Analysis of deformation mechanisms in a textured AZ31 magnesium alloy

J Dittrich¹, J Čapek¹, M Knapek¹ and P Minárik¹

¹Charles University, Department of Physics of Materials, Prague, Czech Republic, EU
²Paul Scherrer Institute, Laboratory for Neutron Scattering and Imaging, Villigen, Switzerland

E-mail: dittrich.jan.cz@gmail.com

Abstract. A combination of advanced in-situ and ex-situ methods providing complementary information was employed in order to reveal the active deformation mechanisms during deformation of a heavily textured commercial magnesium alloy AZ31. Three sets of samples for both compression and tension testing were prepared from the rolled sheet with respect to its identified strong basal texture – normal direction (ND), rolling direction (RD) and 45° between RD and ND. During both compressive and tensile deformation of these samples, the signal of acoustic emission (AE) was concurrently measured. Afterwards, electron backscattered diffraction (EBSD) was used to study the microstructure after the selected stages of deformation. The EBSD analysis revealed that in the samples favourably oriented for extension twinning it played an important role during the plastic deformation, whereas its role in the unfavourably oriented samples was minor. This orientation and deformation mode dependency of extension twinning activation resulted in considerable differences in the deformation behaviour, which also influenced the AE response. A consistent link between the AE signal and the microstructural changes investigated by EBSD was established. Furthermore, to study the dynamics of plastic deformation and twin growth, high-speed camera footage of the sample surface was captured during compressive deformation. The obtained results were in complete accordance with the predicted behaviour, thus providing direct proof of the theory and extending the understanding of the deformation mechanisms.

1 Introduction

The deformation behaviour of magnesium (and its alloys) is rather complex due to its hexagonal close-packed structure with the c/a ratio, 1.623, close to the ideal value of 1.633. In the case of strongly textured materials, such as rolled sheets, the anisotropy of the hcp unit cell translates into anisotropy of the mechanical properties of the sheet, which negatively affects their formability [1-3].

There are two basic modes of deformation, playing critical roles in the deformation of magnesium at room temperature – dislocation slip and mechanical twinning. The slip system with the lowest critical resolved shear stress (i.e. the first one to be activated) is generally the (0001)(1120) basal slip. The next slip system to be activated is typically the {1010}(1120) prismatic slip [4]. The combination of these deformation mechanisms, however, provides only four independent slip systems, none of which provides elongation in the c direction, and thus fails to meet the Von Mises criterion [5]. To fulfil the criterion, either the {1122}(1123) 2nd order pyramidal slip or deformation twinning needs to be activated as well. The critical resolved shear stress for the 2nd
order pyramidal slip at the room temperature (which is the case of this study) is high, and therefore in the grains oriented favourably to do so, the \{10\overline{1}2\}\{10\overline{1}1\} extension twinning (i.e. providing elongation along the c-axis) is preferentially activated [4].

The acoustic emission (AE) phenomenon is defined as a transient elastic wave generated by a rapid release of energy within the deformed material [6]. The AE signal originates from active defects, such as formation and propagation of cracks, nucleation of twins and an avalanche-like movement of dislocations. The non-continuous (burst) AE signal can typically be ascribed to the nucleation of twins (and the initiation and propagation of cracks in the final stages of deformation). While nucleation of twins provides an excellent source of AE, their growth is too slow to be detected [6-8]. The continuous signal originates from the movement of dislocations. In order to be detectable, an extensive number of dislocations need to move rapidly over a sufficient distance.

For the purposes of this work, a so-called hit-based approach to the evaluation of AE events will be employed. In this approach, the AE event is defined by a set of criteria (e.g. threshold level, number of counts, duration). Once the events are separated from the raw signal, their record is systematically evaluated and described by multiple parameters (e.g. amplitude, count rate, energy rate). These parameters are subsequently examined to reveal the particular mechanism of the AE event origin. This approach has successfully been applied by other authors, e.g. [9,10].

The choice of magnesium-based sheet alloys is currently somewhat limited – the most common commercially available sheets are those of the AZ31 alloy. There has been extensive research conducted focusing on the anisotropy in the deformation behaviour of the rolled AZ31 alloy, such as [4] and more recently [11], dealing with the roles of non-basal slip mechanisms and mechanical twinning. The AE technique has also previously successfully been employed to investigate the mechanisms mediating the deformation of AZ31 alloy [12, 13]. The novelty of this study lies in the utilisation of the combination of the acoustic emission technique, providing sensitive yet indirect observations of the dynamical processes during the deformation, and high-speed camera imaging of the loaded sample surface, enabling direct observations of such processes. These observations are complemented by the EBSD-based microstructural analysis of the deformed samples.

