

**An Acoustic Emission Study of Deformation Mechanisms in Novel
Magnesium Alloys during Uniaxial and Low Cyclic Loading**

Contents

Introduction	3
1. Theory	4
1.1. Plastic deformation of metals with hexagonal crystal lattice	4
1.2. Extrusion	6
1.3. Acoustic emission	8
1.3.1. Definition and method of acoustic emission	8
1.3.2. The sources of acoustic emission	8
1.3.3. Terminology and basic parameters of AE	10
2. Experiments	11
2.1. Investigated materials	11
2.2. Experimental methods	11
2.2.1. Deformation tests	11
2.2.2. The acoustic emission measurements	12
2.2.3. Light microscopy	13
3. Results	14
4. Discussion	24
5. Conclusions	27
Options for further research	28
References	29
List of figure captions	31
List of abbreviations and symbols	33

Introduction

The development of industry increases the demands on the mechanical properties of materials. In certain sectors, such as aerospace or automotive industry, the aim is to replace the conventional materials by light alloys without the deterioration of mechanical properties. This is due to the effort to reduce fuel consumption and emission, which are influenced by vehicle weight (reduction of vehicle weight by 10% will reduce the fuel consumption about 5%).

Magnesium alloys belong to the lightest construction metals. Their density is lower than in industry usually used steels or aluminum alloys. One of the first applications (from 1936) of Mg alloys was the crankshafts in the famous Volkswagen – Beetle cars. In the late 70s and early 80s with the expansion of water-cooled engines was significantly reduced the use of Mg alloys due to their low corrosion resistance. In the 90s the use of different Mg alloys was started again, due to the strict limits of emission and fuel consumption. Nowadays, Mg alloys are used in the automotive industry (e.g. Volkswagen and Audi), to produce sport equipment, in electrical industry, in transport and in production of household items. Mg alloys have high specific strength and stiffness. Another advantage of Mg is its easy availability (from 1 liter of seawater we can obtain 1.4 g Mg [1]) and good recyclability. Comparing to polymers, it has better mechanical properties, greater resistance to aging and better electrical and thermal conductivity. On the other side, Mg alloys have poor cold formability, particularly low corrosion resistance and greater thermal expansion than Al alloys.

The term acoustic emission (AE) is closely related to the plastic deformation and fracture processes in materials. This phenomenon is connected with the acoustic noise, which is generated inside the material during plastic deformation. AE is experienced by people in everyday life, e.g. earthquakes, landslides, avalanches, crackling wood.

The first studies in this area are from the 50s of the last century and are associated with the name of a German physicist Joseph Kaiser, who examined metallic and non-metallic materials [2]. In the 60s was started the development of AE methods (nondestructive testing, in-situ measuring) in technical applications, what was demonstrated by the development of new devices for measuring the AE [3].

1. Theory

1.1. Plastic deformation of metals with hexagonal crystal lattice

Magnesium has hexagonal close packed crystal lattice (hcp), which strongly affects its mechanical properties. A limited number of active slip systems in the hcp metals, deformed at room temperature, significantly affect their deformation behavior.

The dislocations, which are one of the line defects of materials, have the highest impact on deformation behavior. According to the direction in which dislocations produce shear stress, they are divided into two types: edge (Fig. 1.1.a)) and screw (Fig. 1.1.b)) ones. Border of inserted half-plane is called as edge dislocation.

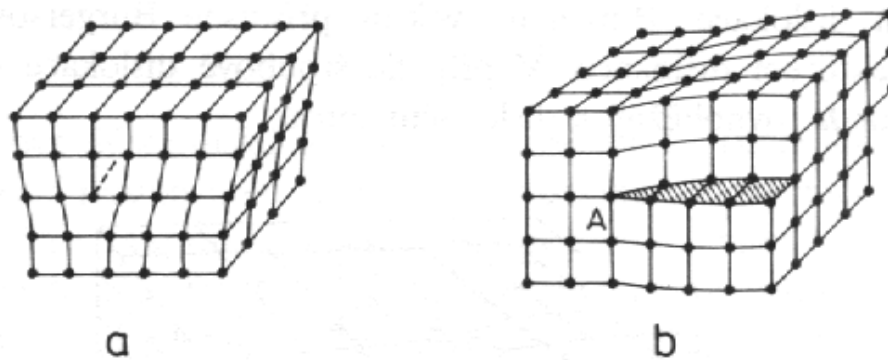


Fig. 1.1. Edge (a) and screw (b) dislocation [4]

During plastic deformation, dislocations are not only formed, but they can move and interact with each other (annihilation, accumulation).

In the hcp metals, it can be distinguished three main slip systems, which are presented in Fig. 1.2. Plane with the highest density of atoms in Mg alloys is the basal plane (0001) with the slip directions $\langle 11\bar{2}0 \rangle$. Other planes are the prismatic $\{10\bar{1}0\}$ and pyramidal (first $\{10\bar{1}1\}$ and second $\{11\bar{2}2\}$ type)[3].

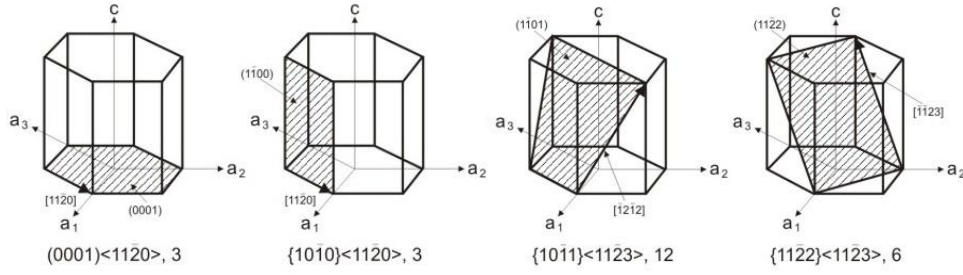


Fig. 1.2. Basic slip planes in hexagonal materials [5]

Microscopic changes in metals with hexagonal structure strongly depend on the ratio of the crystalline axis c to a (shown in Fig. 1.3.). The ideal ratio c/a , i.e. the closest configuration is equal to $\sqrt{\frac{8}{3}}$. If the ratio is greater than the ideal, slip takes place primary in the basal plane in the $\langle 11\bar{2}0 \rangle$ directions. In the case of Mg, where this ratio is close to the ideal value, the prismatic or the pyramidal planes can be activated under certain conditions. The biggest impact on their activation has the deformation temperature or the chemical composition of the material.

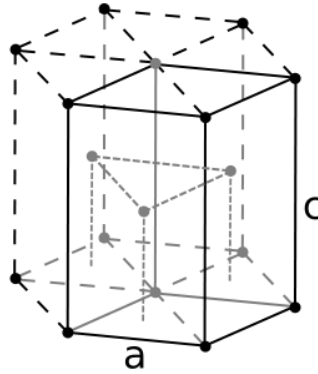


Fig. 1.3. Hexagonal lattice

According to von Mises criterion [6], the compatible plastic deformation in polycrystalline material requires at least 5 independent slips systems. In Mg and its alloys are available only two crystallographically independent directions. The main slip system is the basal plane with the lowest critical resolved shear stress (CRSS) of all slip systems. The CRSS of the pyramidal slip is approximately two orders of magnitude higher. Basal slip systems do not allow deformation in the direction of the c axis and for the activation of pyramidal slip system is required very high CRSS, what leads to the need to activate an additional deformation mechanism at room temperature (RT), which is called twinning.

Twinning is the process when a part of the crystal is shifted to a new position, which is the mirror image of the original, while the mirror plane is the twinning plane (Fig. 1.4.). Twinning is caused by large local stress, e.g. from accumulated dislocations at grain boundaries. Nucleation of twins is homogeneous and depends on the microstructure [7]. This phenomenon contributes to increasing the dislocation density, which leads to the strain hardening of the material. The main twinning systems in Mg alloys are the $\{10\bar{1}2\}$ $\langle 10\bar{1}1 \rangle$ and $\{10\bar{1}1\}$ $\langle 10\bar{1}\bar{2} \rangle$ systems. The first type is tension twinning (produces elongation in the c-axis), the second type is the compression twinning (produces shortening in the c-axis).

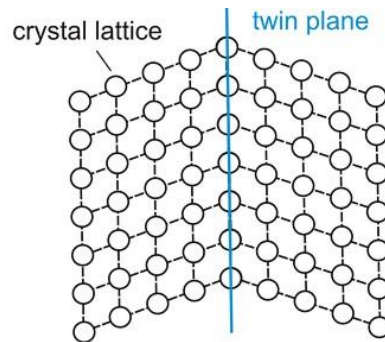


Fig. 1.4. Twin plane [8]

The formation of twins depends not only on the applied stress but also on the temperature and on the grain size. With increasing temperature the CRSS for the activation of non-basal slip systems decreases and therefore above a certain temperature twinning is not required anymore.

1.2. Extrusion

Extrusion is a process, which is used to manufacture semi-finished and also final products. The principle of the extrusion is shown in Fig. 1.5. On the left side is shown a forward (direct) extrusion, in which the material flows in the direction of the extruder while to achieve uniform pressure on the walls of extruded material a hydrostatic medium is applied. In Fig. 1.5. on the right side is shown the backward (indirect) extrusion, in which the material flows against the working direction of the extruder without the use of hydrostatic medium.

