as 19%. The magnesium matrix exhibits a bimodal grain structure, consisting of dynamically-recrystallized (DRX) grains and non-recrystallized grains elongated along the extrusion direction.

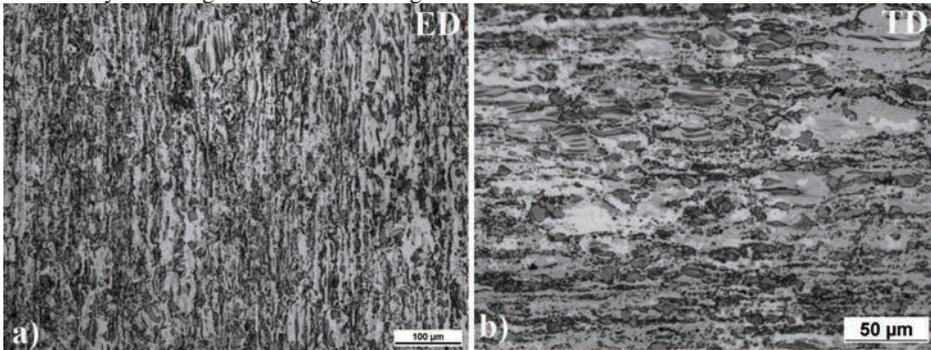


Figure 1: Typical microstructure of the initial state in a) extrusion direction (ED); b) transversal direction (TD). The loading direction is parallel with the vertical axis of the figure.

Figure 2 shows the recalculated (0002) and $(10\bar{1}0)$ pole figures of the magnesium phase measured in the section perpendicular to the extrusion direction. The alloy shows a strong fiber texture with the basal plane parallel to the extrusion direction. This texture component is given mainly by non-recrystallised grains, as it has been shown by several authors [1].

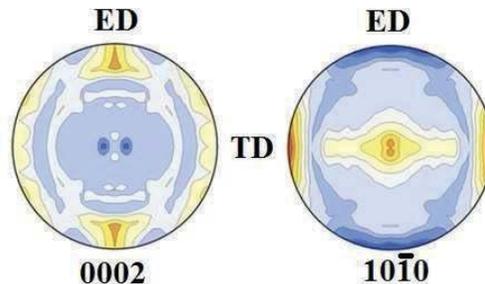


Figure 2: (0002) and $(10\bar{1}0)$ pole figures of the initial specimen showing a strong fiber texture

The deformation curves for the particular directions and the corresponding AE response are plotted in Fig. 3. It is obvious that the yield strength is higher for the ED specimen. Similarly the AE signal is more intensive in this direction. In order to reveal the background of this behavior, ASK analysis was applied on the continuously recorded AE data stream.

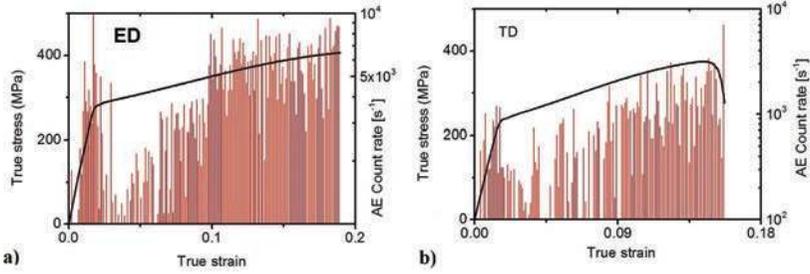


Figure 3: Deformation curves and corresponding AE response as measured for a) ED; b) TD specimens.

Raw AE signals recorded during compression tests were further analyzed using the adaptive sequential k-means (ASK) procedure. The main steps of the procedure can be summarized as follows:

- The recorded waveform streaming data are sectioned into consecutive frames. In our case a single frame corresponds to a 2 ms long “time window”. (see schematic drawing in Fig. 4)
- For each frame, the Fast Fourier Transformation (FFT) of the signal is done, which is used for the calculation of the Power spectral density (PSD) function.
- PSDs in the consecutive frames are analyzed one-by-one. The features of the PSD in the first frame define Cluster 1. All other clusters are defined with respect to this initial cluster. The conditions for new cluster forming are based on k -means method.

