

Influence of pre-compression on tensile behavior in wrought AZ31 studied by the acoustic emission technique

Daria Drozdenko¹⁾, Patrik Dobroň¹⁾, Jan Bohlen²⁾, Sangbong Yi²⁾ and František Chmelík¹⁾

1) Charles University in Prague, Ke Karlovu 5, Prague 2, 12116, Czech Republic.

2) Helmholtz-Zentrum Geesthacht, Max-Planck-Straße 1, Geesthacht, D21502, Germany

ABSTRACT: Deformation mechanisms in extruded AZ31 Mg alloy during one cycle loading (pre-compression followed by tension) are discussed in term of the acoustic emission (AE) response. Due to a fiber texture of the alloy, the level of pre-compression stress significantly influences the subsequent tensile behavior resulting in S-shape of deformation curve. The obtained AE results are correlated to the deformation curves and the differences in the AE count rate were used to reveal changes in underlying deformation mechanisms. Twinning during the pre-compression was followed by detwinning during the tensile loading.

1 INTRODUCTION

Wrought Mg alloys have been intensively studied due to their low density and high damping capacity. Therefore, they can fulfill requirements of many applications in the automotive and aerospace industry. However, their use is often limited due to inherent anisotropy of mechanical properties caused by their *hcp* lattice, texture and homogeneity of materials. Specific crystallographic textures are developed during formation processes, e.g. extrusion, rolling and forging. The strong basal texture of extruded Mg alloys, having basal planes almost parallel to the extrusion direction (ED), favor the occurrence of $\{10\text{-}12\}\langle\text{-}101\text{-}1\rangle$ twins during compression along ED [1]. Thus, a distinct tension-compression asymmetry at the yield strength (YS) during loading along the extrusion direction (ED) in Mg alloys is linked with the formation of those twins [2]. Thus, twinning is a key mechanism of plastic deformation in Mg alloys. Twins significantly influences the material behavior especially during cyclic loading, when twinning – detwinning mechanism induces important changes in the deformation behavior [2]. Tensile loading of the pre-compressed Mg alloy leads to disappearing of $\{10\text{-}12\}$ twins due to an easier activation of the detwinning process than the twinning one [2]. Extensive experimental research was focused on the role of twinning and detwinning also in fatigue behaviour [3,4]. It is, therefore, essential to get more insight into individual mechanisms of deformation (dislocation slip, twinning, twinning - detwinning) during a single cyclic loading with respect to given texture and homogeneity of material. Obtained results can contribute to analyze deformation behavior during repeated loading for several cycles and strain path changes, when twin-twin junctions can form, and they have been shown to play a crucial role in increasing strain hardening and controlling microstructure evolution.

The acoustic emission (AE) studies can be performed concurrently with the deformation tests, allowing the real time monitoring of active deformation mechanisms. The differences in AE signal characteristics can be used to distinguish different types of deformation processes. For example, in [5] a comprehensive set of AE data for basal slip and twinning was obtained during uniaxial compression of Mg single crystals along $\langle 11\text{-}22 \rangle$ and $\langle 11\text{-}20 \rangle$ axis respectively.

The main idea of the paper is to study an influence of pre-compression stress on subsequent tensile behavior of extruded AZ31 alloy during a single cyclic test. Active deformation mechanisms during loading are discussed in term of the AE response. Obtained results for Mg single crystals [5] are used for an interpretation of the AE signal in polycrystalline Mg alloy.

2 EXPERIMENTAL PROCEDURES

Wrought Mg alloy AZ31 ($Mg + 3 \text{ wt}\%Al + 1 \text{ wt}\%Zn + 0.3 \text{ wt}\%Mn$) was fabricated using indirect extrusion at 300°C with an extrusion rate (profile exit speed) of 5 m/min. The extrusion ratio was 1:30, which resulted in round bars with a diameter of 17 mm. The extruded profile exhibits a bimodal microstructure with an average grain size of $20 \pm 1 \mu\text{m}$. There are larger grains elongated in ED, along with a distinct fraction of grains with smaller sizes. The investigated alloy is characterized by a prismatic fiber texture with the highest intensity at the $\langle 10\text{-}10 \rangle$ pole. Thus, a distinct alignment of basal planes parallel to ED, that is, the c-axis is perpendicular to ED, is a characteristic feature of this texture.