The final grain size depends on the parameters of the process, such as temperature and extrusion speed. The advantage of the hydrostatic extrusion against the backward extrusion is in a uniform applied pressure on the surface of the material. There is no direct contact with the tool and therefore does not produce any cracks on the surface of the extruded profile. For this

reason, in hydrostatic extrusion process higher compression ratio could be used, and so the material can achieve finer microstructure [9].

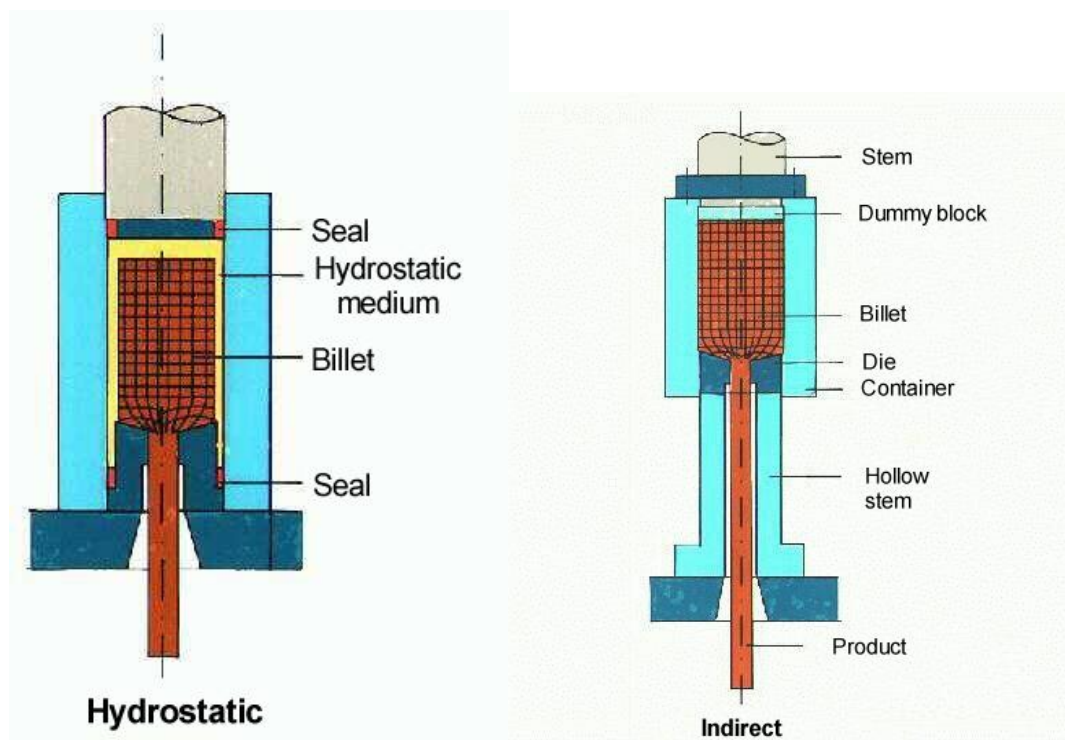


Fig. 1.5. The principle of forward (hydrostatic) and backwards (indirect) extrusion [10]

1. 3. Acoustic emission

1. 3. 1. Definition and method of acoustic emission

Acoustic emission (AE) is defined as an elastic wave, which occurs by sudden release of energy due to local dynamic changes in the structure of the material, which are caused by external or internal forces [11]. This process, from the standpoint of thermodynamics, is short and therefore it can be considered as adiabatic.

Generally, the term AE refers to physical phenomenon in which acoustic signals are observed, e.g. caused by plastic deformation or violation of the investigated material. It also refers to a diagnostic method based on this phenomenon.

This method belongs to the passive non-destructive methods, which gives integral information about the current dynamic state of the material (*in-situ* measurement) and therefore has a high sensitivity to local instabilities.

The disadvantage of the method is the small energy of the majority of generated acoustic pulses, which disappear in the detectors background noise.

1. 3. 2. The sources of acoustic emission

Acoustic emission source is a local process which produces AE signal. The AE signal is a single dynamic process which is caused by rapid release of a certain amount of energy [12].

Depending on the character of elastic waves, two types of AE can be distinguished:

- Continuous emission – elastic waves from large number of sources with a low energy,
- Burst emission – AE signals have the character of time separated pulses with a high energy.

The sources of continuous AE are dislocations, which have dynamic behavior and by their stress field can generate detectable AE. Interaction (annihilation, intersection) and accumulation of dislocations at obstacles, or unpinning from the obstacles lead to continuous AE. Continuous AE also occurs during recrystallization or martensitic phase transformation.

Sources of burst AE are nucleation and propagation of cracks, fracture or corrosion phenomena. The unstable nature of plastic deformation, occurring in certain materials, is caused by twinning or by collective motion of large amount of dislocations. The released energy may be 10 to 14 orders of magnitude greater than in continuous AE [9].

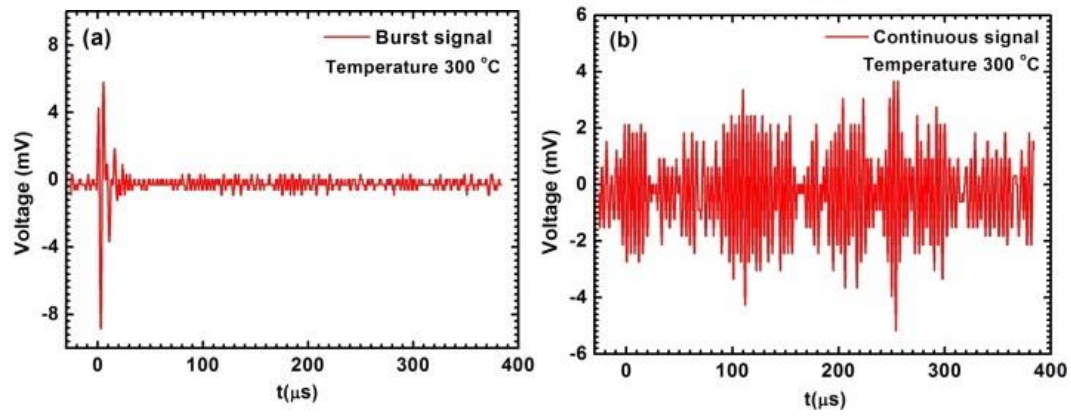


Fig. 1.6. Burst (left) and continuous (right) AE [13]

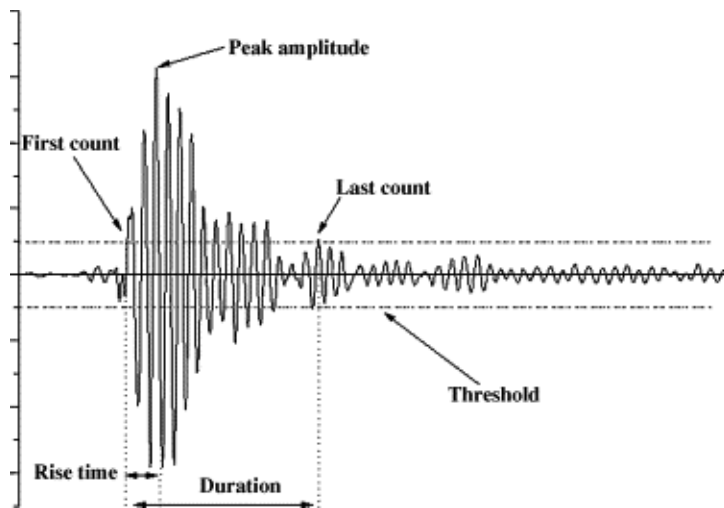


Fig. 1.7. Characterization of burst AE [14]

1. 3. 3. Terminology and basic parameters of AE

- Acoustic emission signal – an electrical signal obtained by detection of one or more acoustic emission events. The signal is described by its amplitude, shape, rise time, duration, energy and its spectrum.
- Acoustic emission event – a local material change giving rise to acoustic emission.
- Emission oscillation – shape of emission signal in one period of such frequency which has the maximum amplitude (peak amplitude) of the entire spectrum.
- Acoustic emission count – the number of times the acoustic emission signal exceeds a threshold (Fig. 1.6.).
- Acoustic emission count rate – the number of the emitted pulses with an amplitude greater than the threshold per a unit time .
- Acoustic emission event rate – the number of the events during a unit time [15].

2. Experiments

2. 1. Investigated materials

Deformation tests were carried out on ZE10 and ZEK100 magnesium alloys whose chemical composition and production conditions are listed in Table 1. (RE – rare earth elements). Both alloys were extruded at 300 °C from the block with original diameter of 93 mm and length of 300 mm. The materials were produced in Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht, D21502 Geesthacht, Germany. The texture measurements are shown in the form of inverse pole figures (Fig 2.1.).

