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Investigating a Twinning–Detwinning Process in Wrought Mg Alloys by the Acoustic Emission Technique

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Abstract

Two extruded magnesium alloys, AZ31 with a bimodal microstructure and ZE10 with a homogeneous microstructure, were pre-compressed and subsequently subjected to tensile loading. The acoustic emission (AE) technique was concurrently applied to determine the activity of particular deformation mechanisms. Significant changes in the AE response were correlated with the inflection points on the tensile curve. Twinning and detwinning processes were analyzed using the AE dataset. Particularly, nucleation of various types of twins and the influence of twins produced by pre-straining on the subsequent hardening behavior were investigated in detail. The output of the AE analysis was related to microstructure observations provided by the electron backscattered diffraction (EBSD) method.

Introduction

Specific crystallographic textures develop during formation processes, such as extrusion, rolling, and forging. The extruded Mg alloys usually exhibit a strong basal texture, where the c-axes of the hcp unit cells in the majority of grains are perpendicular to the extrusion direction. Consequently, a distinct tension–compression asymmetry linked with the formation of twins appears at the yield point during loading along the extrusion direction [1, 2], which significantly influences the material behavior during cyclic loading.

The most common twinning system in Mg alloys is the {10-12} <10-11> extension type, which results in a tensile strain parallel to the c-axis and a compression strain perpendicular to it [3-6]. The only other deformation mechanism that has the same impact is the <c+a> slip. However, its activation requires a considerably higher stress [7] than that for twinning.

Additionally, various “compression” twinning modes, either the {10-11} or {10-13} planes, could be activated when a compression stress parallel to the c-axis is applied [8, 9].

Due to the 86.3° lattice reorientation caused by extension twins, detwinning could be activated in the twinned volumes during subsequent reverse loading [10, 11]. This process is characterized by a thickness reduction or disappearance of existing twin lamellae; that is, the twin lamellae are rotated back to the parent matrix orientation [12]. Hence, if the extension twinning develops during in-plane compression along the extrusion direction (ED) in an extruded Mg bar, detwinning occurs in the twinned volumes at subsequent tensile loading along ED. It can be deduced that twinning is a key deformation mechanism in Mg alloys during cyclic loading, when the occurrence of the twinning–detwinning process significantly influences the deformation behavior. Extensive experimental research has also focused on the role of twinning and detwinning in fatigue behavior [13-15].

During reverse loading, occurrence of detwinning is often reported, for example, in [10-12, 16-26]. Despite intensive research, there are many open questions in this field: the influence of strain path changes on the twinning–detwinning process, the coexistence of detwinning and the formation of new twins, etc.

Acoustic emission (AE) studies can be performed concurrently with the deformation tests, allowing real-time monitoring of active deformation mechanisms. The AE technique is based on the detection of transient elastic waves, which are generated within the material due to sudden localized structural changes. The differences in AE signal characteristics can be used to distinguish different types of deformation processes [27]. In the present work, AE measurements and electron backscattered diffraction (EBSD) are used to investigate strain path dependences of the deformation mechanisms during a single cyclic test consisting of pre-compression followed by reverse tensile loading. The AZ31 and ZE10 specimens, which have similar texture but different grain size distribution, were used. The transition between different deformation mechanisms, such as dislocation slip, twin nucleation, twin growth and detwinning, at different stages of the deformation test were revealed using a combination of AE and EBSD methods.

2. Experimental procedures

Magnesium alloys AZ31 (Mg + 3 wt%Al + 1 wt%Zn + 0.3 wt%Mn) and ZE10 (Mg + 1.3 wt.% Zn + 0.1 wt.% Ce) were fabricated using indirect extrusion at 300°C with an extrusion rate of 5 m/min and 10 m/min, respectively. The extrusion ratio was 1:30, which resulted in round bars with a diameter of 17 mm.

Samples with a gauge length of 15 mm, a diameter of 8 mm, and screw heads on both ends were machined from the round extruded bar parallel to the ED. Pre-compression followed by tension—that is, one-cycle tests—were carried out using the Zwick Z050 and Instron 5882 universal testing machines at room temperature (RT) and an initial strain rate of 10^{-3} s^{-1}.