Samples (gauge length of 15 mm, diameter of 8 mm) with screw heads on both ends were machined from the round extruded bar parallel to ED. Deformation tests (pre-compression followed by tension, i.e., one cycle tests) were carried out using the universal testing machines Zwick Z50 at room temperature (RT) and at an initial strain rate of 10^{-3} s^{-1} .

The AE activity during mechanical testing was monitored by a computer-controlled PCI-2 device, supplied by Physical Acoustic Corporation (PAC). AE was acquired using a miniaturized MST8S (Dakel-ZD Rpety, Czech Republic) piezoelectric transducer with a diameter of 3 mm and with a flat response in the frequency band from 100 to 600 kHz. The sensor was attached to the sample surface using silicon grease and a spring. A preamplifier



with a gain of 40 dB was used. The full scale of the A/D converter was ± 10 V (100 dB). To obtain a comprehensive set of AE parameters, a threshold level detection of 26 dB was applied.

3 RESULTS

To study an influence of the pre-loading level on subsequent tensile test, the samples were firstly pre-compressed up to a stress of 130 MPa, 150 MPa and 200 MPa, respectively, and then subjected to tensile loading up to fracture. For sample pre-compressed up to 200 MPa the resulting stress and the concurrently measured AE signal voltage are plotted against time (Figure 1). Data for whole test is presented in Figure 1a, whereas detail of the tensile part is presented in Figure 1b. As AE response for all three samples is similar the results for samples pre-compressed up to 130 MPa and 150 MPa are not presented here. It can be seen that yielding occurs at a compressive stress of 124 MPa. The plastic flow continues with an increasing (plotted negative) slope of the deformation curve. After the pre-compression and unloading, the tensile curves exhibit a characteristic sigmoidal shape (S-shape), which is typical for compression test of extruded Mg alloys. The deformation curve is correlated to the concurrent AE measurement trough measuring time. The AE signal is very strong during pre-compression, especially in the region of the compressive yield strength (CYS). In contrast, during tensile loading, smaller AE amplitudes were observed (Figure 1).

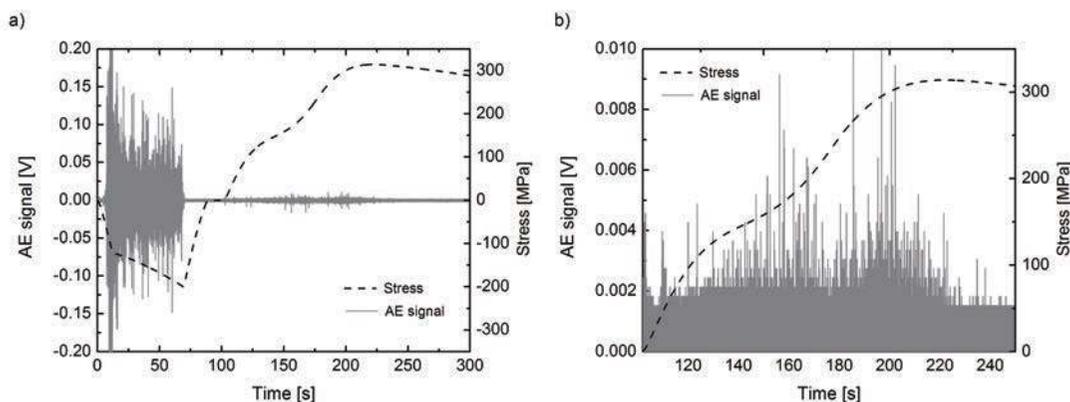


Figure 1 Stress and the AE signal voltage vs. time during one-cyclic loading (a) and its detail for tensile part (b).