	Chemical composition			Extrusion speed (mm/s)	Extrusion ratio
	Zn (wt.%)	RE (wt.%)	Zr (wt.%)		
ZEK100	1.4	0.2	0.6	0.5	1:30
				11	
ZE10	1.3	0.1	-	11	

Table 1. Chemical composition and preparation conditions of the materials

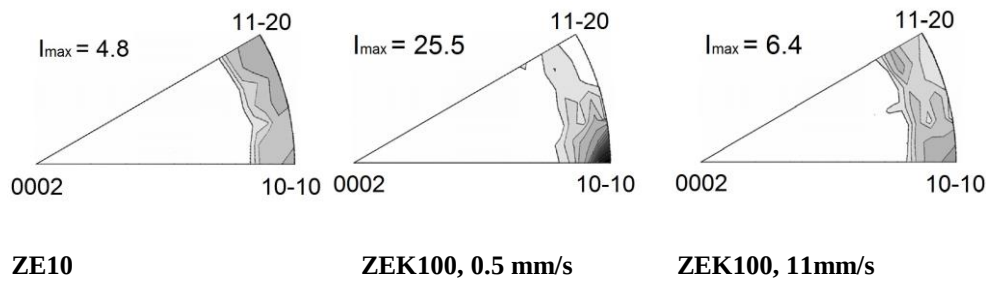


Fig. 2.1. Texture of the alloys

2. 2. Experimental methods

2. 2. 1. Deformation tests

The tension and compression tests were performed in a universal testing machine INSTRON® 5882. The scheme of the device is shown in Fig. 2.2. The samples of 8 mm diameter and the active length of 15 mm were prepared from the extruded Mg alloys.

Deformations were carried out at room temperature (RT) with a constant strain rate of 10^{-3} s^{-1} . The measured data was recorded by using Blue Hill[®] supplied by INSTRON[®] for the testing machine.

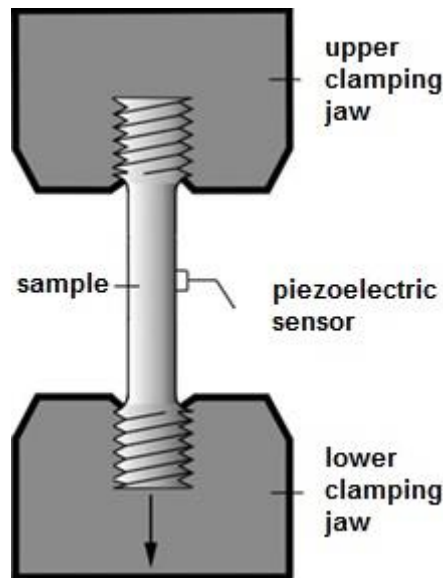


Fig. 2.2. Scheme of the INSTRON[®] aperture

All the data was further processed by program Origin[®] from the company OriginLab[®].

2. 2. 2. The acoustic emission measurements

AE was detected by computer-controlled measuring system DAKEL-IPL from company ZD RPETY-DAKEL (Fig. 2.3.). It allows continuous measurement on four channels with a different amplification (0-20-30-40 dB) at 2 MHz sampling frequency with using 12-bit A/D converter. A preamplifier of 35 dB was used. For measuring AE a piezoelectric sensor MIKRO (the contact surfaces diameter was 2.8 mm [15]) was used. Sensor (Fig. 2.4.) was fixed on a sample by using a clamp and to improve the contact between the sample and the detector silicone grease was used. The threshold level for detecting the AE count rate was set to 30% of the total range of the measurement channel. By comparing the values of individual measuring channels (to prevent overflow or underflow of data) the channel with an amplification of 40 dB was used.



Fig. 2.3. Measuring device DAKEI-IPL [17] Fig. 2.4. MICRO sensor [16]

2. 2. 3. Light microscopy

Samples, before and after deformation, were cut by machine Accutom-50 from Struers[®] along the extrusion direction and perpendicular to this direction. Mounting samples was done by ClaroCit and then they were wet grinding with TegraPol-15 from Struers[®] on SiC grinding papers from 800 to 4000. In addition, they were polished by diamond paste with size of 3 micrometers and finally polished on pat Opchem by using OP-S. The polished surfaces were etched from 7 to 14 seconds with etchant containing of 150 ml ethanol, 40 ml water, 5.5 ml acetic acid and 3-4 g of picric acid.

The microstructure was investigated by microscope OLYMPUS GX51, which was combined with PIXELINE[®] camera connected to the computer. The obtained microstructures were evaluated by using program from NIS – Elements AR[®]. The average grain size d was determined by using the linear intercept method.

3. Results

The microstructures of investigated materials are shown in Figs. 3.1. (ZE10) and. 3.2. (ZEK100).

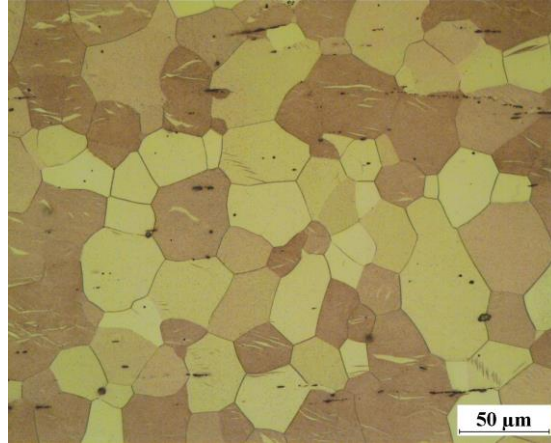


Fig. 3.1. Microstructure of ZE10 alloy, extrusion speed of 11 mm/s

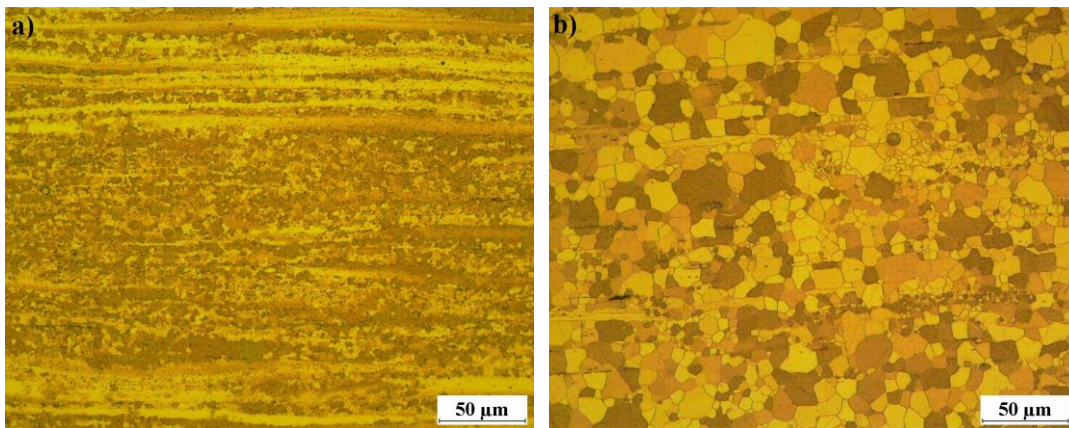


Fig. 3.2. Microstructure of ZEK100 alloy, extrusion speed of a) 0.5 mm/s, b) 11 mm/s

The ZE10 alloy has a homogeneous polyhedral microstructure without any occurrence of elongated grains in the extrusion direction (ED). The average grain size is $(27 \pm 1) \mu\text{m}$. The extrusion speed significantly influences the microstructure of ZEK100 alloys. After extrusion at 0.5 mm/s, the alloy has partially recrystallized microstructure with elongated grains in the ED. In this case, the average grain size cannot be determined by using light microscopy. In ZEK100 alloy, the use of the higher extrusion speed (11 mm/s) leads to bimodal microstructure with an average grain size of $(17 \pm 1) \mu\text{m}$ and $(5.6 \pm 0.5) \mu\text{m}$, respectively. It is

obvious, that by using lower extrusion speed a fine grained, however less homogeneous, microstructure can be achieved (Fig 3.2). Using the same extrusion speed (11 mm/s) for both ZE10 and ZEK100 results in fine grained microstructure in ZEK100.

The time dependence of the tensile stress and the AE count rate is plotted in Figs. 3.3.-3.5. All deformation curves exhibit a typical shape for polycrystalline materials with a linear part at the beginning of the test, what corresponds to elastic deformation.

In the area of elastic deformation, a certain AE activity can be observed and its maximum correlates with a macroscopic yield point (Fig. 3.3., Fig 3.5.). During plastic deformation, the AE activity decreases with increasing strain hardening. The lowest AE activity is observed in the ZEK100 alloy with extrusion speed of 0.5 mm/s, while the highest one is in the ZE10 alloy.