2 Material and experimental methods
All experiments were conducted using a rolled sheet of the commercial AZ31 magnesium alloy (3% wt. Al, 0.8% wt. Zn, 0.2% wt. Mn). The sheet exhibited a strong basal texture in the normal direction (figure 1). Sets of samples with three different orientations with respect to the detected initial structure for both compressive and tensile tests – the selected orientations being normal direction (ND), rolling direction (RD) and 45° between RD and ND (denoted 45) – were prepared. All samples were of a rectangular cross-section, where the dimensions of samples for compressive tests were 10 mm x 6.5 mm x 6.5 mm, and the active area dimensions of the tensile samples were 6 mm x 3 mm x 2 mm.

![Figure 1. Inverse pole figures (IPF) of the ND, RD and 45 oriented samples.](image-url)
All deformation tests were performed at room temperature with the initial strain rate of $10^{-3}$ s$^{-1}$ using the Instron 5882 universal deformation machine. Concurrently with the deformations tests, the AE signal was measured. To record the broad AE spectra, the PAC PICO AE sensor was employed using the threshold level of 22 dB and 24 dB for the compressive and tensile tests. The signal was preamplified by the PAC 2/4/6 preamplifiers with a 40 dB gain in the frequency band 100 – 1200 kHz and recorded with the sampling rate of 5 MHz using the PAC PCI-2 device. The microstructure of samples deformed to selected strain (i.e. 1% plastic strain in compression and 1%, 5% and 10% plastic strain in tension) was investigated using electron backscattered diffraction (EBSD) with the EDAX EBSD camera installed in the ZEISS Auriga Compact scanning electron microscope. The high-speed camera footage of the sample surface during compressive deformation using the MTI SEMTester 1000 deformation table was captured via the PHOTRON FastCam SA-Z 2100K camera. Samples for both high-speed camera footage and EBSD were mechanically ground down to 0.25 µm diamond paste. In the case of EBSD samples, this was followed by ion-polishing using the Leica EM RES102 machine.

3 Results and discussion

The deformation curves from both compressive and tensile tests of all orientations of samples are shown in figure 2 (marked by dashed lines). For each curve, its yield point is marked (denoted $\sigma_{0.2}$). From the texture of the material (figure 1), it follows that the RD samples are favourably oriented for the $\{10\overline{1}0\} \langle 1\overline{1}2\overline{0} \rangle$ prismatic slip and for the $\{10\overline{1}2\} \langle 1\overline{1}2\overline{1} \rangle$ extension twinning in compression. The 45 samples are preferentially oriented for the (0001) $\langle 1\overline{1}2\overline{0} \rangle$ basal slip in both modes of deformation. The preferred slip system for the ND samples is the $\{11\overline{2}2\} \langle 10\overline{1}3 \rangle$ 2nd order pyramidal slip and namely the $\{10\overline{1}2\} \langle 10\overline{1}1 \rangle$ extension twinning in tension [14].

Figure 2. AE count rates and deformation curves for the ND, RD and 45 oriented samples deformed in compression and tension.

In accordance with these expectations, the deformation curves, namely of the RD, partly also of the 45 oriented samples in compression and ND (45) samples in tension exhibit sigmoidal S-shape evolution typical for deformation governed by the $\{10\overline{1}2\} \langle 10\overline{1}1 \rangle$ extension twinning [15]. The samples oriented unfavourably for twinning, i.e. ND in compression and RD in tension, exhibit a convex deformation curve indicating predominant slip character of deformation. The values of yield strength for both modes of deformation are significantly higher for the samples oriented unfavourably for twinning due to the necessity to activate the 2nd order pyramidal slip to achieve homogenous plastic deformation.
The AE count rate (i.e. the frequency of oscillations exceeding the threshold level) as a function of true strain is shown for all modes of deformation and orientations of samples in figure 2 and marked by (partially transparent) full lines. Higher AE count rate values indicate more pronounced and rapid activity of deformation mechanisms. The maxima of the AE count rate for all samples in both deformation modes were achieved near their respective yield points. The slightly more pronounced response from the samples oriented favourably for extension twinning can be explained by higher AE energy for twin nucleation than for dislocation slip [8].