Results of tensile parts of the tests after pre-compression up to various level of the stress are shown in Figure 2. It can be seen that the higher the compressive stress is the more pronounced is the S-shape of the tensile curve, see Figure 2a. The AE streaming data were parameterized and represented as the AE count rate – the count number per time unit [6] at a given threshold voltage level. Such parameterization offers important information about collective dynamic processes which occur during plastic deformation. In order to find the link between the particular deformation stages and the AE response, the true stress vs. true strain and AE count rates vs. time are correlated and presented in the same plot (Figure 2b-d for samples pre-compressed up to 130 MPa, 150 MPa, 200 MPa, respectively). During pre-compression, the AE count rates exhibit similar maximum at the CYS in all three cases (not presented here). During subsequent unloading from pre-compression level of stress to zero stress any detectable AE is not produced. In contrast, during the tensile loading, the AE count rate exhibits distinct changes with increasing strain (Figure 2b-d). Evolutions of the AE count rate are different with respect to different level of pre-loading. Changes in the AE activity could be related to the inflection points on the deformation curves. In all three cases of pre-compression, the maximum of the AE count rate occurs when stress-strain dependences are saturated. With applying higher pre-compression stress, an additional AE count rate peak, related to the plateau after yielding (S-shape), is observed. The AE count rate peaks become more pronounced with increasing level of pre-loading (Figure 2c-d). At the terminate stage of the deformation test a strong decrease in the AE count rate is observed in all three cases.

4 DISCUSSION

In Mg alloys plastic deformation starts in grains favorably oriented for $\langle a \rangle$ dislocation glide in the basal and prismatic plane [1]. Thus, collective movement of dislocations produces detectable AE signals even before achieving the macroscopic YS (Figure 1a). To retain the compatibility of plastic deformation during the compression test, with respect to very high CRSS for the activation of non-basal slip systems [1], the occurrence of twins is required. Due to strong basal texture of extruded AZ31 Mg alloy, where the basal planes are almost parallel to ED, the $\{10\text{-}12\}\langle\text{-}101\text{-}1\rangle$ twin system activates with applying the compression stress along ED. The plastic deformation proceeds by basal slip in grains reoriented due to twinning and, after reaching CRSS for

activation of non-basal slip systems, it proceeds also by $\langle c+a \rangle$ dislocation slip. Therefore, the twin nucleation was found as the main deformation mechanism at the macroscopic yield, and it significantly influences the YS. Thus, the observed low CYS and the strong AE signal are unequivocal signs of the activity of this mechanism. This is supported by the investigations conducted on Mg single crystals [5], where twinning was associated with burst AE signals with high amplitudes and basal slip was accompanied by a low amplitude AE signal. Moreover, in [7] it was concluded that the twin nucleation is an excellent source of AE, contrary to the twin growth, which does not contribute to the AE response. Other combined studies, provided by EBSD technique and light microscopy [8], AE with neutron diffraction [9], have shown that $\{10\bar{1}2\}\langle -101\bar{1} \rangle$ twins nucleate at the beginning of plastic deformation.