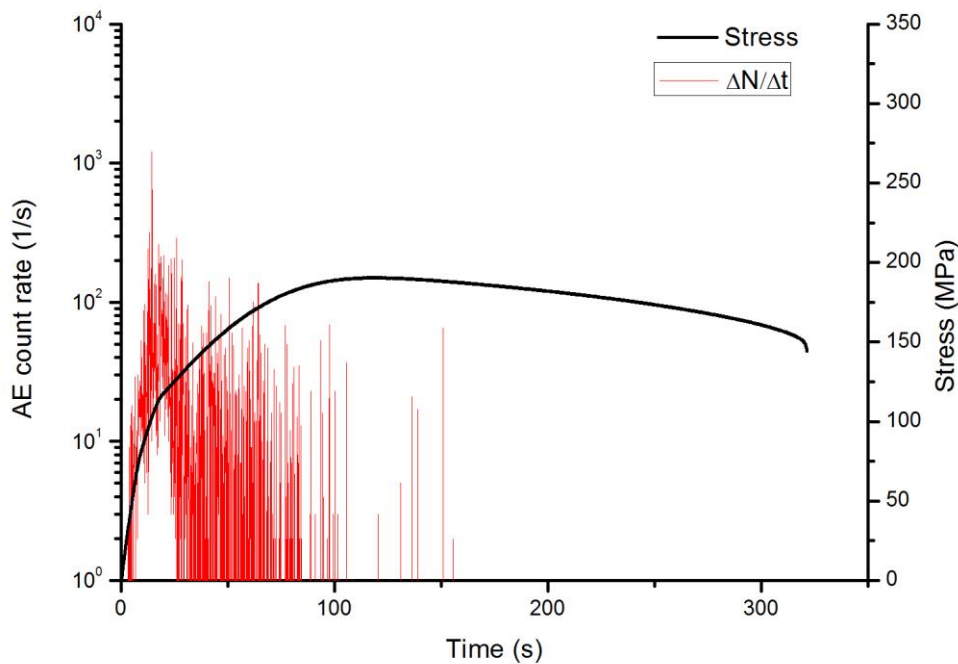


Fig. 3.3. Tensile test of ZE10 alloy with AE measurement

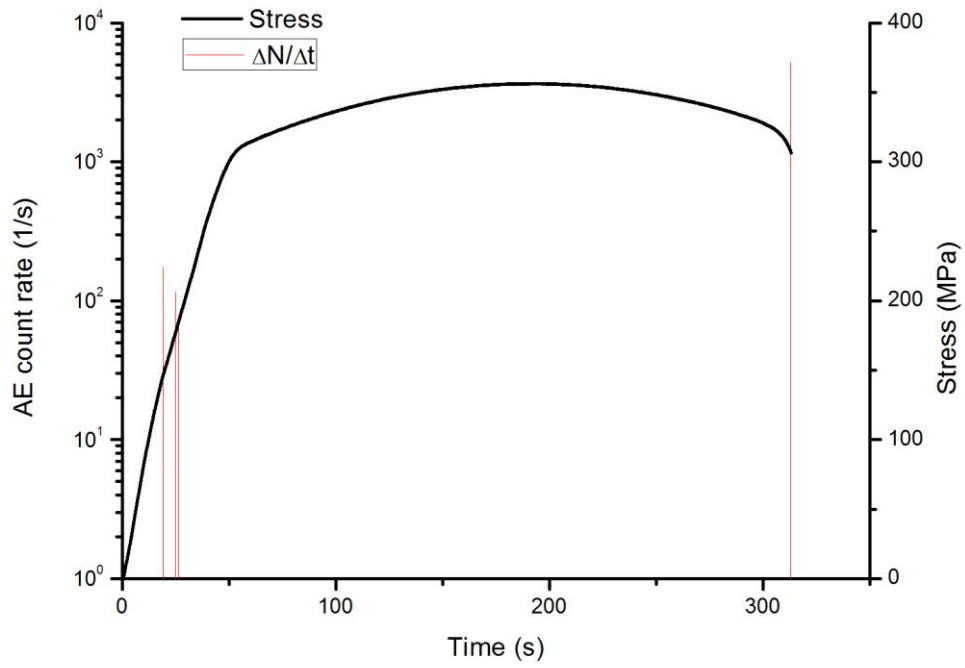


Fig. 3.4. Tensile test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement

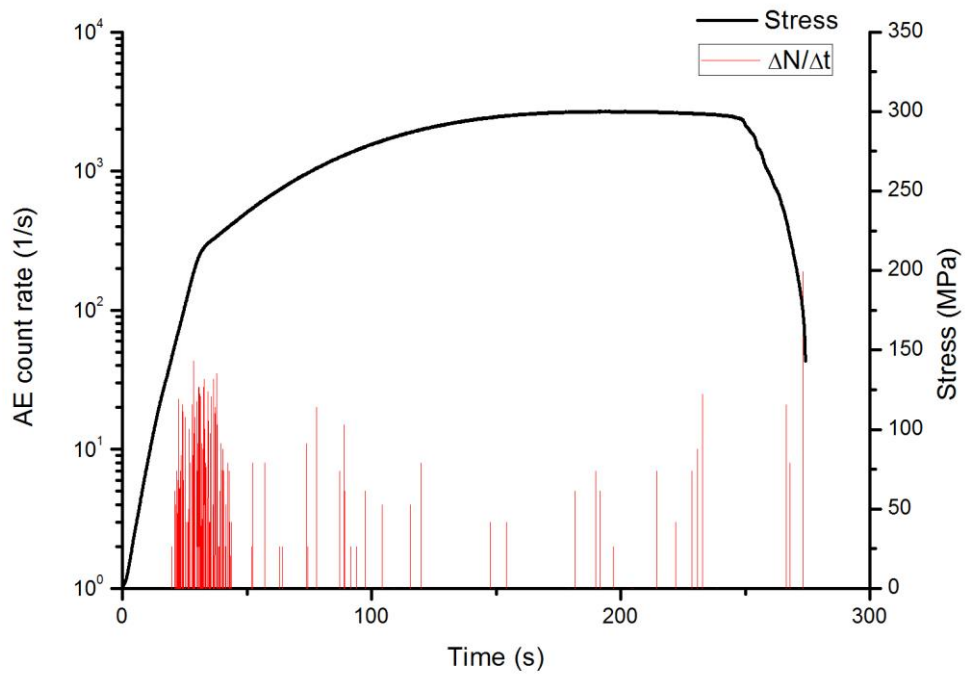


Fig. 3.5. Tensile test of ZEK100 alloy with extrusion speed of 11 mm/s with AE measurement

The time dependence of the compressive stress and the AE count rate is presented in Figs. 3.6. – 3.8.. Similar to the tensile deformation tests, the maximum of the AE activity is observed at the macroscopic yield point and its subsequent decrease is linked to increasing plastic deformation. In compression, the AE activity is significantly higher than in tension. The lowest AE activity is measured in ZEK100 with extrusion speed of 11 mm/s. For ZEK100 alloys two peaks of AE signals are detected. The first peak is located in the area of yield point and the second one is in the area of significant strain hardening. In slower extruded ZEK100 alloy the second AE peak is approximately one order of magnitude lower than the first one. The ZEK100 alloy with the smaller extrusion speed emits the most AE signal during compression, while during deformation in tension almost no AE activity was detected.