However, while in compression the AE response diminishes rather abruptly after reaching its peak, the decline in tension is much more gradual. This phenomenon can be explained by a different manner of twinned volume increase – during compression, most of the \{10\overline{1}2\}\{10\overline{1}1\} extension twins nucleate around the yield point, and with further deformation, the already nucleated twins grow laterally (undetected by AE). On the other hand, during continued tensile deformation, the twinned volume increases via nucleation of new twins, which produces a detectable AE signal and thus prolongs its decline. This explanation of the so-called compression-tension asymmetry has been experimentally confirmed by combining the AE and neutron diffraction techniques [16].

The EBSD observations of the deformed microstructure, shown in figure 3, agree with the above-stated conclusions. The RD sample deformed in compression (figure 3b) exhibits a significant amount of \{10\overline{1}2\}\{10\overline{1}1\} extension twins, many of which penetrate grain boundaries and stretch across multiple grains due to their adjacent stress fields [17]. Muránsky et al. [18] argue that this phenomenon (so-called autocatalytic nucleation of twins) causes the surge in the AE activity near the macroscopic yield point (cf. figure 2). The twinning-wise unfavourable ND and partially favourable 45 samples (figures 3a and 3c) then exhibit only a small amount of heavily twinned grains. Their deformation is thus being governed by dislocation slip mechanisms.

**Figure 3.** IPF orientation maps of the microstructure of a) ND, b) RD and c) 45 oriented samples deformed in compression up to 1% plastic strain and the ND sample deformed in tension up to d) 1%, e) 5% and f) 10% of plastic strain. Deformation was applied along the out-of-plane axis in fig a) and in the horizontal axis in figs b) to f).
The microstructural evolution of the ND sample deformed in tension depicted in figure 3 confirms that with advancing plastic deformation, the twinned volume increases preferentially via nucleation of new tensile twins rather than growth of the already nucleated ones (cf. figures 3d and 3e). The ND tensile sample deformed up to 10% of plastic strain (figure 3f) then exhibits heavily deformed microstructure where all deformation mechanisms, including the \{10\bar{1}\}-\{10\bar{1}\} double twinning were activated (as was confirmed by reorientation analysis). At these later stages of deformation, the dislocation mean free path is reduced due to increasing dislocation density and newly formed twin boundaries, which causes the decrease of the AE activity (cf. figure 3).

Figure 4. High-speed camera images of a) microstructure of the RD sample compressed before the yield point, b) twinning band nucleated near the yield point, c) the twins forming this band marked by red colour, d) another twinning band nucleated ca. 1.5 MPa after the yield point and e) twins forming the second band marked by blue colour. Deformation was applied along the vertical axis.

The high-speed camera footage was captured during the compressive deformation of the samples. The footage confirmed that the deformation of the ND samples was governed by dislocation slip systems since no deformation twinning was observed. The 45 samples tended to retain more homogenous microstructure, while in the case of the ND samples, isolated bands of heavily deformed microstructure alternated with bands of significantly less deformed microstructure. In the RD sample, the \{10\bar{1}\}\{10\bar{1}\} extension twins nucleated mainly in the vicinity of the macroscopic yield point. The nucleation of twins was non-continuous and occurred in bands oriented perpendicularly to the loading direction, as is illustrated in figure 4. Barnett et al. [19] observed similar twinning bands in the extruded AZ31 alloy related to the Lüders phenomenon. The bands observed in this study are not likely to be caused by the Lüders behaviour since no plateau in the stress-strain curve following yielding was detected. However, in the said study [19], numerous twin population propagating from the sample edges perpendicularly to the loading direction with the increase of load was detected even in a coarse-grained sample, which exhibited no Lüders behaviour. The authors reason this behaviour by the preferential nucleation of extension twins on the sample edges due to higher stress values caused by friction and possible irregularities. Unlike the Lüders-like band propagation, however, in order for further twins to nucleate and thus accommodate the strain, an increase in stress is necessary, which means that no yield plateau is formed. It seems likely that analogous argumentation can be applied to explain the behaviour observed in this study.

The detected heterogeneous character of deformation may skew the results obtained by highly localised techniques, such as EBSD, especially if only one sample surface area is inspected. Therefore, such observations should either be performed using a significant statistical dataset or accompanied by methods taking information originating from more extensive sample areas into account.
4 Conclusions

The main results of this investigation, employing the combination of deformation tests with simultaneous AE recording, EBSD measurements and high-speed camera imaging to study the deformation behaviour of a rolled magnesium alloy AZ31, can be summarised as follows:

- The deformation behaviour followed the favourability for the activation of the \{10\overline{1}2\}\{10\overline{1}1\} extension twinning, arising from the different sample orientations with regards to the detected basal texture of the rolled sheet.
- A compression-tension asymmetry of the deformation behaviour was detected in the AE response and consistently linked to the EBSD microstructural observations.
- A heterogeneous character of deformation, displayed by the formation of deformation bands oriented perpendicularly to the loading direction, was observed during the compressive deformation by the high-speed camera imaging.