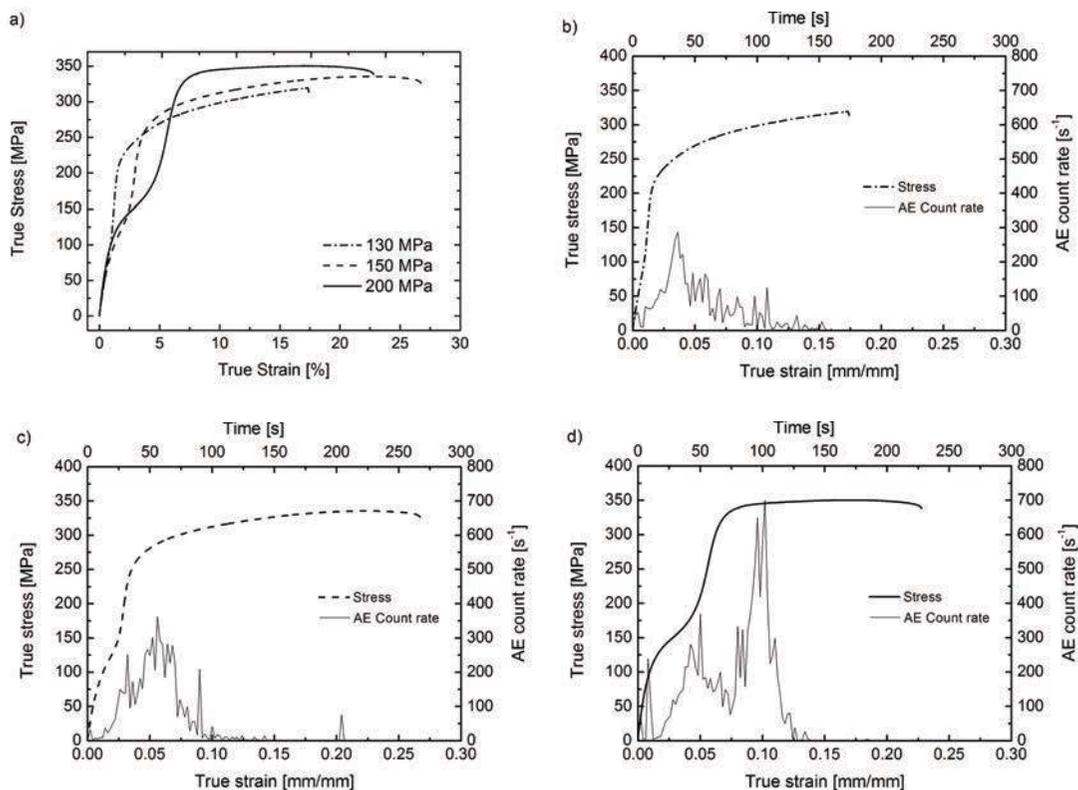


Figure 2 True stress vs. true strain (a) curves for the tensile loading of pre-compressed AZ31 Mg alloy. Deformation curves correlated with the acoustic emission count rate for the tensile loading of AZ31 Mg alloy after pre-compression up to 130 MPa (b), 150 MPa (c), 200 MPa (d).

The AE signal, observed shortly after the YS, has significantly lower amplitudes. This drop in the AE signal amplitudes indicates a change in the dominant deformation mechanism, and it is connected with the transition from the twin nucleation to dislocation slip and twin growth. Nevertheless, a few twins could still nucleate during increase in the compression load.

Unloading from the pre-loading compressive stress (cf. Figure 1a) to zero stress does not produce any detectable AE signals, which corresponds to the closing of dislocation sources. Furthermore, twin thinning may be an active relaxation mechanism during unloading. Similar to the growth or thickening of twins, detwinning or thinning is basically a movement of twin boundaries, and therefore no detectable AE signal [7] is expected as a result of this mechanism.

The subsequent tensile loading re-opens dislocation sources, and therefore, collective dislocation motion produces detectable AE signals. The AE response during entire tensile loading is significantly lower than during pre-compression (Figure 1). Basically, two opposite processes influence the AE activity during tensile loading. Namely, the increasing number of detwinned grains supports the AE activity through the rise in the flight distance and the free length of moving dislocations. On the other hand, the increasing dislocation density implies a stronger barrier for their movement, and therefore generally reduces the AE signal in the tensile part of the test.

According to Christian and Mahajan [10], a higher stress is required for nucleation than for the propagation of twins. Therefore, during reverse loading, detwinning is easily activated due to the already existing twin

boundaries. Based on this, the lower YS for the reverse tension than that for the pre-compression could be explained (Figure 1a for sample pre-compressed up to 200 MPa). Unlike the usual shape of the tensile curve, after pre-compression, the deformation curve for the tensile part is very similar to the compression part of the curve and it has S-shape. Twinning and detwinning, due to their strong polar nature, result in large reorientations of the crystal lattice (86.3°), which macroscopically gives rise to the characteristic S-shape stress-strain behavior, preceding the terminate strain-hardening region. A similar behavior was also observed during cyclic testing in the textured AZ31 sheet [2].