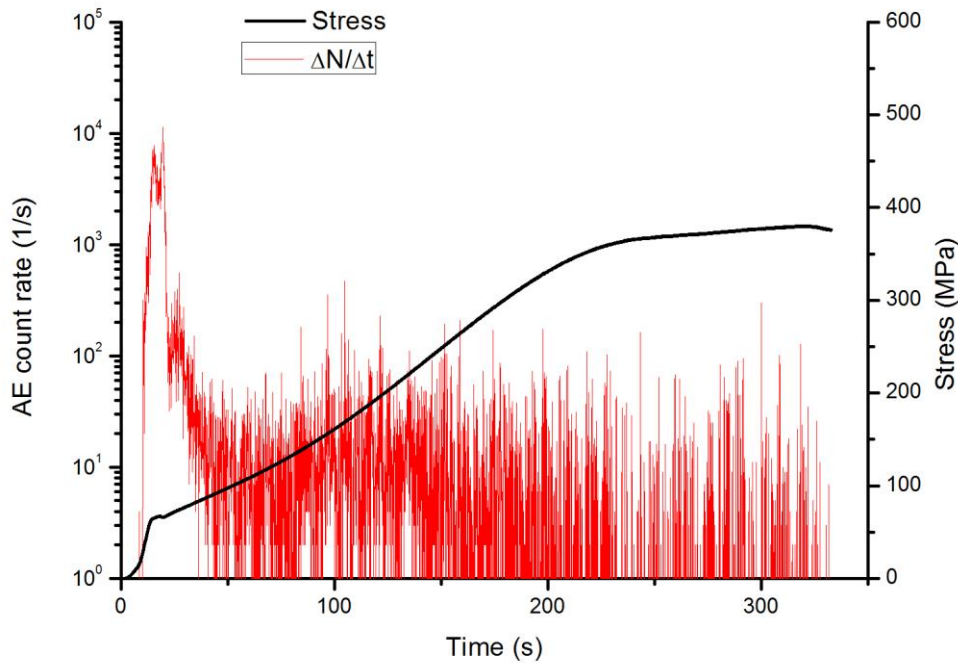


Fig. 3.6. Compression test of ZE10 alloy with AE measurement

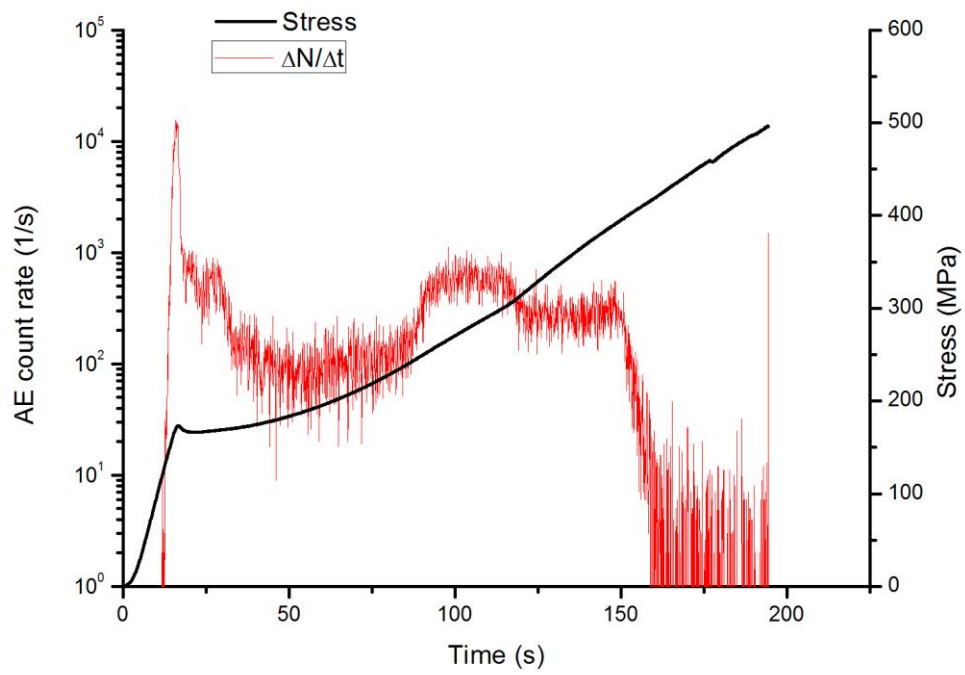


Fig. 3.7. Compression test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement

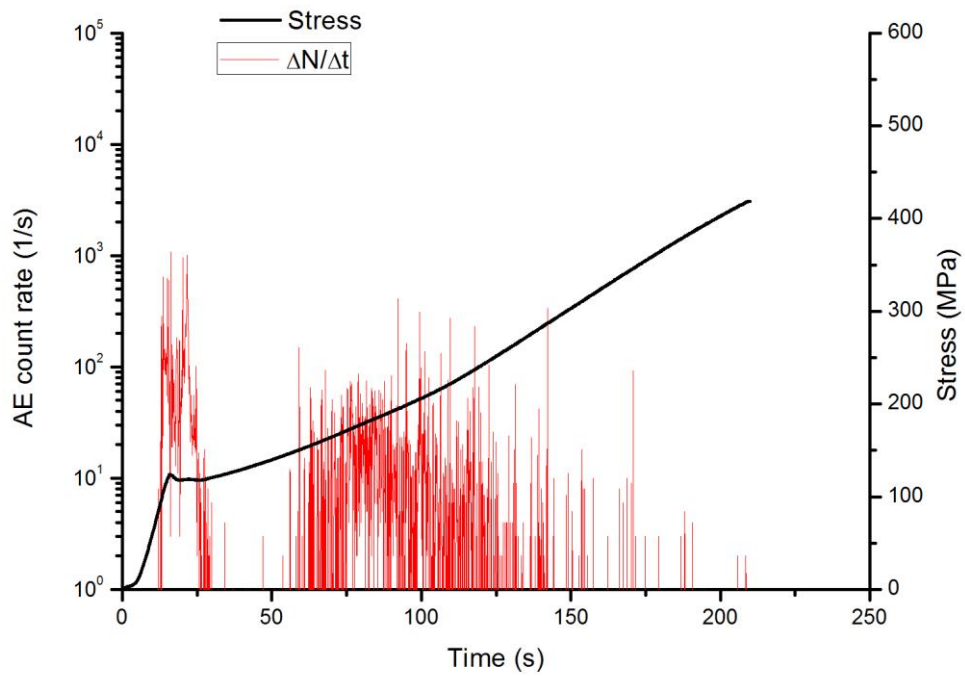


Fig. 3.8. Compression test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement

The microstructure of the ZE10 alloy after compression is shown in Fig 3.9. Microstructure contains a lot of twins, which are distributed throughout the microstructure.

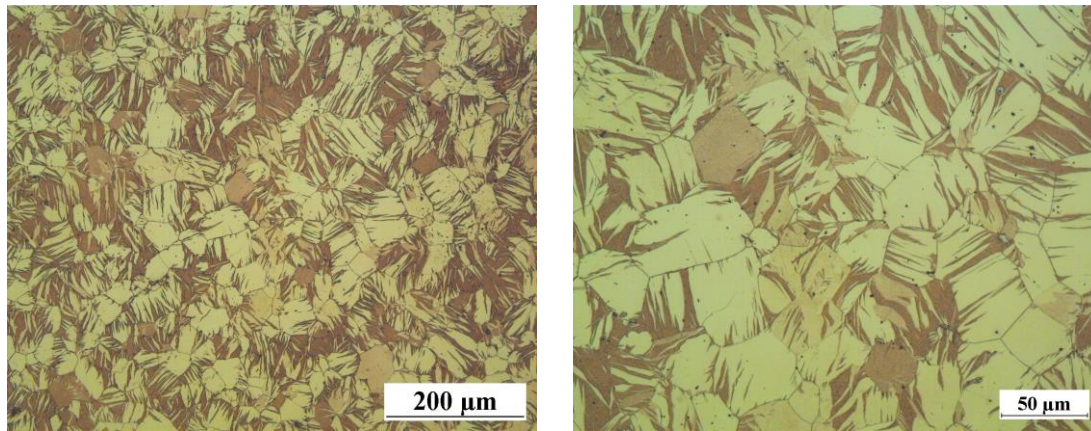


Fig. 3.9. Microstructure of ZE10 alloy after compression

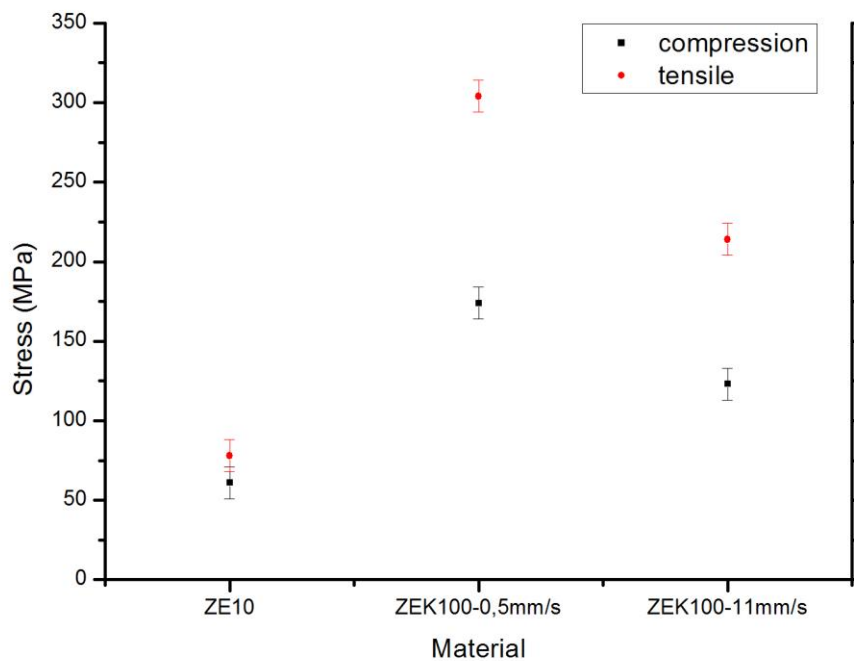


Fig. 3.10. Tension-compression asymmetry at yield point

The values of the yield strength (YS) after deformation in compression and tension are shown in Fig. 3.10. The largest difference between the YS values is observed for the ZEK100 alloy with extrusion speed 0.5 mm/s and the smallest one for the ZE10 alloy. Due to the fact, that we have only one measurement, the error in the determination of the YS was estimated from measurement errors of sample size and the strength.

The stress and the AE count rate vs. time dependence for cyclic loading of the alloys are presented in Figs. 3.11. – 3.13.. The samples were first deformed in compression (positive stress) and subsequently in tension (negative stress) in order to study twinning – detwinning process in Mg alloys. For ZE10 alloy (Fig. 3.11.), the sample was deformed in compression up to 160 MPa and then in tension until fracture. The maximum of the AE count rate correlates with macroscopic yield point and subsequently decreases with increasing plastic deformation. After reaching the 160 MPa and changing the loading direction was not detected any AE activity until reaching the tensile half cycle, where significant activity was detected again. The ZEK100 alloy with slower extrusion speed (0.5 mm/s) was deformed in compression up to 250 MPa and in tension until fracture (Fig. 3.12.). The ZEK100 alloy with higher extrusion speed (11 mm/s) was deformed in compression up to 200 MPa and in tension until fracture (Fig. 3.13.). It is noteworthy that in the tension half cycle of the ZEK100 alloys no AE signal was detected.

