USE OF ACOUSTIC EMISSION FOR THE COMPLEX STUDY OF MAGNESIUM ALLOYS

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Abstract

Acoustic emission (AE) has been used to study magnesium alloys for many years. The advantage of AE is getting the data from the entire volume with high time resolution. The determination of the source of AE is an important task. We use a new method of evaluation of the AE signal: Adaptive Sequential K-means (ASK) analysis and its advantages for studying mechanical and corrosion properties of magnesium alloys. It uses the discrete Fourier transformation and statistical approach to discriminate between the different sources of AE. The results are presented on mechanical tests of rolled AZ31 sheet with high texture and corrosion behavior of pure magnesium and WE43 magnesium alloy.

Keywords: Magnesium, acoustic emission, mechanical testing, corrosion

1. INTRODUCTION

1.1. Acoustic emission

The acoustic emission (AE) has been found as a powerful non-destructive technique for study of deformation mechanisms. It gives information from entire volume about the dynamic processes during plastic deformation or corrosion processes. Although the sources of AE have generally different characteristics, it is still challenging task to make difference between them because of their concurrent activity[1, 2]. Moreover, the signal is modulated by the sample shape, microstructural characteristics or characteristics of the sensor. The classic approach of evaluation of AE data, so called hit-based processing, is based on setting parameters, which define the AE event (threshold level, hit definition time, …) [3]. The system saves the parameters of the AE event (amplitude, duration, counts, energy, …). In materials science, this approach can be successfully applied for general characterization, when the main goal is the investigation of the influence of the experimental and material parameters on the deformation behavior (e.g. [4, 5]).

There is a general agreement that the waveforms of particular AE processes are different [6], therefore, the spectrum-based analysis, when the characteristic features of frequency domain of the waveforms are investigated, can help to discriminate between the different sources of AE. In our work we applied the algorithm of Pomponi and Vinogradov [7, 8] - Adaptive Sequential K-means (ASK) analysis. Data are sectioned into consecutive individual realizations (“frames”) with length in order of μs and the power spectral density (PSD) function $G(\omega)$ is calculated for each frame computing the discrete Fourier transformation.

$$G(\omega)=\sum_{t=t_0}^{t_1} f_t\, e^{-2\pi i t \omega} \qquad (1)$$

where t_0 and t_1 are the time boundaries of selected frame and ω is a frequency, which is in range from 0 to $f/2$, where f is the sampling frequency. Discrimination between the signals coming from different sources is done on the statistical basis.

1.2. Magnesium alloys

Magnesium alloys represent the lowest density structural metals (along with toxic beryllium) and, thus, is a highly interesting material for modern applications. Moreover, it is possible to get outstanding mechanical properties with biocompatibility and increased corrosion resistance by alloying of magnesium [9, 10]. The investigation of mechanical properties of magnesium alloys is still a challenging task. The hexagonal close-packed structure causes complex deformation behavior of magnesium alloys, where the concurrent activity of several deformation mechanisms (basal slip, non-basal slip, twinning) is necessary. The activity of individual deformation mechanism is strongly dependent on the texture, alloying elements or temperature.

The investigation of corrosion behavior of magnesium alloys as an important task as well. The biocompatibility of magnesium and its corrosion properties predicts the use of magnesium alloys as biodegradable implants in medicine. There are three stages of pitting corrosion when exposed to environments containing chlorine ions: pit nucleation, development of metastable pits, which either repassivate or grow into stable pits further propagating into the material [11]. These processes are the source of AE and the signal analysis can provide deeper analysis of the corrosion processes.

2. MATERIAL AND EXPERIMENTAL METHODS

The deformation tests were performed on rolled AZ31 sheet. The coordinate system related to the sheet is the rolling direction (RD), normal direction (ND) - out of the sheet plane and transverse direction (TD). The sheet has a strong basal texture in ND (**Figure 1**). For deformation tests, samples of 6.5x6.5x10 mm^3 were cut out from the billets. The samples were cut in the RD, 45° and ND. Texture in RD is favorably oriented for twinning and prismatic slip, the 45° samples are favorably oriented for basal slip and the samples in ND are oriented favorably for the 2nd order pyramidal slip. The samples were deformed at the deformation rate 10^{-3} s^{-1}.