From analysis of tensile deformation curve and AE responses, it can be seen that detwinning process is more significant in case of high pre-compression (up to 200 MPa) and negligible for sample pre-compressed up to 130 MPa just achieving CYS. It is suggested that in last case, twin volume fraction is too small for massive detwinning process and it can not significantly influence shape of the deformation curve. Therefore, an additional AE count rate peak related to S-shape of deformation curve (Figure 2c-d) is associated with collective dislocation motion in grains after detwinning.

For all three cases the upcoming decrease in the AE count rates at terminate stage of deformation is connected with the decrease in the free path of moving dislocation due to an increase of dislocation density resulting in the strain hardening of the material. Similar effect can be seen for Mg single crystal compressed along $\langle 11-20 \rangle$ axis [5].

5 CONCLUSIONS

To study in detail possible deformation mechanisms, such as dislocation slip and twinning, twinning-detwinning, during cyclic loading rather than damage process a single cyclic test consisting of pre-compression followed by tension were performed. Especially the influence of pre-compression stress of subsequent tensile loading was analyzed. The transition between different deformation mechanisms, such as dislocation slip, twin nucleation, twin growth and detwinning, at different stages of the deformation test were discussed in term of the AE response. Detwinning has a similar influence on the deformation behavior to twinning: S-shape of the deformation curve. During twin growth and detwinning, the AE response shows events with lower amplitudes than during twin nucleation. Thus, the AE activity during tensile loading is a result of collective dislocation processes, while neither thickening nor thinning of twins obviously do not contribute to the AE response. For samples pre-compressed up to 150 MPa and 200 MPa an increase in the free length of moving dislocations during twin thinning (detwinning) leads to an increase in potential dislocation movement in detwinned grain fraction; therefore, an additional AE count rate peak is observed.

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REFERENCES

- [1] P.G. Partridge, (1967) "The crystallography and deformation modes of hexagonal close-packed metals", *Metallurgical Reviews*, Vol. 12, pp.169-194.
- [2] X.Y. Lou, M. Li, R.K. Boger, S.R. Agnew, R.H. Wagoner, (2007) "Hardening evolution of AZ31B Mg sheet", *International Journal of Plasticity*, Vol 23, pp. 44-86.
- [3] S. Begum, D.L. Chen, S. Xu, A.A. Luo, (2009) "Low cycle fatigue properties of an extruded AZ31 magnesium alloy", *International Journal of Fatigue*, Vol. 31, pp. 726-735.
- [4] J. Koike, N. Fujiyama, D. Ando, Y. Sutou, (2010) "Roles of deformation twinning and dislocation slip in the fatigue failure mechanism of AZ31 Mg alloys", *Scripta Materialia*, Vol. 63, pp. 747-750.
- [5] D. Drozdenko, J. Bohlen, F. Chmelík, P. Lukáč, P. Dobroň, (2016) "Acoustic emission study on the activity of slip and twin mechanisms during compression testing of magnesium single crystals", *Materials Science and Engineering: A*, Vol. 650, pp. 20-27.
- [6] Standard Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/vessels (2011).
- [7] C.R. Heiple, S.H. Carpenter., (1987) "Acoustic emission produced by deformation of metals and alloys - A review: Part II", *Journal of Acoustic Emission*, Vol. 6, pp. 215-237.
- [8] A. Ghaderi, M.R. Barnett., (2011) "Sensitivity of deformation twinning to grain size in titanium and magnesium", *Acta Materialia*, Vol. 59, pp. 7824-7839.
- [9] O. Muransky, M.R. Barnett, D.G. Carr, S.C. Vogel, E.C. Oliver., (2010) "Investigation of deformation twinning in a fine-grained and coarse-grained ZM20 Mg alloy: Combined in situ neutron diffraction and acoustic emission", *Acta Materialia*, Vol. 58, pp. 1503-1517.
- [10] J.W. Christian, S. Mahajan., (1995) "Deformation twinning", *Progress in Materials Science*, Vol. 39, pp. 1-157.