Mechanisms of plastic instability and fracture of compressed and tensile tested Mg-Li alloys investigated using the acoustic emission method

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ABSTRACT. The results of the investigation of both mechanical and acoustic emission (AE) behaviors of Mg4Li5Al alloy subjected to compression and tensile tests at room temperature are compared with the test results obtained using the same alloy and loading scheme but at elevated temperatures. The main aim of the paper is to investigate, to determine and to explain the possible influence of factors related with enhanced internal stresses such as: segregation of precipitates along grain boundaries or solute atoms along dislocations (Cottrell atmospheres) or dislocation pile-ups at grain boundaries which create very high stress concentration leading to fracture. The results show that the plastic instabilities are related to the Portevin–Le Châtelier phenomenon (PL effect) and they are correlated with the generation of AE peaks. The fractography of breaking samples was analyzed on the basis of light (optical), TEM and SEM images.

KEYWORDS. Lightweight Alloys; Acoustic Emission; Fracture; Portevin–Le Châtelier Phenomenon; Twinning; Dislocations; Shear Bands.

INTRODUCTION

Unsatisfying ability to plastic deformation at elevated temperatures, mostly leading to the reduction of deformability and/or hot plasticity is characteristic for many metals and alloys [1-4]. The occurrence of hot-shortness phenomenon, followed by the fracture of intercrystalline character is an important reason for technological difficulties. Our previous investigations of mechanical properties of alloys with the application of AE method were conducted for the Mg-Li-Al and related composites generally in the context of the method of intensive deformation processes [5-9] leading to their excellent mechanical properties, such as great strength and plasticity or even superplasticity. On the other hand, our investigations of metal and alloy plastic instability using the AE technique were carried out [10-12] mainly in the context of basic aspects of PL effect, twinning or shear band in both poly- and single metal and alloy crystals. The fracture and strengthening properties of Mg-Li based alloys (and composites) were investigated, for example in [13-16], but without the use of AE method, which proved to be a very useful technique of material examination. In this work the results of the investigations of the correlation between the AE phenomenon, the plastic instability, induced by PL effect, twining or shear bands, and the both, intergranular and transcrystalline fracture of Mg4Li4Zn and Mg4Li5Al alloys subjected to tensile and compression tests at wide range of elevated temperatures are presented.

Alloys based on magnesium with lithium, as the lightest ones from among the known metallic construction materials, are very attractive from the point of view of their application as the materials for light, yet durable constructions to be used in the automotive industry (e.g. car engine housings) or aerospace (e.g. light housings of computers). The basic Mg-Li alloys exist in three phase areas. The hexagonal α phase appears in the concentration range of Li up to 4 wt.%. If the content of Li is more than 12 wt.% - the β phase of cubic lattice occurs. The alloys of Li content from 4% up to 12 wt.% form the $\alpha+\beta$ two-phase mixture. The mechanical properties of α phase are worse from that of the β phase which is more plastic and thus reveals good machinability and weldability. Alloying additions, e.g. Al (or Zn) from 3% to 5%, slightly increase the density of the alloy, but lead to the precipitation of coherent particles of transition phase, θ-$MgLi_2Al$, which additionally strengthens the matrix and leads to the improvement of mechanical properties [14]. The present paper addresses the optical microscopic, as well as TEM and SEM observations of the failure of samples after tensile and compression tests.

EXPERIMENTAL

Compression and tensile tests

The compression tests were carried out using INSTRON-3382 tensile testing machine, additionally equipped with a specially constructed channel-die which guaranteed plastic flow only in the compression direction (normal direction – ND) and in the direction parallel to the channel axis (elongation direction – ED). In this way the plane state of strains was ensured, since the deformation was impossible in the direction perpendicular to the channel walls (transverse direction – TD). The traverse velocity of the testing machine was 0.05 mm/min (the compression speed was $10^{-3}s^{-1}$). Samples of the Mg4Li5Al alloys for compression tests had the cubic shape of side 10 mm. The overall look on the testing arrangement and the instrumental details are presented in Fig.1.