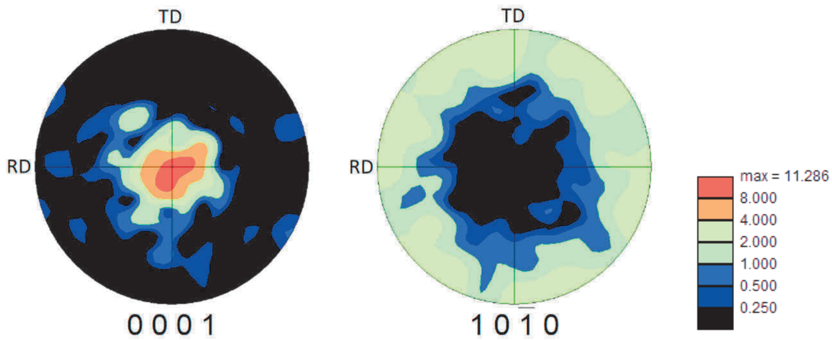

Figure 1 Pole figure of rolled AZ31 alloy

The corrosion tests were performed on two different magnesium alloys, pure magnesium and WE43 (Mg - 3.8 wt.% Y - 2.6 wt.% RE - 0.45 wt.% Zr - 0.01 wt.% Mn), both under as-cast condition. In order to minimize the influence of secondary phases, the cast billets were homogenized and solution treated at 525 °C for 16 h (T4 treatment) and quenched into water. The grain size after the treatment was ~110 and ~1000 µm for Mg and WE43, respectively. Samples with dimensions of 10 x 10 x 50 mm were cut from both materials and one side of each sample was wet ground using a 1200 grit SiC paper shortly before each test. A standard three electron setup was used for the electrochemical experiments. The samples were exposed to neutral 0.1 M NaCl solution at room temperature which was prepared using deionized water. The samples were kept at open circuit potential (OCP) at room temperature for 300 s. Anodic potentiodynamic polarization was subsequently carried out from 150 mV below OCP at a scan rate of 1 mV/s.

The AE was measured by the PCI-2 device from Physical Acoustics Corporation. Piezoelectric wideband PAC Micro30S AE sensor was used in order to record the broad AE signal spectra needed for the analyses. The signal was recorded with a sampling rate of 5 MHz for the deformation tests and 2 MHz for corrosion tests and the signal was preamplified by a PAC 2/4/6 preamplifier (60 dB gain).

3. RESULTS

3.1. Deformation tests

The deformation curves with raw AE response are shown in **Figure 2**. The yield stress of RD and 45 samples is the same, 88 MPa, while 150 MPa for ND sample. Also the shape of RD and 45 curves exhibits the S-shape which is typical for the $\{10\bar{1}2\}$ extension twinning, while the curve of the sample in ND has a convex shape. In contrast, the AE response is similar for ND and 45 directions and much stronger for the RD.