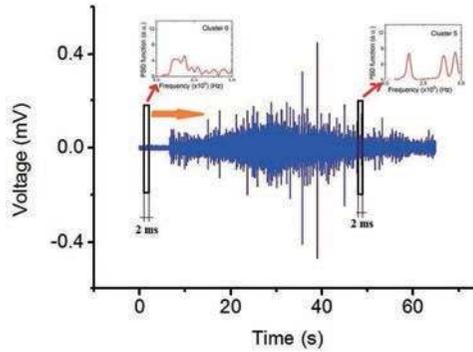


Figure 4: Schematic drawing of ASK procedure

When the clustering procedure is completed, a dominant AE source mechanism is assigned to each cluster. This assignment consists of two basic steps:

1. Checking the time of the appearance of the elements in a given cluster. The elements in Cluster 1 belong to the background noise, since the recording of AE data is always launched *before* the starting of the deformation test.
2. Checking characteristic features of the PSDs, as energy, frequency distribution etc.

Finally, the increment of the number of elements with time (Cumulative Number of Elements) can be plotted, which describes the activity of particular deformation mechanisms in time.

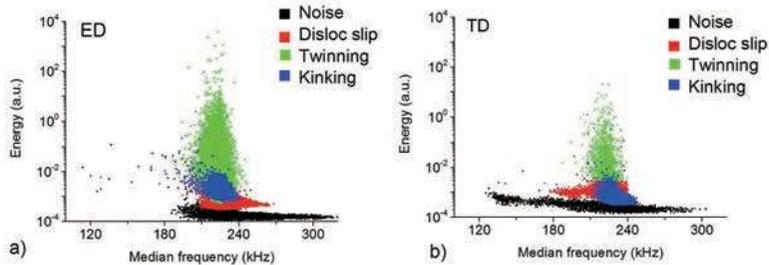


Figure 5: Energy – median frequency cross plot for a) ED; b) TD. The colors represent various AE source mechanisms

Based on the above mentioned algorithm, 4 main clusters have been identified. In order to facilitate the cluster assignment to the deformation mechanisms, we constructed the Energy vs. Median Frequency cross-plot (Fig. 5). It is obvious that for both specimens orientations the characteristic features of the clusters are similar. The evolution of the number of elements in the particular clusters is plotted in Fig. 6.

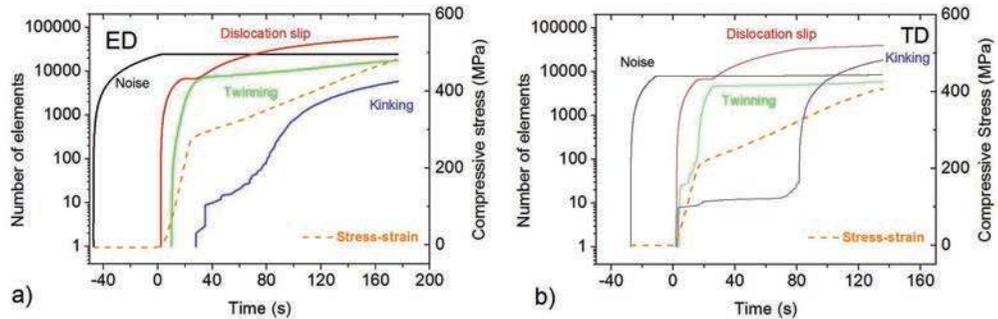


Figure 6: Time evolution of the number of elements in particular clusters, having different AE source mechanisms for a) ED; b) TD specimen.

Using information given by Figs. 5 and 6, the following mechanisms can be assigned to the clusters:

- Cluster 1 – Background noise (color code in figures – black)

Since the elements in this cluster appear before launching of the straining, this cluster naturally belongs to the background noise. They are typically low energy, broad frequency signals, as it was reported by several authors [7].

- Cluster 2 – Dislocation slip (color code in figures – red)

The number of events in this cluster starts to increase from the very beginning of the deformation. This behavior corresponds to the basal slip, which has the lowest critical resolved shear stress (CRSS) [8]. As the deformation progresses, further dislocation mechanisms (prismatic and pyramidal slip) contributes to this cluster. However, this analysis could not distinguish them properly. The dislocation cluster has a characteristic tear shape, which is given by shortening of the mean free path for dislocation movement at higher stresses, where the dislocation density is larger than that at the beginning.