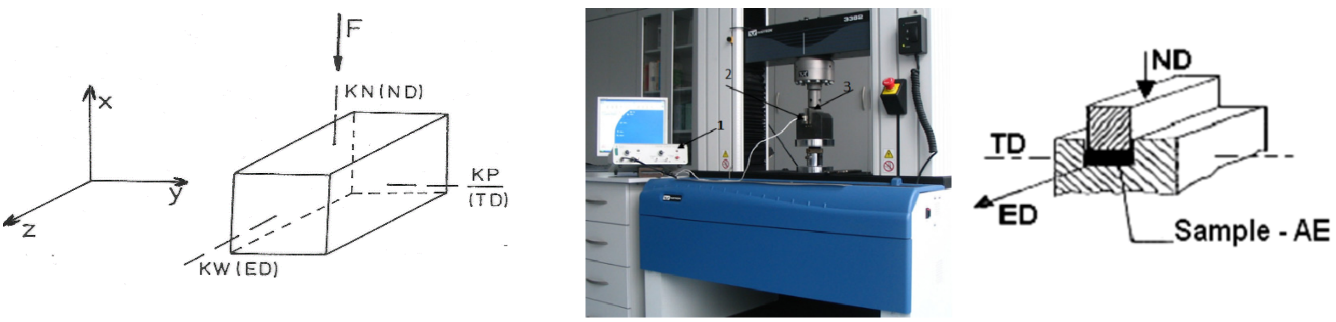


Figure 1: The experimental set-up used to record the AE signals generated in compressed samples:1 – AE analyser, 2 - AE sensor, 3 – Channel-die used as sample holder.

The Mg-Li and Mg-Li-Al alloys were produced in cooperation with the Institute of Materials and Machine Mechanics of the Slovak Academy of Sciences in Bratislava. The basic Mg-Li alloys were obtained by the method of induction melting of magnesium of 99.99% purity and lithium of 99.5% purity. Series of Mg-Li and Mg-Li-Al alloys used in this study were prepared by casting of raw materials in a steel crucible at 800°C with subsequent pouring into a cooled steel mould in a chamber of vacuum induction furnace (Balzers) under low argon pressure (1000 Pa, of 99.999% purity) after previous evacuation (10^{-2}Pa). The applied preparation procedure was similar to that reported in [15] and used also in our previous works [5-9]. Then, the alloys were machined into standard specimens with rectangular cross-section. The tensile tests at elevated temperatures were carried out using a special temperature chamber connected with the Zwick 1200 testing machine using a flat dog-bone samples of operating dimensions 2x6 mm. The elongation was measured with a laser extensometer. The force was recorded with load cell at 100 kN capacity. A temperature chamber was used to control the test temperature. The specimens were elongated at RT, 50°C, 100°C, 150°C and 200°C. The strain tests were performed at the same speed as the above mentioned one.

Acoustic emission measurements

The AE method has been applied to the investigations of poly- and single crystals of metals and alloys by the authors of the present paper for many years [5,7,10,17]. The investigations concentrated on explaining the correlations between AE descriptors and the mechanisms of deformation of the materials subjected to the tensile and compression tests. The obtained results allowed the authors to put forward the following thesis: the dominant contribution to the recorded AE signals is derived from the collective movement of many dislocations, associated with their acceleration as well as with the synchronized annihilation of many dislocations, including the annihilation at the free surface of the deformed material.