Acknowledgements

The authors are grateful for the financial support of the Czech Science Foundation under the contract 19-08937S. J. D. is grateful for the financial support of the Grant Agency of Charles University under contract 117120. P.M. acknowledges partial financial support by ERDF under project No. CZ.02.1.01/0.0/0.0/15_003/0000485.

References

[1] Kelly E W and Hosford W F 1968 Deformation characteristics of textured magnesium Tran. Metall. AIME 242 (4) p 654
[2] Avedesian M M and Baker H 1999 Magnesium and Magnesium Alloys (ASM Speciality Handbook) (ASM International)
[3] Yukutake A, Kaneko J and Makoto S 2013 Anisotropy and non-uniformity in plastic behavior of AZ31 magnesium alloy plates Materials Transactions 44 (4) pp 452-457
[4] Agnew S R and Duygulu O 2005 Plastic anisotropy and the role of non-basal slip in magnesium alloy AZ31B Int. J. Plast. 21 (6) pp 1161-1193
[5] Mises R von 1928 Mechanik der plastischen Formänderung von kristallinen Zamm – Z. Angew. Math. Me. 8 (3) pp 161-185
[6] Heiple C R, Carpenter S H and Carr M J 1981 Acoustic emission from dislocation motion in precipitation-strengthened alloys Met. Sci. 15 (11-12) pp 587-597
[7] Heiple C R and Carpenter S H 1987 Acoustic Emission Produced by Deformation of Metals and Alloys - A Review: Part I J. Acoust. Emiss. 6 (2) pp 177-204
[8] Heiple C R and Carpenter S H 1987 Acoustic Emission Produced by Deformation of Metals and Alloys - A Review: Part II J. Acoust. Emiss. 6 (4) pp 215-237
[9] Chmelik F, Klose F B, Dierke H, Sachl J, Neuhauser H and Lukáč P 2007 Investigating the Portevin-Le Chatelier effect in strain rate and stress rate controlled tests by the acoustic emission and laser extensometry techniques Mater. Sci. Eng. A 462 (1-2) pp 53-60
[10] Dobroň P, Bohlen J, Chmelik F, Lukáč P, Letzigr D and Kainer K U 2007 Acoustic emission during stress relaxation of pure magnesium and AZ magnesium alloys Mater. Sci. Eng. A 462 (1-2) pp 307-310
[11] Hou D, Liu T, Chen H, Dongfeng S, Chuhua R and Pan F 2016 Analysis of the microstructure and deformation mechanisms by compression along normal direction in a rolled AZ31 magnesium alloy Materials Science & Engineering: A 660 pp 102-107
[12] Bohlen J, Chmelik F, Dobroň P, Kaiser F, Letzigr D, Lukáč P and Kainer K U 2004 Orientation effects on acoustic emission during tensile deformation of hot rolled magnesium alloy AZ31 Journal of Alloys and Compounds 378 pp 207-213
[13] Meza-Garcia E, Dobroň P, Bohlen J, Letzigr D, Chmelik F, Lukáč P and Kainer K U 2007 Deformation mechanisms in an AZ31 magnesium alloy as investigated by the acoustic emission technique Materials Science & Engineering: A 462 pp 297-301
[14] Čapek J, Knapěk M, Minářik P, Dittrich J and Máthius K 2018 Characterisation of deformation mechanisms in Mg alloys by advanced acoustic emission methods Metals 8 (8) pp 644-655
[15] Prasad K E, Li B, Dixit N, Shaffer M, Mathaudhu S N and Ramesh K T 2014 The dynamic flow and failure behavior of magnesium and magnesium alloys JOM 66 (2) pp 291–304
[16] Čapek J, Máthius K, Clausen B, Strašká J, Berna P and Lukáč P 2014 Study of the loading mode dependence of the twinning in random textured cast magnesium by acoustic emission and neutron diffraction methods Materials Science & Engineering: A 602 pp 25-32
[17] Šiška F, Strail Ľ, Čiček J, Ghaderi A and Barnett M 2017 Numerical analysis of twin thickening process in magnesium alloys Acta. Mater. 124 pp 9–16
[18] Murášky O, Barnett M R, Carr D G, Vogel S C, Oliver E C 2010 Acta Materialia 58 pp 1503-1517
[19] Barnett M R, Nave M D, Ghaderi A Yield point elongation due to twinning in a magnesium alloy 2012 Acta Materialia 60 pp 1433-1443