In-situ neutron diffraction and acoustic emission investigation of twinning activity in magnesium

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Abstract. The neutron diffraction and acoustic emission (AE) techniques have been used for in-situ investigation of deformation twinning activity in cast polycrystalline magnesium at room temperature. The combination of these two techniques results in obtaining complementary information about the twinning mechanism during the straining. It is shown that loading mode significantly influences the twin nucleation and the twin size. In tension, the twin nucleation is observed during the entire test, while in compression only at the beginning of the plastic deformation. Nevertheless, the overall twinned volume does not differ. The optical micrographs support the above mentioned conclusions.

1. Introduction
The magnesium and its alloys are widely used in transportation industry due to their excellent strength to density ratio. However, the poor formability of magnesium alloys represents a substantial limitation for applications. Thus the developing of material with better strength-ductility ratio is of great importance.

The poor ductility of magnesium is most commonly rationalized in terms of dominancy of basal slip. In hexagonal materials the mechanisms of plastic deformation depend on the ratio c/a of crystallographic axes. In magnesium alloys, this value is close to the ideal, i.e. to $\sqrt{8/3}$. Therefore, in magnesium, the main slip system comprises the basal plane (0001) with the close packed directions of $\frac{1}{3}[1 \bar{1} 2 0]$ [1]. While these vectors are perpendicular to the c axis, the slip in the a direction cannot produce strain parallel to the c axis. According to von Mises criterion [2], the activity of five independent slip systems is required for the plastic deformation of polycrystals, but this condition in magnesium is not fulfilled [3]. The basal and prismatic slip planes together dispose with 4 independent slip systems. Thus either the second order pyramidal system or deformation twinning must be activated. At ambient temperatures, the critical resolved shear stress of the second order pyramidal system is much higher than that for twinning [4]. Consequently, in the common case, the twinning is the only active deformation mode that can provide straining along the c-axis at room temperature [5]. The $10 \bar{1} 2 \langle 1 0 \bar{1} 1 \rangle$ twinning mode has been found as the most significant in magnesium alloys [6].

The main advantage of the neutron diffraction technique consists in the deep penetration depth of thermal neutrons – the diffraction data usually characterize the entire volume of the specimen, especially its internal stress state [7, 8]. Recently, the in-situ neutron diffraction method has been
utilized also for characterization of deformation mechanisms in different materials as e.g. shape memory alloys [9, 10] or hexagonal materials [11]. It has been shown, that the integrated intensity of particular reflections is sensitive on the structural change (e.g. martensitic transformation) or reorientation of the crystal lattice (e.g. twinning, deformation texture) during the loading.

The acoustic emission (AE) technique yields information on the dynamic processes involved in plastic deformation of alloys [12]. The AE originates from transient elastic waves generated within a material owing to sudden localized irreversible structure changes. It has been shown [13] that deformation twinning and dislocation glide are the major sources of AE in hexagonal closed packed materials.

The advantage of both methods consist in giving information from entire volume in real time, contrary to the e.g. microscopy or electron backscattered diffraction methods, where only a small volume of the specimen is investigated in post-mortem state. Both, the neutron diffraction and the acoustic emission have been found as excellent method for study deformation processes in magnesium alloys [14, 15], since they provide complementary information: the AE is sensitive only to twin nucleation, whereas neutron diffraction characterize their growth as well [15].

The present work focuses on the study of twinning activity in cast polycrystalline magnesium with a random texture. The influence of the loading mode on twinning is discussed in detail. The influence of the twinning on AE signal and neutron diffraction pattern is also presented.

2. Experimental

Cast polycrystalline magnesium with 1 wt.% Zr content was used for the experiment. The specimens possessed a random texture and had an average grain size of 110 μm. Tensile testing was carried out using cylindrical specimens with a diameter of 5 mm and gauge length of 25 mm. Compression testing was performed using samples with a rectangular cross-section of 15 mm x 15 mm and height of 30 mm. Both tensile and compression testing took place at room temperature, at a strain rate of $2 \times 10^{-4}$ s$^{-1}$ and were finished at 4% strain. The strain was controlled by using an extensometer. A standard neutron powder diffraction instrument MEREDIT (Medium Resolution Diffractometer) [16] equipped with a loading frame was used for in-situ neutron diffraction measurements. The loading frame was installed in a vertical position, this arrangement provide information on evolution of radial component of the lattice strain tensor. Mechanical loading of the sample was interrupted for ~6 hours in order to collect the neutron diffraction pattern from the strained part of the specimen at a constant level of applied strain. Diffraction patterns were collected between 4 and 144° 2θ with step of 0.08° and time per step of 210 sec. FullProf Suite Tool [17] was used for the refinement of the measured diffraction patterns.

AE was monitored using a computer controlled PCI-2 device (Physical Acoustic Corporation). The facility incorporated a piezoelectric transducer with a flat response between 50 and 650 kHz and a preamplifier giving a gain of ~40 dB. The threshold level of detection was set as 35 dB.

The specimens for light microscopy were mechanically polished and finally etched (10 s) in a solution of 10% nitric acid.

3. Results

Tensile and compression true stress-true strain curves of specimens deformed to fracture, are shown in Fig. 1. The loading mode influences the shape of the deformation properties. The work hardening rate in compression is significantly higher at the beginning of the plastic deformation than that in tension. Both the yield stress and elongation exhibit an asymmetry. This, so called tension-compression asymmetry is well known [18] and it is connected with twinning phenomenon [18].