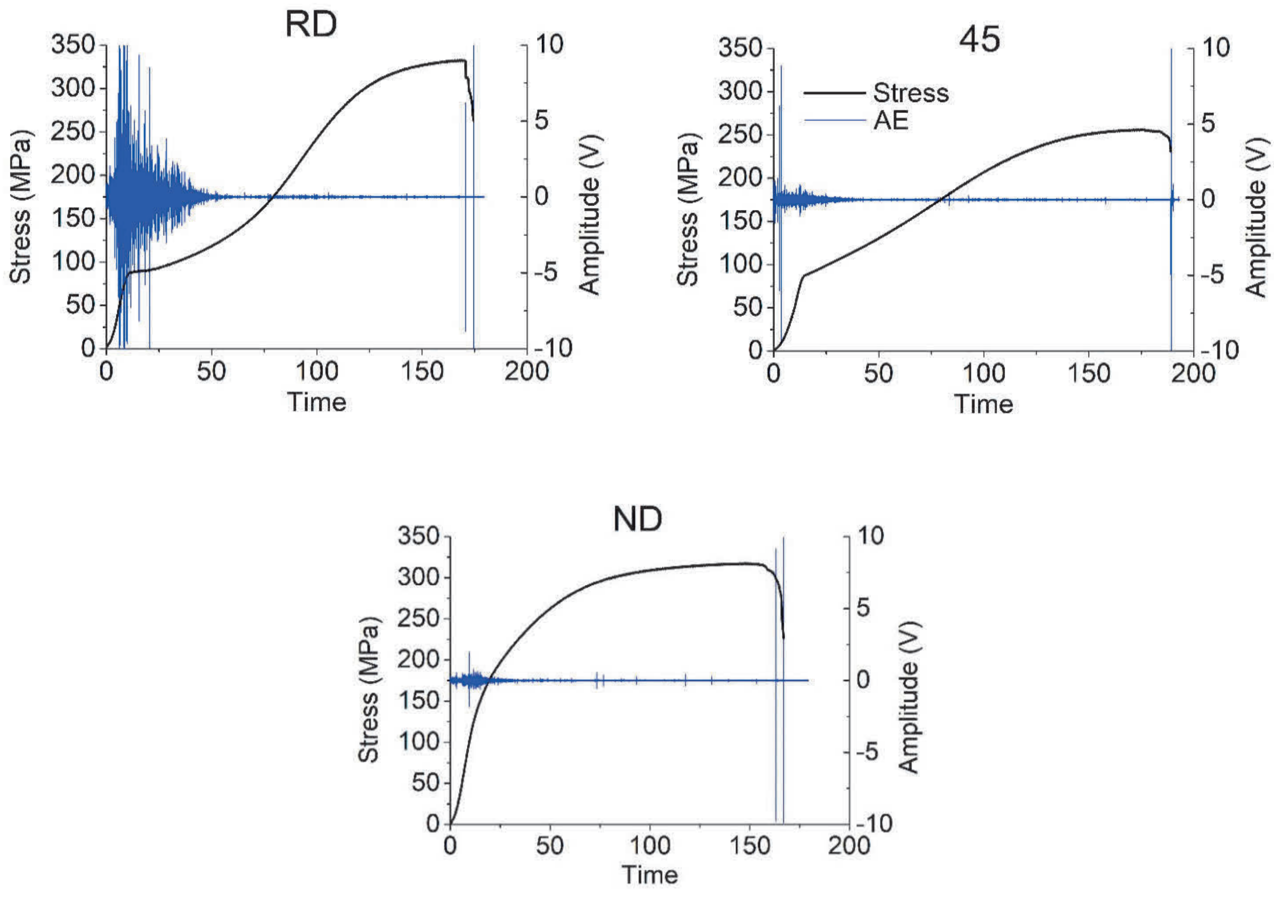

Figure 2 Deformation curves with the raw AE signal

The raw signal was evaluated by the ASK analysis. The signal was naturally separated into 4 different clusters which were assigned to noise, basal slip and twinning (**Figure 3**). For criterion of assigning the sources to clusters see [12]. All the samples exhibit the early appearance of basal slip which is consistent with the low CRSS of basal slip. The main difference between the samples is the activity of twinning. The cluster assigned to twinning is dominant around the yield point for all the orientation. The RD sample exhibits twinning up to 6% of deformation. This is consistent with the dominant orientation of the sample and the fact that twinning can accommodate up to 6.4% of deformation[13]. The twinning cluster is followed again by the basal one. This predicts that basal slip is a dominant deformation mechanism in twins. This does not mean that basal slip is not active during the previous deformation stage, only that the twinning is the stronger source of AE.