- Cluster 3 – Twinning (color code in figures – green)

The elements in this cluster have high energy and a relatively narrow frequency range. Such a feature is characteristic for burst-type twinning signal. Also the appearance of this cluster in the early stage of the straining indicates the twinning origin of elements, since the CRSS of $\{10\bar{1}2\}$ -type extension twinning is as low as several MPa [8].

- Cluster 4 – Kinking (color code in figures – blue)

The average energy level of the signals is slightly higher compared to cluster 2 (dislocation slip). The cluster has a tear shape, but the frequency range is narrower than that for the dislocation slip. This cluster becomes dominant in the frequency spectrum from approximately 40 MPa. These features indicate the kinking-origin of this cluster. As it is mentioned in the introduction, the kinking is caused by avalanche-like motion of basal dislocations. Such a mechanism produces a relatively high energy signals [9], which is in good agreement with our experimental data. Owing to the structure of the LPSO phase, the initiation of kinking requires higher stresses [1].

If we compare the time evolution of the number of elements in the particular clusters (Fig. 6), we can see that noise and dislocation slip clusters behave similarly. The dominance of background noise terminates as the deformation starts. The dislocation slip has a large jump at the onset of the straining, as a consequence of the basal slip. There is a further jump on this curve in the vicinity of the macroscopic yield, which is caused by activation of non-basal $\langle a \rangle$ -slip in the prismatic and pyramidal planes [5]. The twinning is more significant in ED. This feature is evident also from Fig. 7, where the AE data are plotted together with diffraction data. It is obvious that there are more twins nucleated in ED (cf. AE data – green line) and their volume is significantly larger (cf. diffraction data – red scatter). This behavior can be elucidated with the initial texture, where in ED both large and small grains are favorably oriented for twinning. In contrast, only the non-recrystallized large grains can undergo twinning in TD. Consequently, both number and volume fraction of twins are larger in ED. There is also a difference in kinking activity. Since the stiff, elongated LPSO phase is aligned with the ED, load transfer takes place from the matrix to the LPSO during compression of ED specimens. This mechanism is less effective in TD case [10]. After reaching

a certain level of stress, kinking takes place. Again, owing to the orientation of the LPSO phase, kinking requires lower stress in ED, and it progresses gradually. In TD, kinking can be observed from approx. 80 MPa and its onset is more dramatic (Fig. 7).

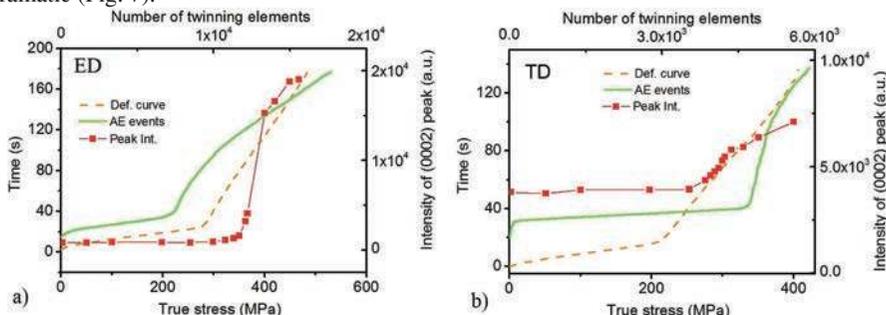


Figure 7: Change of the intensity of (0002) diffraction peak and evolution of number of twinning elements as a function of true stress for a) ED; b) TD specimen

4 CONCLUSIONS

The dependence of the deformation mechanisms with respect to the specimen orientation was investigated in Mg97Y2Zn1(at.%) alloy having LPSO phase. The following conclusions can be drawn:

- Higher yield stress and strength was observed for ED - which is given by more effective load transfer from matrix to LPSO phase.
- There is a significant difference in the twinning activity – owing to the initial texture more grains are involved in twinning process in ED. Consequently, both the twinned volume and the number of nucleated twins is larger for this specimen. In contrast, the dislocation slip seems to be similar for both directions.
- The onset of kinking is shifted to higher stresses in TD.

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