The deformation twinning in magnesium is accompanied by intensity variations between the $hkl$ diffraction peaks corresponding to the parent and twin grain families, as it was shown by Gharghouri et al. [19]. The direction of the intensity change depends on the loading mode and the applied experimental diffraction geometry, i.e. on the mutual position of the diffraction and applied load vectors. In the given experimental set-up we used the radial mode, where all diffraction vectors are perpendicular to the direction of the applied load. The diffraction patterns of the initial state and after
4% compression strain are shown in Fig. 2 measured on compression sample. The intensities of reflections \(\{10.0\}\) and \(\{00.2\}\) change evidently with increasing strain.

**Figure 1.** True stress-true strain curves deformed in tension and compression at room temperature.

**Figure 2.** Diffraction patterns measured in compression a) initial state; b) after 4% strain.

**Figure 3.** Schematic of the twinning mechanism with respect to the detector bank position a) in tension; b) in compression.

**Figure 4.** Evolution of the integrated intensity with strain for reflections (00.2) and (10.0) as a function of the loading mode.

In tension, the \(\{10.1\}\) twinning produces \(c\)-axis extension in grains, whose \(c\)-axis is parallel to the loading direction. Since this mechanism reorients basal planes by 86.3° (Figs. 3a and 4), an increase in the intensity of the \(\{00.2\}\) peak is expected. Consequently, the intensity of the parent \(\{10.0\}\) reflection decreases. On the other hand, during the compression test the prismatic \(\{10.0\}\) orientations have high Schmid factor for \(\{10.1\}\) twinning [20]. Therefore the intensities of the monitored peaks behave by the opposite way (Figs. 3b and 4). In order to compare the overall twinned volume during the tension and compression, respectively, we have normalized the measured integrated intensities. 0% is the integrated intensity of the particular peak measured in the initial state. Fig. 5 represents the absolute value (in percents) of the change of integrated intensity of the reflection \(\{00.2\}\) in comparison to the initial state as a function of the strain. It can be seen that the values are within the experimental error the same. The twinned volume in random textured cast magnesium alloys has been studied by several authors [21-24]. The numerical simulations indicate that in compression the twinned volume is larger (35% in compression vs. 10% in tension) [21, 24]. An opposite view was given by Xu et al. [23], who didn't find during their TEM investigation noticeable differences in the extent of twinning of the tensile and compressive AM50 samples. Our results support the latter opinion.
The comparison of acoustic emission response for tension and compression depicted in Fig. 6 revealed a different evolution of twinning. The signal recorded in compression (Fig. 6a) reached its maximum at approx. 1% strain in the low stress plateau range. The jump in the intensity of the \{00.2\} reflection indicate that this maximum is connected with activation of the tensile $\text{\textit{10\,\textit{T}_2}}$ twinning system. Similar behavior has been observed in compressed cast magnesium by Klimanek and Pötzsch [25]. Using light microscopy they found a maximum in the apparent mean volume fraction of the twins in the range of $0.05 - 0.1\%$. The rapid decrease of the AE signal after reaching of the maximum is caused by twin growth and change in the deformation mechanism. In compression, fast growth of $\text{\textit{10\,\textit{T}_2}}$ twins was observed [26]. The AE method is sensitive only to twin nucleation, not to twin growth [13], thus the AE signal decreased. The continuing of deformation is followed by activation of non-basal slip systems [27, 28]. This mechanism increases the dislocation density and consequently reduces the mean free path of moving dislocations. Since the latter parameter is directly proportional to the AE signal strength [12], the AE signal decreased under the detectable limit.

![Figure 5. Change of integrated intensity of the reflection \{00.2\} in comparison to the initial state for both loading modes.](image)

![Figure 6. Dependence of acoustic emission signal on loading mode (a) – compression, (b) – tension. The true stress-true strain curves are depicted in blue. The evolution of corresponding integrated intensities is represented by scatter charts.](image)
During the whole tensile test the burst AE signals were observed. After the reaching the maximum, the signal level decreases gradually (Fig. 6b). This behavior indicates that twin nucleation took place during the entire test. In tension, the nucleation of twins requires higher stresses and their growth is limited [29]. The variation of the integrated intensity corresponds to the AE findings. In the elastic region the intensity of the parent and twin plains is negligible. When the yield point is reached, a strong increase could be observed. The microstructures observed after both tension and compression tests support the above mentioned consideration. After the compression test large thick twins could be observed (Fig 7a). On the other hand, the tensile specimen exhibits a lot small twins (Fig 7b).

4. Conclusions
In the current study the deformation twinning in weak textured cast magnesium during both tensile and compression test was investigated. 10\bar{T}2 tensile twinning was observed in both loading modes.

The neutron diffraction measurements revealed that the overall twinned volume during the tensile and compression deformation does not differ within the experimental error. Nevertheless, differences in the twinning mechanism and twin size were found by means of acoustic emission measurement. In compression, the twin nucleation was observed entirely at the beginning of the plastic deformation. The subsequent straining caused twin growth. On the other hand in tension, significant twin nucleation was observed during the entire test. The light optical microscopy examination showed that the twin size in tension is significantly smaller.

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