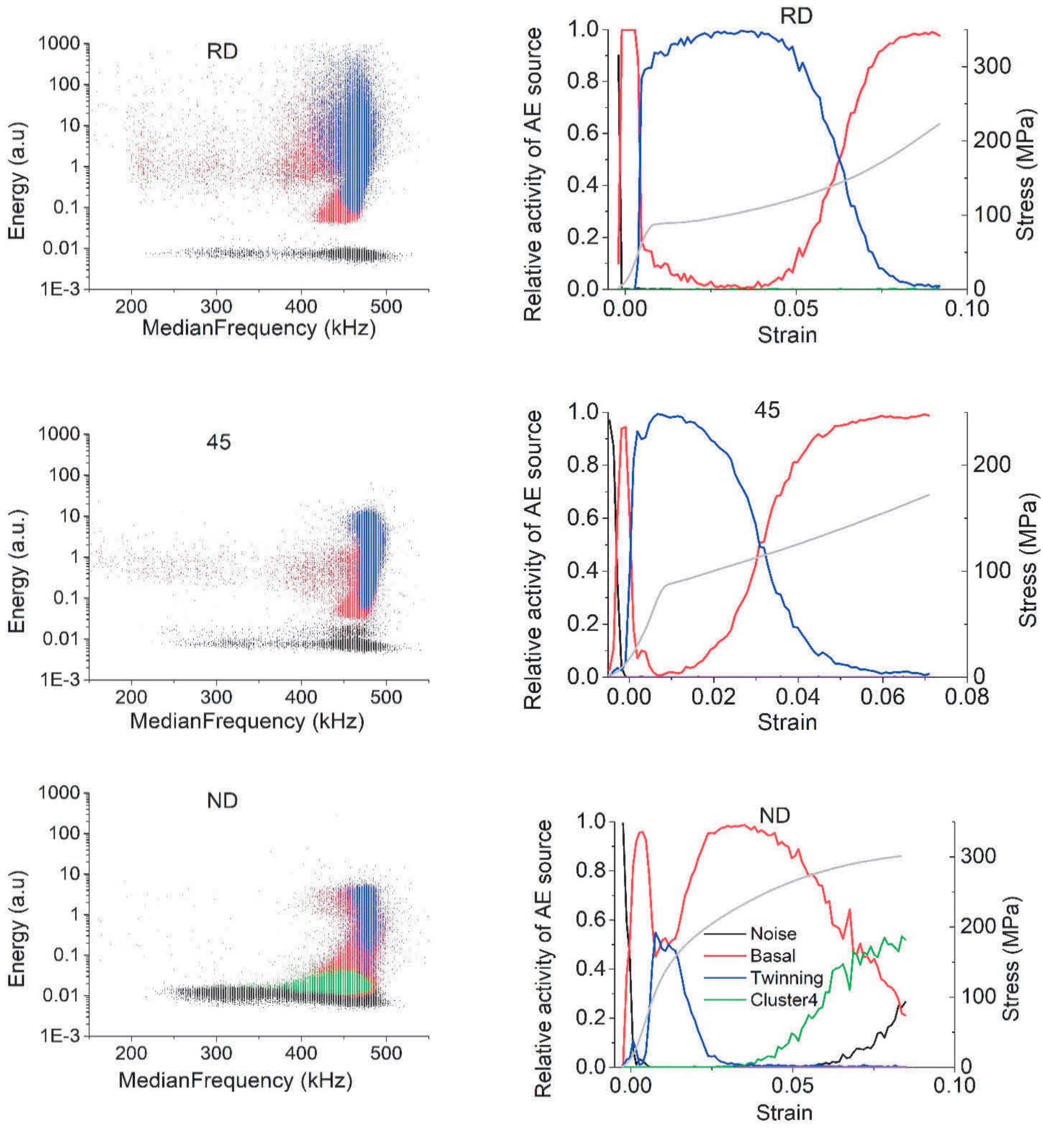

Figure 3 Relative activity of AE sources evaluated by ASK analysis and the projection of the clusters in median frequency vs. energy diagrams

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The sample 45 exhibits similar AE as RD sample. The twinning cluster is again dominant around the yield point, however, the basal slip starts to be dominant around 3% of deformation. The result is consistent with the fact, that the texture is more favorable for basal slip that for twinning and basal slip is the main deformation mechanism.

The appearance of the twinning cluster in ND sample is limited to the yield point and the twinning is not a dominant mechanism. The ND sample is oriented unfavorably for twinning, therefore, only few grains can undergo twinning[14]. Another difference between the ND sample and the others is the appearance of another source of AE marked as Cluster 4. The assignment of the deformation mechanism to this source of AE is more complicated and further investigation would be necessary. It can be either the 2^{nd} order pyramidal slip or compressive twinning.

3.2. Corrosion tests

The AE results recorded during potentiodynamic tests are presented at **Figure 4** together with the current density and its derivation. It is possible to observe, that at the moment when the current density starts to increase significantly also the AE response rise. Another important observation is that the AE response is significantly higher for WE43 than for pure Mg. However, these parameters do not say anything about the source of AE.