The EBSD technique was used to reveal the microstructure and texture of the extruded profiles in the as-received condition and after loading to a certain stress. Orientation patterns were collected using field emission gun scanning electron microscopes (Zeiss Ultra 55 and FEI Quanta 200 equipped with EDAX/TSL EBSD systems). The mapping was carried out with a step size of 0.4 µm and an acceleration voltage of 15 kV. The data acquisition software program TSL-OIM Analysis 7.0 was used to process the EBSD data. The sample surface was grinded, polished and subsequently electropolished in an AC-2 solution (Struers) at -26°C, 33 V for 90 s. To improve the quality of the surface prior to measurements, the samples were rinsed in 0.5% nital (nitric acid in methanol) and ethanol and dried with pressurized air.

A computer-controlled PCI-2 device, supplied by Physical Acoustic Corporation (PAC), was used to monitor the AE activity during mechanical testing. The AE signal was acquired using a miniaturized MST8S (Dakel-ZD Rpety, Czech Republic) piezoelectric transducer (Ø 3 mm) with a flat response in the 100–600 kHz frequency band. The sensor was attached to the sample surface using a clamp. The good acoustic contact was ensured by using vacuum grease. A preamplifier with a gain of 40 dB was used. The AE parameters were obtained by hit-based processing, in which the threshold level was set to 26 dB. The AE streaming data were parameterized and represented as the AE count rate, which is the count number per time unit [28] at a given threshold voltage level.

3. Results

3.1. Initial microstructure and texture

The microstructure and texture of AZ31 and ZE10 in the as-received condition, determined from EBSD measurements, are shown in Fig. 1. The extruded profile of AZ31 exhibits a bimodal microstructure with an average grain size of 20 ± 1 µm. There are larger grains elongated in ED, along with a distinct fraction of grains with smaller sizes. In contrast, the

ZE10 magnesium alloy has a fully recrystallized microstructure with an average grain size of 26 ± 1 μm and a more homogeneous grain structure (Fig. 1). For both extruded profiles, a prismatic fiber texture was observed with the highest intensity at the <10-10> pole in the case of AZ31 and a distribution of intensities along the arc between the <10-10>- and <11-20>-poles in the case of ZE10. A distinct alignment of basal planes parallel to the ED—that is, the c-axis is perpendicular to the ED—is a characteristic feature of these textures.

3.2. AE response: effect of pre-compression on subsequent tensile deformation

To investigate the microstructure development during the test, a condition with partially twinned grains was preferred. In [16], it was shown that the microstructure of AZ31 after pre-compression up to 150 MPa is characterized by existing of extension twins, giving a partly twinned microstructure. This is the reason why samples of both alloys, AZ31 and ZE10, were pre-compressed to 150 MPa and then subjected to tensile loading. To find the link between the particular deformation stages and the AE response, the stress, concurrently measured AE signal voltage (Figs. 2a and 3a) and AE count rates (Figs. 2c–d and 3c–d) are plotted against time. It can be seen that yielding occurs at a compressive stress of (124 ± 1) MPa for AZ31 and (81 ± 1) MPa for ZE10. The plastic flow continues with an increasing (negative) slope of the deformation curve. After the pre-compression and unloading, the tensile curves have a characteristic S-shaped form for both alloys.

The AE signal (Figs. 2a and 3a) is very intensive during pre-compression, especially in the vicinity of the compressive yield point. Large amplitude AE events are characteristic in this region. In contrast, during tensile loading, smaller AE amplitudes were recorded. It is noteworthy that the AE signal amplitudes are significantly higher during the entire test for ZE10 than for AZ31. This is particularly significant during the tensile part; see the details in Figs. 2b and 3b.

The AE count rate *vs*. the time curve for the compression part of the test (Figs. 2c and 3c) exhibited a maximum at the compressive yield point. The subsequent unloading from 150 MPa to zero stress does not produce any detectable AE.

During the tensile loading of AZ31, the AE count rate exhibits distinct changes, revealing three maxima with increasing strain (Fig. 2b). In the case of ZE10, two significant maxima in the AE count rate are observed (Fig. 3b). Both alloys exhibit a strong decrease in the AE count rate at the terminal stage of the deformation test. Changes in the AE activity could be related to the inflection points on the deformation curve for both Mg alloys.