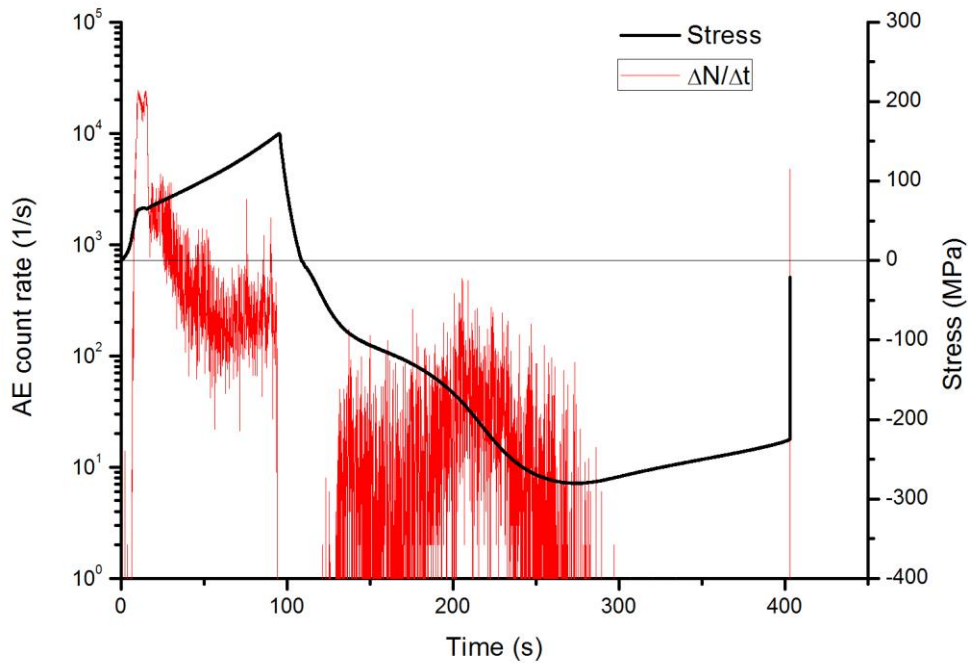


Fig. 3.11. Compression-tension test of ZE10 alloy with AE measurement

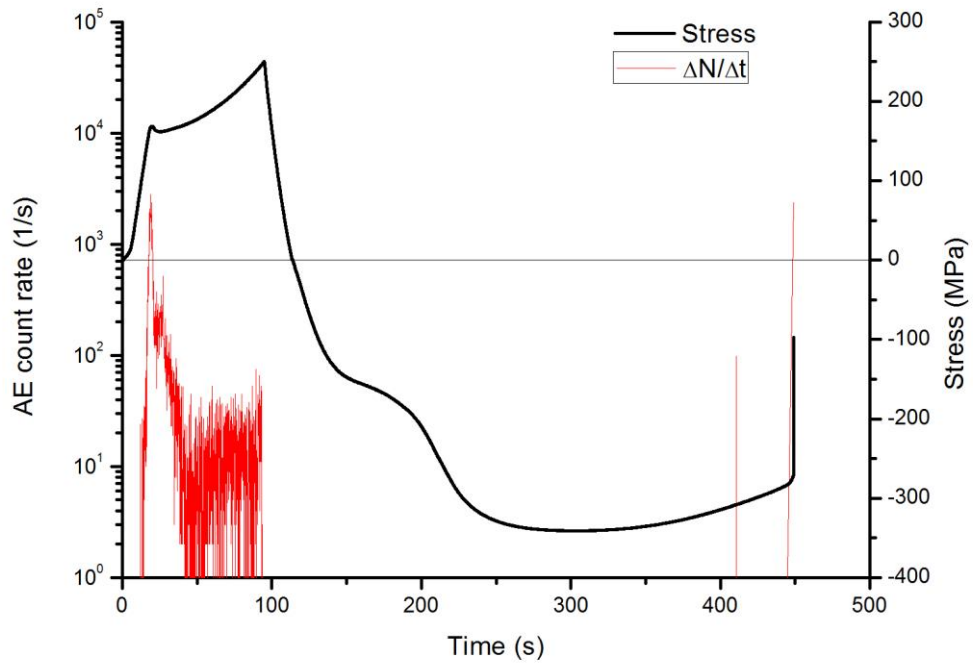


Fig. 3.12. Compression-tension test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement

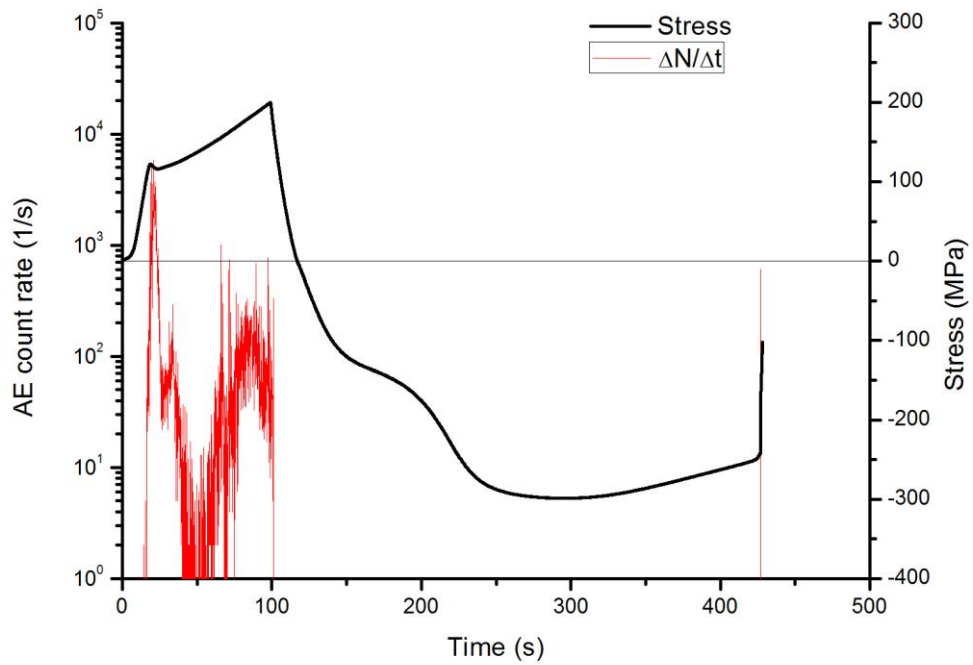


Fig. 3.13. Compression-tension test of ZEK100 alloy with extrusion speed of 11 mm/s with AE measurement

In Fig. 3.14. are plotted the tensile half cycles vs. strain of the ZE10 alloy, which were pre-deformed in compression (up to 80, 160 and 200 MPa). Higher pre-compression leads to higher maximum stress in tensile half cycle. Using pre-compression up to 80 MPa, the tensile stress-strain curve has the typical shape of tensile deformation. With increasing pre-compression, the tensile curves exhibit so called S-shape, which is characteristic for the compression tests in extruded Mg alloys.

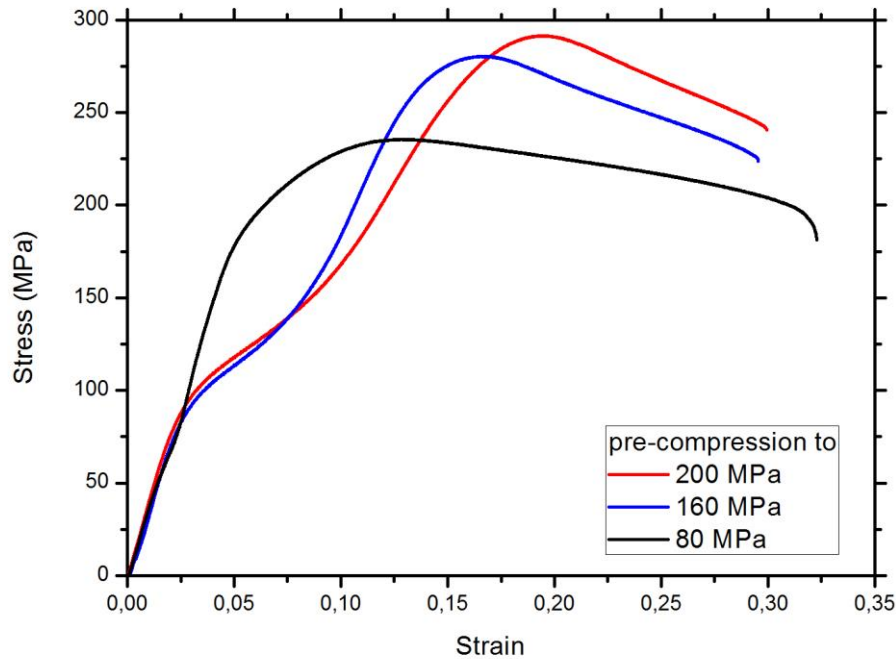


Fig. 3.14. Tensile half cycles of deformation tests of ZE10 alloy after compression up to various stresses

The cyclic test of the ZE10 alloy is shown in Fig. 3.15. The sample was compressed (positive stress) up to 160 MPa and subsequently subjected to tensile loading (negative stress) up to 160 MPa. The value of the YS was increased with the increasing number of cycles. The AE activity in both half cycles (compression and tension) decreases with the increasing number of cycles. The highest AE activity was detected in the first compression half cycle at the macroscopic yield point. In this half cycle two maxima of AE signals were observed at the yield point. In following compression half cycles, only one maximum in the AE count rate per half cycle was detected.