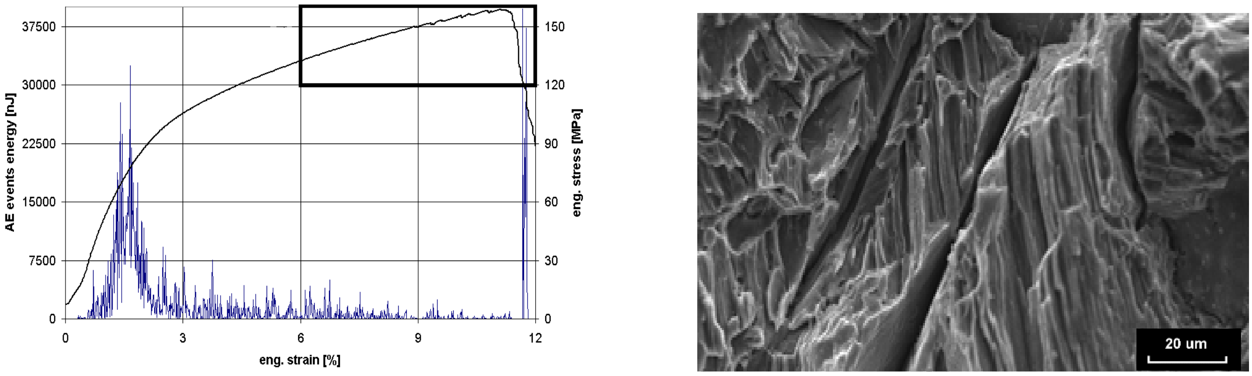
The measuring system of AE signal was functionally coupled with the stress/strain testing machines and it was described in more details in [5,18]. A broad-band piezoelectric sensor (standard WD type, certified by Physical Acoustics Corporation) enabled to record the acoustic pulses in the frequency range from 100 kHz to 1 MHz. AE signal processing unit was realized with application of the 9812 ADLINK type card hosted in a PC computer. Owing to the suitable software, the analysis of the energy and the time duration of the individual events could be carried out, because the dedicated program determined the time of AE event occurrence, its maximum amplitude and the moment of a significant decline of AE signal amplitude.

In the case of compression test the contact between the detector and the sample in a channel-die was maintained by means of a steel rail of a shape of rectangular prism of 100 mm length and 10x10 mm cross section which formed a natural waveguide. In order to eliminate the undesired effects of friction against the channel walls, each sample was covered with Teflon foil. Since there was no possibility to place the AE sensor directly in the sample undergoing the tension test in high-temperature chamber therefore the AE sensor was coupled with elastic spring to the steel clamps which was holding the sample and was operating at room temperature condition due to the contact with the machine framework. In other words the waveguide was attached to grips and mounted outside the temperature chamber. The AE sensor was connected to the waveguide. The amplification of the AE analyser was 86 dB and the threshold voltage of the discriminator was 1.17 V. The output signal was converted into voltage and amplified with a low-noise charge-sensitive preamplifier. A full-wave rectifier drove the integrator at the output of which an envelope of a single AE pulse was obtained. The signals from the mean-value detector were transmitted directly to the voltage discriminator; those which exceeded the threshold level being counted only once, corresponding to a single recorded AE event. The amplitude and duration of the AE event was measured via analog to the digital acquisition system what enabled the calculation of the energy E of AE events using the approximate formula, $E = 0.5\, v^2_{max}\, \Delta t$, where v_{max} was the maximum value of AE signal in the course of the event, and Δt was its duration.