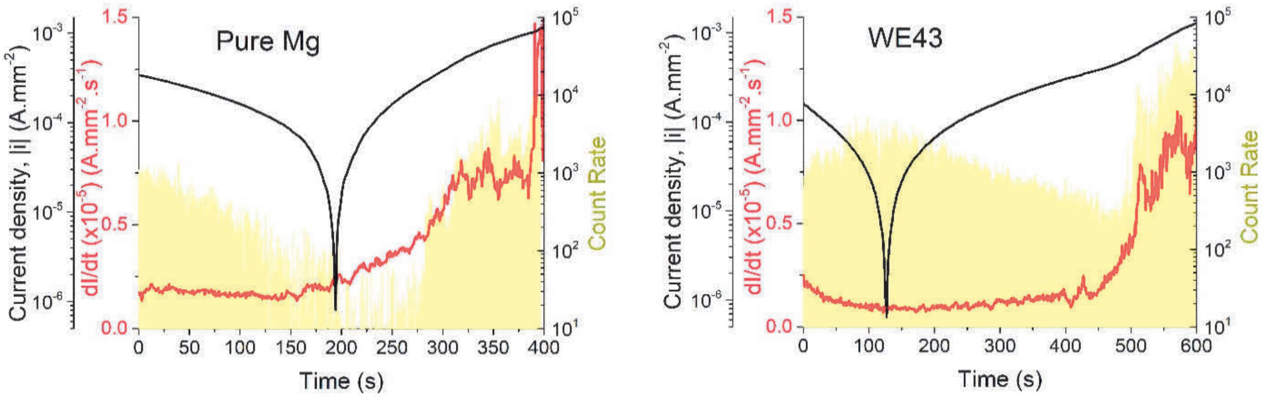

Figure 4 Acoustic emission count rate recorded during the anodic polarisation test on pure Mg and WE43 alloy displayed together with the absolute value of current density and its derivation

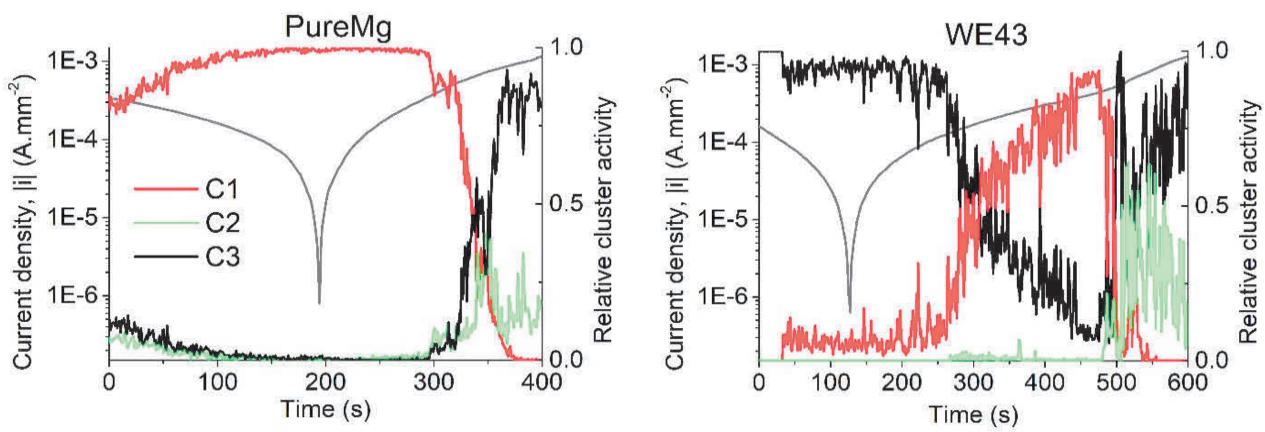

Figure 5 Time evolution of the relative activity of clusters for pure Mg and WE43 alloy

The ASK analysis done on the raw AE signal is shown at **Figure 5**. The signal was separated into three clusters (C1, C2, C3). Different evolution of a relative cluster activity can be observed. The corrosion process of WE43 alloy can be divided into 3 stages. Stage I is characterized by the high activity of cluster 3. This stage is present only for WE43 alloy. Cluster 1 dominates during the stage II and the concurrent activity of clusters 2 and 3 was measured during the stage III. The WE43 alloy is known to create the stronger protective layer [15]. The first stage of the corrosion process in WE43 will be the creation of the protective layer which is followed by its rupture. This is in agreement with our results. Cluster 3 (black) can be assigned to the ion exchange and cluster 1 (red) can be assigned to the rupture of the protective film. The cluster 2 (green) is active mainly at the end of the corrosion process. This suggests that the source is the stable pitting corrosion.

4. CONCLUSION

We showed that the advanced analysis of AE signal can provide additional information about the deformation and corrosion processes in magnesium alloys. The ASK analysis was used to determine the active deformation mechanisms in the textured AZ31 samples. The results show the decrease of the twinning activity with the rotation of the basal planes in the direction of loading, which is consistent with the decrease of the Schmid factor for twinning.

The ASK analysis of AE signal recorded during the corrosion of pure Mg and WE43 showed different evolution of corrosion mechanisms for both alloys. The results are consistent with the known facts about the samples. We can conclude that the ASK analysis proved to be powerful tool to investigate the properties of magnesium alloys.

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