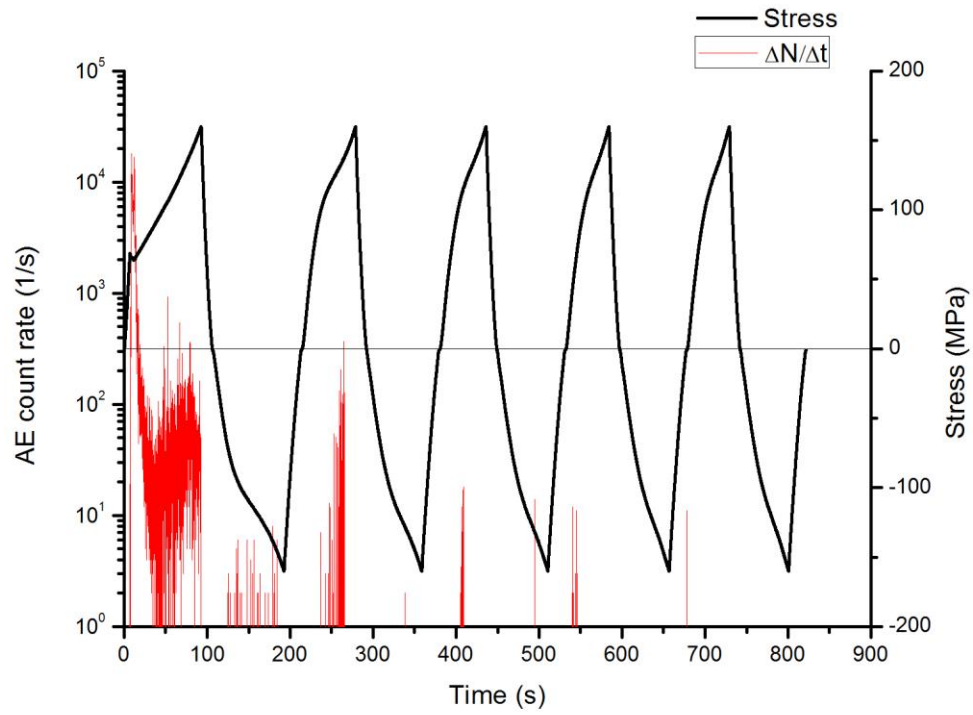


Fig. 3.15. Cyclic test of ZE10 alloy with AE measurement

4. Discussion

Comparison of the microstructures in ZE10 and ZEK100 magnesium alloys (Fig. 3.1. and 3.2.) has shown the influence of the chemical composition and extrusion parameters (extrusion speed) on the final microstructure of Mg alloys. It was observed in [18, 19] that zirconium (Zr) reduces the grain size in aluminum-free Mg alloys. Therefore, the smaller grain size of ZEK100, compared with ZE10 alloy, is the result of the presence of Zr in the alloy. Extrusion speed also affects the grain size, whereas the slower speed leads to a fine-grained microstructure, which is less homogeneous than microstructure after faster extrusion.

The results of the deformation tests are consistent with microstructure observations. Higher values of deformation stress in ZEK100 than in ZE10 alloy can be explained by the Hall-Petch relation [4]:

$$\sigma_{\varepsilon} = \sigma_{\varepsilon_0} + K_{\varepsilon} d^{-1/2}, \quad (1)$$

wherein σ_{ε_0} is the necessary stress for the movement of dislocations, K is material constant (i.e. Hall-Petch parameter) and d is the average grain size.

The greatest AE activity during the tensile deformation is detected for the ZE10 alloy, which has the largest grains. It has been shown in [20] that the grain size has an influence on the AE activity, where the AE count rate decreases with decreasing average grain size. It can be explained by the large free path for collective motion of dislocations in large grains, while in the fine-grained microstructure is higher amount of grain boundaries which are obstacles for the dislocation motion.

In addition to the grain size, a significant factor, which affects the mechanical properties and hence the AE, is the preferred grain orientation (texture). Texture measurements, presented in form of inverse pole figures (Fig. 2.1.), have shown in all studied Mg alloys a strong texture with basal planes (0001) nearly parallel to the extrusion direction (ED). This orientation is favorable for the activation of twins if the compression stress is applied along the ED. Due to twinning, the basal planes are reoriented by 86.3° and the dislocation slip can continue in these basal planes. During tensile loading, twinning almost does not contribute to the overall deformation [21]. Twinning can be activated either during compressive loading in direction parallel to basal planes or during tensile loading in a direction perpendicular to the basal planes [22].

In tensile tests, the maximum of AE at the beginning of plastic deformation is usually explained by the massive multiplication of dislocations. The following decrease in the AE count rate is result of forest dislocations.

In compression tests of extruded Mg alloys, twinning is an excellent source of AE [20] and it is responsible for the strong AE signals. The reduce of the AE activity is caused by decrease of twinning intensity, which increases the dislocation density in individual slip systems, their interactions, and thereby leads to the reduction of the mean free path for their movement [9].

During compression tests of ZEK100 alloy with slower extrusion speed, higher AE activity than in faster extruded ZEK100 alloy was detected, what is not consistent with the previous observations [20, 23, 24]. This may be caused by the fact that the microstructure was not fully recrystallized and contains several large grains, which may form larger twins (stronger AE). Therefore, despite a smaller average grain size, higher AE activity in slower than in faster extruded ZEK100 alloy was observed.

Two AE peaks were observed in both ZEK100 alloys, which were subjected to compression loading. Similar AE activity was described in the extruded AZ31 alloy with inhomogeneous microstructure [25]. Electron Back-Scattered Diffraction (EBSD) images have shown that the activation of twinning depends on the grain size. First twins are formed in the larger grains and with increasing stress twinning process starts in smaller ones.

Large compression-tension asymmetry at the yield stress (YS) is the result of the hcp lattice and strong deformation texture, which was formed during the extrusion process. Compression along the ED leads to the formation of twins, which are responsible for low YS [26]. It is obvious that the ZE10 alloy has the lowest stress values and also the lowest compression-tension asymmetry compared to ZEK100 alloys (Fig. 3.11.). It has been proven that grain refinement in Mg alloys may reduce the tension-compression asymmetry at the YS due to the reduction of the influence of twinning on the total deformation [27]. However, in our case the YS asymmetry increases with decreasing average grain size. This can be explained by the inhomogeneous microstructure. The ZE10 alloy has a homogeneous microstructure with the largest average grain size among all three alloys. According to the Hall-Petch relation, ZE10 alloy also has the lowest yield stress. On the contrary, the ZEK100 alloys have inhomogeneous microstructure with the presence of large grains, which are elongated in the ED. The average grain size is smaller than in ZE10 what results in higher YS values in both loading mode.

In ZEK100 alloys during low cyclic loading in tensile half cycles no AE signals were detected. Absence of AE is caused by fine-grained microstructure and a relative high dislocation density due to pre-compression. Grain boundaries and sessile dislocations act as barriers for movement of dislocations. In the ZE10 alloy, quite high AE activity is detected which can be produced by the collective motion of dislocation and by nucleation of new twins. In the tensile half cycle of extruded Mg alloys, another phenomenon can be also observed. It is called detwinning and stands for vanishing twins, which were created in compression half cycle [28]. Twins can disappear or can become narrower under reverse loading. AE signals are recorded only due to nucleation of twins, because their growth or thickening is slow process that cannot produce detectable AE.

The higher applied compressive stress, i.e. more produced twins in the microstructure, the stress-strain curves tensile half cycle become similar to compression curves with its characteristic S-shape (Fig. 3.14.). The twins created in compression half cycle can disappear in tensile half cycle in order to maintain the compatibility of plastic deformation.

It has been shown that the stress which is needed for the nucleation of new twins is higher than stress required for their “motion”, i.e. enlargement or reduction the twin boundaries [29]. This implies that untwinning, i.e. contraction of existing twins and re-orientation to the original orientation of the parent grain has priority over the nucleation and growth of new twins. Therefore, no AE activity in tensile half cycles is detected due to fine grained microstructure and detwinning process.

From the cyclic loading test of ZE10 alloy is clear that the AE activity decreases with increasing number of cycles. The absence of the AE activity in the fifth cycle shows a large strain hardening and the saturation of twins.

6. Conclusions

Various extrusion speeds lead to significant changes in the microstructure of ZEK100 alloy. With increasing extrusion speed the microstructure is more homogeneous. The presence of Zr in the ZEK100 alloy helps to stabilize the microstructure, what results in a fine-grained microstructure compared to ZE10 alloy. Based on the texture measurements (basal planes are parallel to the extrusion direction), which were performed in Germany and the AE measurements it can be stated that twinning is more important during compression test.