RESULTS AND DISCUSSION

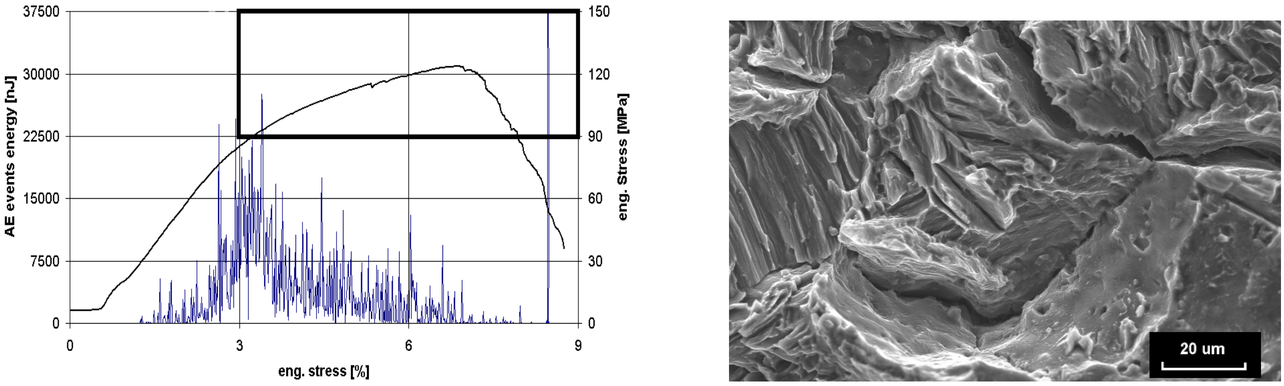
Mg4Li5Al alloys – tensile tests

Figs.2 to 5 show the results of preliminary examinations of the influence of plastic instabilities on the fracture of tensile tested Mg4Li5Al alloys at room (Fig.2a), and elevated temperatures 100ºC (Fig.3a), 150ºC (Fig.4a) and 200ºC (Fig.5a) together with the corresponding SEM images of fracture (Fig.2b to 5b). They show that the plastic instabilities, related to the PL effect, twinning and/or shear band are correlated with the generation of AE events, and affect the final form of fracture. It is necessary to note here that the maximally high AE level at the beginning of the test is related to the yield point (including microplasticity) and it is a result of the creation and operation of dislocation sources. This is a well known fact in literature (see e.g. [8]), and we discuss the AE behavior after this maximum. Similarly, the high AE at the end of test is related to the breaking of the sample (with exception, sporadically, when local contact between the sensor and the sample at 200°C was probably not completely correct, as in Fig.8).



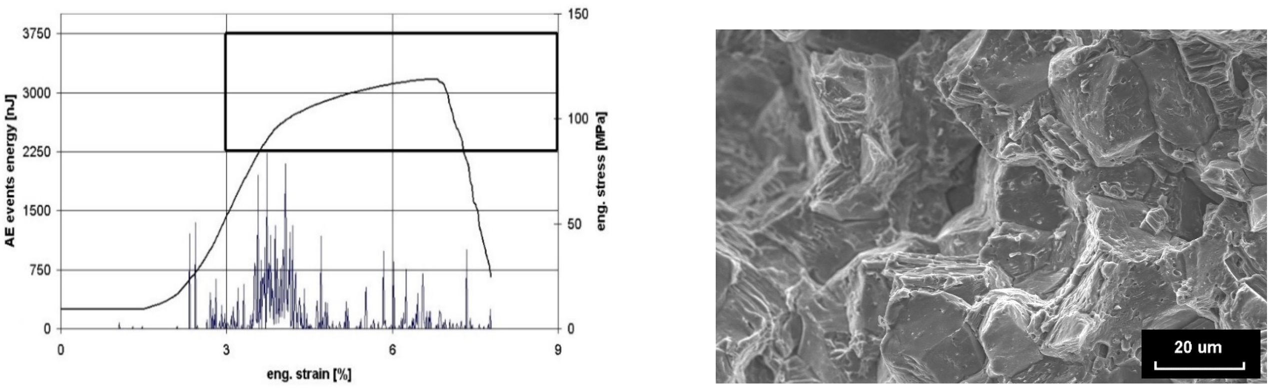
(a) (b)

Figure 2: AE events energy and engineering stress versus time during tensile testing of as-cast Mg4Li5Al at room temperature (a) and corresponding SEM image of fracture (b).



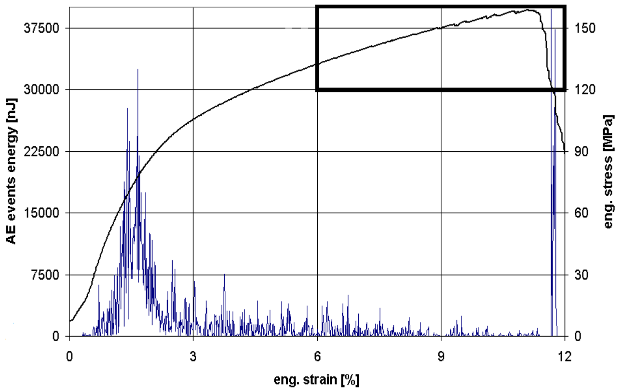
(a) (b)

Figure 3: AE event energy and engineering stress (a) versus time during strain test of Mg4Li5Al at elevated temperature (100°C), and corresponding SEM image of fracture (b).