3.3. Microstructure evolution during compression–tension cycle

3.3.1 Characterization of AZ31 with bimodal microstructure

Samples for EBSD analysis were pre-compressed up to 150 MPa and subsequently loaded in tension up to specific stress levels; these stresses were chosen according to the changes in the shape of the deformation curve and the AE activity (Fig. 2d). The microstructures of samples after only pre-compression and after pre-compression and tension up to 95 MPa, 150 MPa, 230 MPa, and 275 MPa are denoted in Fig. 2d as A, B, C, D and E, respectively, and are presented in Fig. 4.

The analysis of the misorientation angle distribution of the EBSD map of the sample after pre-compression (Fig. 4a) confirms the presence of {10-12}<10-11> extension twins with a misorientation angle of 86.3° around the <11-20> axis. During reverse tensile loading, the extension twins become thinner. The microstructure development can be tracked in Fig. 4b–d taken from samples B through D. Sample D (Fig. 4d) shows a twin-free microstructure; therefore, reverse tensile loading up to 230 MPa seems to be sufficient to complete the detwinning process. A further increase in tensile stress leads to the nucleation of compression twins with a misorientation angle of 56° around the <11-20> axis, which can be seen in the example for sample E.

The evolution of the crystallographic texture during the test evaluated from EBSD measurements is illustrated in Fig. 5. After pre-compression along ED, the main intensity of the basal planes is observed in ED. During the subsequent tension along ED, its intensity decreases, as is obvious in Fig. 5c–d. At the terminal stage of tensile loading, a strong, prismatic fiber texture similar to that of the initial state of the material is observed (Fig. 5a and 5f).

Fig. 6 shows the microstructure of the pre-compressed AZ31. The grains without twin boundaries are marked in green, and the boundaries of the {10-12}<10-11> extension twins are marked in red. It can be observed that smaller grains are mostly free of twins, whereas larger grains contain twins. The textures of these two fractions of the microstructure are plotted in Fig. 6b for small grains without twins and Fig. 6c for larger grains containing twins. The latter fraction exhibits a texture with two distinct components—one around the <0001> pole, related to the orientation of twins, and the other clearly concentrated around the prismatic poles, related to the orientation of parent grains. In the case of the smaller grains, the texture is comparable with the initial texture, perhaps with a slightly more distinct component in the <10-10> pole. Nevertheless, both fractions of grains have orientations similar to the basal planes parallel to ED; hence, a relatively larger effect of grain size on the activity of twins is expected.

The inspection of twinned grains reveals

- the activation of different variants of extension twins.
- that in some grains, there are two groups of twins with a misorientation angle of 6° between them. As an example, a detailed view of the orientation map of the microstructure of sample B is presented in Fig. 7a.
- that pre-existing twins can provoke twin nucleation in the neighboring grains, and, consequently, twins become connected at the grain boundaries.

- that no compression twins are found at the early stages of tensile loading. Only after the shrinkage of extension twins do new twins (Fig. 4e), particularly compression twins, begin to nucleate and grow. A better overview of compression twins is given in Fig. 7b–c.

The same test concept was applied to ZE10. Fig. 8 shows the area fractions of twins for both alloys. After pre-compression, the twin area fraction for ZE10 is larger than that for AZ31, and during reverse loading, it is reduced similarly in both alloys.

3.3.2 Characterization of ZE10 with homogeneous microstructure

The orientation map of the microstructure of ZE10 after pre-compression (sample I in Fig. 3d) is depicted in Fig. 9a. The microstructure contains many {10-12}<10-11> extension twins giving a high twin area fraction (47%) of the analyzed area. A small number of grains are completely twinned as shown in Fig. 10, and contrary to AZ31, no grains are twin-free.

The orientation maps for the samples that were subsequently loaded in tension up to 95 MPa, 130 MPa, 180 MPa, and 275 MPa (denoted as samples II, III, IV, and V, respectively) are presented in Fig. 9b–e. The twins become thinner than those in sample I during tensile loading up to 95 MPa, cf. Fig. 9a–b. The detwinning process continues with increasing tensile stress (Figs. 8 and 9b–d). A small number of extension twins are still observed in sample IV. After increasing the tensile stress to 275 MPa (sample V), the detwinning process seems to be complete, and new twins are nucleated. Similar to AZ31, the nucleation of compression twins takes place only after the complete detwinning of extension twins. The orientation map and corresponding illustration of parent grains and compression twins are shown in Fig. 11b–c. For sample V, some twins are of the {10-12}<10-11> extension type. Different colors indicate that different twin variants operate from those observed during pre-compression (Fig. 11a and b).