The AE technique was used for the study of twinning activity during compression tests. Two maxima in the AE count rate, which were observed in both ZEK100 alloys, can be explained by their inhomogeneous microstructure. The first AE maximum is related to the macroscopic yield point, at which a massive multiplication of dislocations and formation of twins occur. The second AE maximum is caused by twinning in smaller grains. First twins are formed in bigger grains and with increasing plastic deformation twinning occurs in smaller grains.

The tension-compression asymmetry was also studied. It was found for the ZEK100 alloy that with decreasing average grain size the asymmetry increases, what is in contrast with previously published results. This “unusual” behavior needs to be attributed to the inhomogeneous microstructure, where the average grain size is not the appropriate parameter for the characterization of the inhomogeneous microstructure. As it was already mentioned, the inhomogeneous microstructure influences the activation of twins and therefore is also responsible for changes in mechanical properties of Mg alloys. Higher twinning activity leads to lower YS.

During low cyclic loading of ZE10 and ZEK100 alloys, a different AE activity was observed. In the ZE10 alloy, the AE signals in the tensile half cycle were detected while in the ZEK100 alloys no AE activity was observed.

For the ZE10 alloy, the deformation curves of tensile half cycles after different pre-compression were compared. The higher is pre-compression the more similar is the shape of the tensile stress-strain curve to the compression curve (characteristic S-shape). Such behavior is the result of so called detwinning process, which occurs during subsequent tensile loading of pre-compressed samples and is also manifested by shrinkage of twins.

Options for further research

From obtained results it is possible to propose an appropriate extrusion speed, which will lead to fine-grained and homogeneous microstructure and also to required mechanical properties.

The raw AE signal can be subjected to further processing by using of AE parameters in order to distinguish dislocation slip from mechanical twinning. Characterization and better understanding of twinning in Mg alloys contributes to achievement such an improvement of their mechanical properties.

Further statistical processing of the obtained AE parameters (i.e. the amplitude or the energy of emission events) can be used for a study of dislocation dynamic during plastic deformation.

References

- [1] http://kdf.mff.cuni.cz/~koudelkova/U3V/Drozd_horcik.pdf (status to the day 01/12/2014).
- [2] J. Kaiser: *Dr. – Ing. Dissertation: Untersuchung über das Auftreten von Geräuschen beim Zugversuch, TU München* (1950).
- [3] K. Máthis: *Doktori értekezés: Deformációs mechanizmusok magnézium alapú könnyűfém ötvözetekben, ELTE, Általános Fizika Tanszék* (2004).
- [4] P. Kratochvíl, P. Lukáč, B. Sprušil: *Úvod do fyziky kovů I., Praha* (1984).
- [5] <http://www.intechopen.com/source/html/32760/media/image19.jpeg> (status to the day 01/12/2014).
- [6] R. von Mises: *Mechanik der plastischen Formaenderung von Kristallen, Z. Angew. Math. Mech.*, 6 (1928) 85.
- [7] R. L. Bell, R. W. Cahn: *The Dynamics of Twinning and the Interrelation of Slip and Twinning in Zinc Crystals, Proc. Rpy. Soc.* 239 (1957) 494.
- [8] <https://wwwf.imperial.ac.uk/earthscienceandengineering/rocklibrary/learntwinning1.php> (status to the day 01/12/2014).
- [9] P. Dobroň: *Doktorská dizertačná práca: Štúdium mechanických a fyzikálnych vlastností nových, konštrukčných materiálov na báze horčíka metódou akustickej emisie, MFF UK* (2007).
- [10] <http://www.fzs.tu-berlin.de/lib/html/en/> (status to the day 20/05/2013).
- [11] *Akustická emise – Metodické pokyny, Smernica celoštátneho defektoskopického strediska, Brno* (1987).
- [12] *Zkoušení materiálů a výrobků metodou akustické emise – Názvosloví, Smernica celoštátneho defektoskopického strediska* (1987).
- [13] J. Xu, E.-H. Han, X. Wu: *Acoustic emission response of 304 stainless steel during constant load test in high temperature aqueous environment, Corros. Sci.* 63 (2012)91.
- [14] B. Legros, P.-X. Thivel, Y. Bultel, M. Boinet, R.P. Nogueira: *Acoustic emission: Towards a real-time diagnosis technique for Proton Exchange Membrane Fuel Cell operation, J. Power Sources* 195/24 (2010) 8124.
- [15] Acoustic Emission Standard Terminology ASTM E1316-05
- [16] <http://www.dakel.cz/index.php?pg=prod/sens/micro> (status to the day 01/12/2014).
- [17] <http://www.dakel.cz/index.php?pg=prod/dev/ipl> (status to the day 01/12/2014).

- [18] D. H. StJohn, M. Qian, M. A. Easton, P. Cao, Z. Hildebrand: *Grain Refinement of Magnesium Alloys*, *Metall. Mater. Trans A* 36 (2005) 1669.
- [19] M. Qian, A. Das: *Grain refinement of magnesium alloys by zirconium: Formation of equiaxed grains*, *Scripta Mater.* 54 (2006) 881.
- [20] C. R. Heiple, S. H. Carpenter: *Acoustic Emission Produced by Deformation of Metals and Alloys – A Review: Part I*, *J. Acoust. Emission* 6 (1987) 177.
- [21] I. J. Polmear: *Light Alloys*, Melbourne (2006).
- [22] W. F. Hosford: *The Mechanics of Crystals and Textured Polycrystals*, Oxford University Press, Oxford (1993).
- [23] C. R. Heiple, S. H. Carpenter: *Acoustic Emission Produced by Deformation of Metals and Alloys – A Review: Part II*, *J. Acoust. Emission* 6 (1987) 215.
- [24] J. Bohlen, P. Dobroň, J. Swiostek, D. Letzig, F. Chmelík, P. Lukáč, K. U. Kainer: *On the influence of the grain size and solute content on the AE response of magnesium alloys tested in tension and compression*, *Mater. Sci. Eng. A* 462 (2007) 302.
- [25] P. Dobroň, F. Chmelík, S. Yi, K. Parfenenko, D. Letzig, J. Bohlen: *Grain size effects on deformation twinning in an extruded magnesium alloy tested in compression*, *Scripta Mater.* 65 (2011) 424.
- [26] P. Dobroň, F. Chmelík, K. Parfenenko, D. Letzig, J. Bohlen: *On the Effect of the Extrusion Speed on Microstructure and Plastic Deformation of ZE10 and ZEK100 Magnesium Alloys - an Acoustic Emission Study*, *Acta Phys. Pol. A* 122/3 (2012) 593.
- [27] J. Jain, W. J. Poole, C. W. Sinclair, M. A. Gharghouri: *Reducing the tension–compression yield asymmetry in a Mg–8Al–0.5Zn alloy via precipitation*, *Scripta Mater.* 62 (2010) 301.
- [28] Y.N. Wang, J.C. Huang: *The role of twinning and untwinning in yielding behavior in hot-extruded Mg–Al–Zn alloy*, *Acta Mater.* 55 (2007) 897.
- [29] R. E. Reed Hill, R. Abbaschian, *Physical Metallurgy Principles*, PWS Publ. Company, Boston (1994).

List of figure captions

1.1. Edge (a) and screw (b) dislocation	4
1.2. Basic slip planes in hexagonal materials.....	5
1.3. Hexagonal lattice.....	5
1.4. Twin plane.....	6
1.5. The principle of forward (hydrostatic) and backwards (indirect) extrusion	7
1.6. Burst (left) and continuous (right) AE	9
1.7. Characterization of burst AE	9
2.1. Texture of the alloys.....	11
2.2. Scheme of the INSTRON [®] aperture	12
2.3. Measuring device DAKEL-IPL	13
2.4. MICRO sensor.....	13
3.1. Microstructure of ZE10 alloy, extrusion speed of 11 mm/s.....	14
3.2. Microstructure of ZEK100 alloy, extrusion speed of a) 0.5 mm/s, b) 11 mm/s.....	14
3.3. Tensile test of ZE10 alloy with AE measurement.....	15
3.4. Tensile test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement	16
3.5. Tensile test of ZEK100 alloy with extrusion speed of 11 mm/s with AE measurement	16
3.6. Compression test of ZE10 alloy with AE measurement	17
3.7. Compression test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement	18
3.8. Compression test of ZEK100 alloy with extrusion speed of 11 mm/s with AE measurement	18
3.9. Microstructure of ZE10 alloy after compression	19
3.10. Tension-compression asymmetry at yield point.....	19
3.11. Compression-tension test of ZE10 alloy with AE measurement	20
3.12. Compression-tension test of ZEK100 alloy with extrusion speed of 0.5 mm/s with AE measurement.....	21
3.13. Compression-tension test of ZEK100 alloy with extrusion speed of 11 mm/s with AE measurement.....	21

3.14. Tensile half cycles of deformation tests of ZE10 alloy after compression up to various stresses.....	22
3.15. Cyclic test of ZE10 alloy with AE measurement.....	23

List of abbreviations and symbols

AE	acoustic emission
Al	aluminum
CRSS	critical resolved shear stress
d	grain size
hcp	hexagonal close packed
K	material constant
Mg	magnesium
RE	rare earth elements
RT	room temperature
YS	yield strength
Zn	zinc
Zr	zirconium
$\sigma_{\epsilon 0}$	stress needed for the movement of dislocations