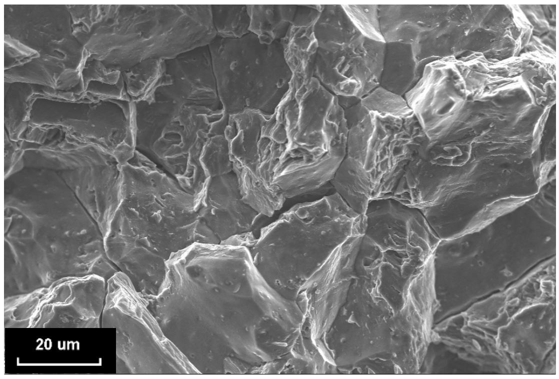


(a) (b)

Figure 4: AE event energy and engineering stress (a) versus time during strain test of Mg4Li5Al at elevated temperature (150°C), and corresponding SEM image of fracture (b).



(a)



(b)

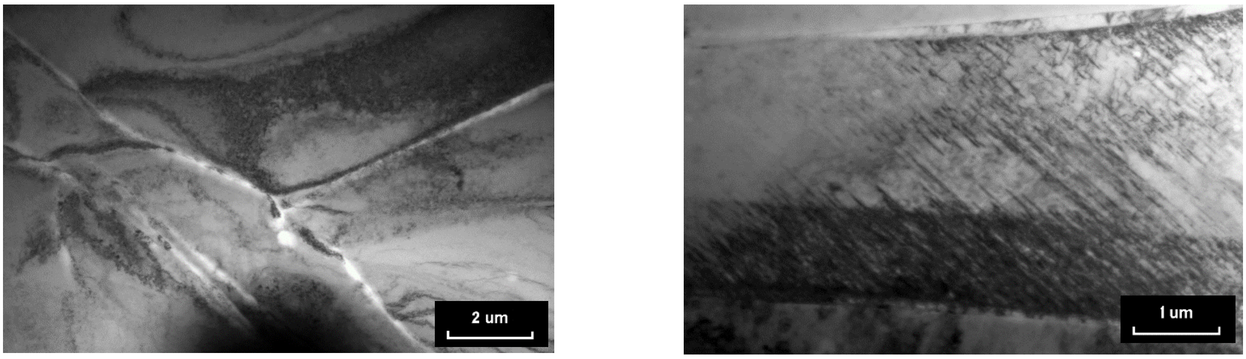
Figure 5: AE event energy and engineering stress (a) versus time during strain test of Mg4Li5Al at elevated temperature (200ºC), and corresponding SEM image of fracture (b).

The analysis of the AE and PL effects and their relation to the fracture (Figs.2 to 5) is performed with reference to the situation at room temperature (Fig.2a), where the fracture is of transcrystalline character (Fig.2b). In this case the brittle (fissile) fracture is bound with discontinuities of the fault type and the contribution of surface deformation and traces of intercrystalline fracture. Next, it can be seen in Fig.3, that the AE activity and intensity at 100ºC (Fig. 3a) is visibly greater than at room temperature. The fracture type observed in Fig.3b has not essentially changed, and it is still transcrystalline with a distinct contribution of intergranular fracture and traces of surface deformation. However, the behavior of AE has drastically changed at 150ºC (Fig.4a, AE in scale of tenfold smaller). The both, AE activity and intensity are considerably lower than in the previously discussed cases. The fracture is still transcrystalline (Fig.4b), however, cracking along the grain boundaries prevails. The traces of the plastic strain of surface boundary and the single voids and cavities are also observed. From the above analysis we can suppose that the observed changes in the AE behavior may signal the beginning of the transition from one to another kind of fracture. It seems that this supposition is confirmed by the next observations, illustrated in Fig.5. The AE at 200ºC (Fig.5a) is again of higher level, though slightly lower than earlier, whereas the fracture (Fig.5b) is of intercrystalline character with a traced contribution of ductile and fissile surfaces.
The above presented results may be additionally supported by the calculations of the mean values of the local drops of external stress, total sum of AE event counts and AE event energy calculated in the range of strains (or duration of each compression test) corresponding up to maximal value of external load. The mean energy per one AE event was also calculated. These values are presented in Tab. 1. It can be seen that all values achieve minimum just at 150ºC when the discussed fracture transition is beginning.
It is worth to emphasize here that the relation between the AE activity (AE local peaks) and the PL effect (which accompanies local jumps of external stresses), according to [8,11], may be quite well explained in terms of collective and accelerated movement of many dislocations generated in single slip planes by the sources which are alternately active and blocked by solute atoms (Cottrell atmospheres). It is strongly suggested in papers [6,8,10,11], that also the contribution to AE signals may originate from the synchronized of both, internal and surface annihilation of many dislocations.