The evolution of the crystallographic texture of ZE10 during pre-compression and the subsequent tension is shown in Fig. 12. During pre-compression along ED, basal planes change orientation to ED due to extension twinning. Thus, a strong texture component in ED appears (Fig. 12b). During subsequent tensile loading, the intensity of this main peak decreases, as shown in Fig. 12c–e. At the terminal stage of tensile loading, the texture has a prismatic fiber character and is similar to that of the initial state of the material (Fig. 12a and f).

As mentioned above, during pre-compression, extension twins reorient basal planes from the initial orientation, parallel to ED, into an orientation perpendicular to ED. At the terminal stage of tensile deformation (275 MPa, sample V), the newly nucleated extension twins reorient the original lattice (marked in yellow in Fig. 11c) by 86.3° around ED; that is, basal planes of the twinned fraction (marked in red) remain oriented mainly parallel to ED. A schematic illustration of twin variants in specific grains is presented in Fig. 11b.

It should be noted that the orientations of the c-axes of some grains are close to ED (such as grain 1 in Fig. 11b–c). This is inconsistent with the original texture of ZE10 before pre-compression; in the original texture, the c-axis orientations of the grains are perpendicular to ED. Therefore, these grains are assumed to be already fully reoriented as a result of twinning during pre-compression.

4. Discussion

General findings during pre-compression and reverse tensile loading

AE signals appear even before reaching YP (Figs. 2a and 3a), suggesting the beginning of dislocation slip before yielding. This was also observed in previous works [29, 30]. The twin nucleation was found as the main deformation mechanism at the macroscopic yield, and it significantly influences the yield stress. Thus, the observed low compression yield strength (CYS) and strong AE signal are unequivocal signs of the activity of this mechanism.

This is supported by the investigations conducted in [27, 31, 32], which all demonstrated 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 the EBSD technique and light microscopy [33], AE with digital image correlation [34], and AE with neutron diffraction [35], have also shown that {10-12}<10-11> twins nucleate at the beginning of plastic deformation.

Based on the misorientation analysis of the EBSD maps (Figs. 4a, 9a), the activation of {10-12}<10-11> extension twins is revealed in alloys after pre-compression. The extension twin activity can also be observed in the texture development, during which a strong peak appears in the ED of the (0001) pole figure; this can be seen for pre-compressed samples A and I for AZ31 and ZE10, respectively, and is not visible in the as-received condition (Figs. 5 and 12).

The AE signal, observed shortly after the YP, has significantly lower amplitudes. This decrease 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 an increase in the compression load.

Unloading from the compressive stress of 150 MPa (Figs. 3–4) 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, so no detectable AE signal [31, 36, 37] is expected as a result of this mechanism.

According to Christian and Mahajan [38], a higher stress is required for nucleation than for the propagation of twins. Therefore, during reverse loading, detwinning is easily activated owing 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 (Figs. 2a, 3a).

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 has a sigmoidal shape, including an inflection point. Twinning and detwinning, owing to their strong polar nature, result in large reorientations of the crystal lattice (86.3°), which macroscopically gives rise to the characteristic "S-type" stress–strain behavior, preceding the strain-hardening region. A similar behavior was observed during cyclic testing in the textured AZ31 sheet [11].

The subsequent tensile loading reopens dislocation sources, so collective dislocation motion produces detectable AE signals. Consistently, with increasing tensile load, the AE count rates also increase until reaching a certain level of stress (Figs. 2d and 3d). The upcoming decrease in the AE count rates is connected with the decrease in the free path of the moving dislocation owing to an increase in dislocation density resulting in the strain hardening of the material [39, 40].

For both alloys, the AE response during the entire tensile loading is significantly lower than during pre-compression. 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. However, the increasing dislocation density implies a stronger barrier for their movement and therefore generally reduces the AE signal voltage and the AE count rate in the tensile part of the test.

Microstructure and texture effects on the deformation behavior

Despite similar general findings for both alloys, certain distinct differences in the deformation behavior of the alloys are observed and are detailed in the following paragraphs.

The significantly higher AE signal in the case of ZE10 compared with that of AZ31 (Figs. 2a–b and 3a-b) can be explained by the differences in the microstructures. ZE10, with its homogeneous microstructure and an average grain size of 26 μm, has a lower number of grain boundaries as potential obstacles for the dislocation movement, whereas AZ31 has a bimodal microstructure and an average grain size of 20 µm. Similar results were found by previous studies [41, 42], in which it was reported that a larger grain size results in a stronger AE response.

In the case of pre-compressed AZ31, it was observed that smaller grains do not twin (cf. Fig. 6). It is mentioned above that there is no distinct difference between the textures of the twin-free grains and the original orientations of twinned grains. In the case of pre-compressed ZE10, all grains contain twins, which is likely a result of a homogeneous microstructure and grains with larger average grain size than in AZ31. In fact, non-twinned grains, which were observed in AZ31, are essentially not found because of the absence of the fraction of small grains in the microstructure of ZE10. In addition, some grains show numerous twins in both alloys, and different extension twin variants are clearly visible. The larger twinned area fraction and higher AE activities around the compressive yield points in ZE10 indicate higher twinning activity than in ZE10. This is also consistent with lower CYS in ZE10 compared with AZ31. In conclusion, there is a grain size dependence of twin nucleation, indicating that a higher number of twins will nucleate for larger grain sizes. This result is specifically related to the grain size; no texture effect is revealed (Fig. 6).

The difference in the texture, in the context of the difference in grain orientation, could result in differences in the activation of twin variants during plastic deformation. Despite the similar initial texture of AZ31 and ZE10, the EBSD analysis of both alloys shows that different variants of twins are activated independently of the initial orientation of the grains. In previous works [30, 43, 44] Schmid factor calculations have been used to study the probability of the activation of specific extension twin variants, with respect to the loading axis.

Out of six possible twin variants [30], two activated from different “sides” of the hcp cell from two groups of twins in large grains. This is especially visible in the elongated grains of AZ31;

see the illustration in Fig. 7a. A misorientation angle of 6° between two groups of twins is explained by the characteristic angle of 86.3° for extension twins. Such groups are observed in grains with different orientations with the c-axis oriented along <10-10> and along <11-20>; hence, no texture effect is revealed for either alloy.

Reverse loading after pre-compression

The detwinning process proceeds similarly in both alloys and simultaneously in grains with different orientations; thus, no grain size or texture dependencies were revealed. The detwinning progress is observed in both EBSD orientation maps and texture developments. In the case of ZE10, compared with AZ31, a higher level of applied reverse stress is required for detwinning, owing to the large twinned area after pre-compression. In summary, twinning and detwinning processes, in terms of boundary mobility, are not size dependent. In contrast, twin nucleation is size dependent.

The AE count rate peak at the last stage of plastic deformation is related to active dislocation motion and the further nucleation of extension and compression twins. Thin twins are observed in grains with different orientations (Figs. 4e and 9e) when the detwinning process seems to be completed. In both alloys, compression and extension twins are observed. Extension twinning in ZE10 occurs in grains with their initial orientation and in grains that were completely twinned during pre-compression. Fully twinned grains become favorably oriented for the nucleation of extension twins during reverse loading, as seen in Fig. 11b. A detailed analysis of the EBSD maps shows that during reverse loading in grains with the initial orientation extension, twinning rotates the c-axes around ED, whereas during pre-compression, rotation of the c-axes occurs toward ED; see the illustration in Fig. 11a. This could be explained by the preference in the activation of another twin variant [30] with respect to the loading direction, which contributes to strain accommodation of the material.

The activities of compression and extension twins, which occur only after detwinning—that is, at a later stage of deformation—support the idea that the stress required for nucleation of new twins is higher than the stress required for moving the twin boundaries, [3, 45]. The last AE count rate peak is located specifically in the range of the test with the highest strain hardening rate—that is, at the inflection point of the stress–strain curve. This AE count rate peak is connected to the new twin nucleation. In general, compression twins of {10-11} and {30-32} types have been observed [9] at large tensile strains in Mg and its alloys.

5. Conclusions

The strong basal texture of the extruded ZE10 and AZ31 Mg alloys favors the activation of extension {10-12}<10-11> twins during compression in ED. In the case of AZ31, small grains contain a smaller number or no twins, whereas multiple twins nucleate in larger grains. This is also supported by AE measurements, which show that compared with AZ31, the AE signal in the case of ZE10 has higher amplitudes of events. The differences in the homogeneity and the grain size distribution indicate that twin nucleation is grain size dependent.

During reverse loading along the extrusion direction, detwinning is a significant deformation mechanism and is preferred at the expense of nucleation and growth of new twins. Detwinning has a similar influence on the deformation behavior to twinning (S-shape of the deformation curve). It was also found that twinning and detwinning, in terms of boundary mobility, are not grain size dependent.

During twin growth and detwinning, the AE response shows events with lower amplitudes than during twin nucleation. Therefore, neither thickening nor thinning of twins obviously contribute to the AE response. In this part of the test, dislocation slip dominates in the AE response. An increase in the free length of moving dislocations during twin thinning leads to an increase in potential dislocation movement; therefore, an increase in the AE response during reverse loading is observed.

Based on the grain size dependence of twin nucleation and the dislocation motion, compared with AZ31, a stronger AE signal for ZE10 is observed during the entire test. The higher dislocation mobility observed here can be explained by the higher mean free path of dislocation motion due to a lower concentration of obstacles such as grain boundaries.

In both AZ31 and ZE10 alloys, new compression and extension twins appear after completing the detwinning process at a later stage of plastic deformation. This is because higher stresses are required for their nucleation compared with the detwinning mechanism. Newly created twins contribute to strain accommodation in relation to the orientation of the parent grains. Moreover, during tensile loading, another type of extension twins nucleate compared with those that are active during pre-compression. The nucleation of twins at this stage of deformation results in an additional AE count rate peak.

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Fig. 1 Orientation maps on the longitudinal sections and inverse pole figures in ED of AZ31 (a) and ZE10 (b) and their grain size distribution (c) and (d), respectively.

Fig. 2 AE response during loading of AZ31: stress, AE signal voltage *vs.* time (a) and its detail (b); AE count rate *vs.* time curves during pre-compression (c) and tension (d).

Fig. 3 AE response during loading of ZE10: stress, AE signal voltage *vs.* time (a) and its detail (b); AE count rate *vs.* time curves during pre-compression (c) and tension (d). Note that the scale of the AE signal is 10 times higher in Fig. 3b than that in Fig. 2b to reveal the shape of the AE response clearly.

Fig. 4 Orientation map of AZ31 (ED is vertical) after pre-compression up to 150 MPa (a) and subsequent tension up to 95 MPa (b), 150 MPa (c), 230 MPa (d), and 275 MPa (e).

Fig. 5 Crystallographic texture of AZ31 round bar (ED is perpendicular to pole figures) in the initial state (a) and at various deformation stages: after compression up to 150 MPa (b) and tension up to 95 MPa (c), 150 MPa (d), 230 MPa (e), and 275 MPa (f).

Fig. 6 Image quality map (a) of AZ31 (ED is vertical) after pre-compression, texture of (b) grains without twins (green) and (c) grains with {10-12}<10-11> extension twins (boundaries are marked in red) and (d) their grain size distributions.

Fig. 7 AZ31: (a) detailed orientation map for sample B and illustration of the activated extension twin variants; (b) image quality map and pole figure for sample E shows the presence of compression twins (blue). The parent orientation is marked as “M.” = “Matrix”; extension twin variants are denoted as “tw1(2)” (misorientation angle of 86.3°).

Fig. 8 Dependences of the twin area fractions on applied reverse loading stress (tensile part).

Fig. 9 Orientation map of ZE10 (ED is perpendicular to the map) after pre-compression up to 150 MPa (a) followed by tension up to 95 MPa (b), 130 MPa (c), 180 MPa (d), and 275 MPa (e) (samples: I–V in Fig. 3d, respectively).

Fig. 10 Image quality map (a) of ZE10 (ED is perpendicular to the map) after pre-compression up to 150 MPa; texture (b) and grain size distribution (c).

Fig. 11 ZE10: orientation map and illustration of activated twin variants for samples I (a) and V (b). The parent orientation is marked as "M." = matrix; extension twins are denoted as "tw." (misorientation angle of 86.3°), and compression twins are denoted as "c.tw." (misorientation angle of 56°). (c) Image quality map and pole figure for the sample V (ZE10). Extension twins are marked in red; compression twins are marked in blue.

Fig. 12 Crystallographic texture of ZE10 (ED is perpendicular to the pole figures) in the initial state (a) and at various deformation stages: after compression up to 150 MPa (b) and tension up to 95 MPa (c), 130 MPa (d), 180 MPa (e), and 275 MPa (f).