Mean values → Temperature	Local jumps of external stress [Mp]	Sum of AE event counts	Sum of AE event energy [nJ]	Energy per one AE event [nJ]
RT	0.6	21393	1389000	64.9
100ºC	0.8	26696	1780000	66.7
150ºC	in errors range	1039	61000	58.7
200ºC	0.1	20197	1200000	59.4

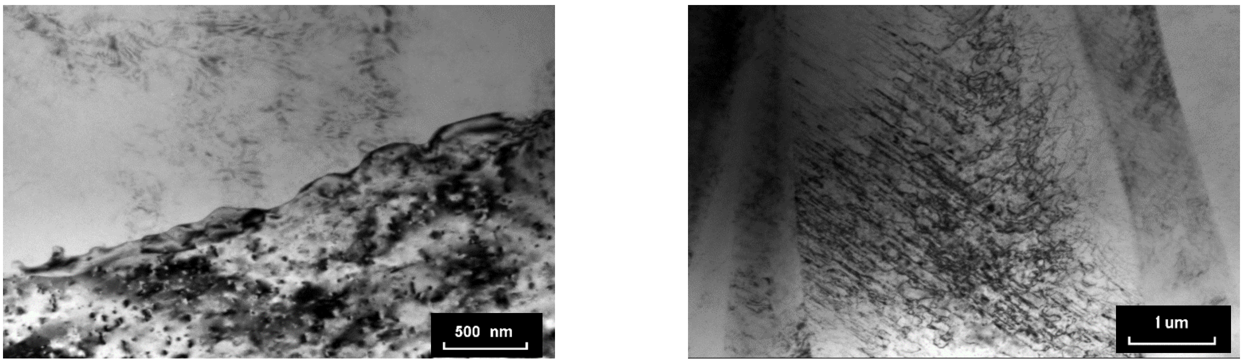
Table 1: The stress and AE parameters for Mg4Li5Al alloys tensile tested at elevated temperatures.

Fig.6 and Fig.7 present corresponding TEM pictures observed after tensile tests of Mg4Li5Al alloy at 150ºC and 200ºC, respectively. It is possible to say generally, that, in the microscale, the changes are not as evident as in the SEM images, which illustrate the fracture changes in the macroscale. The TEM pictures, however, show that the microcracks may be formed due to the stress concentration caused by the condensation of inclusions and/or dislocation assembles at the grain boundaries. We suppose that these dislocation tangles can be the result of the dislocation pile-up formation which, however, undergo scattering at elevated temperature due to e.g. dislocation climb and/or recovery processes. On the other hand, in the context of plastic deformation, the TEM pictures suggest also that twinning may possibly contribute to the plastic instability, whereas, in turn, the microtwins intersection may also play a role in microcracking occurrence.



(a) (b)

Figure 6: The TEM pictures of Mg4Li5Al alloy after tensile test at 150ºC: (a) inclusions, microcracks and dislocation tangles at grain boundaries, (b) microtwins intersection and inclusions at twin boundaries.



(a) (b)

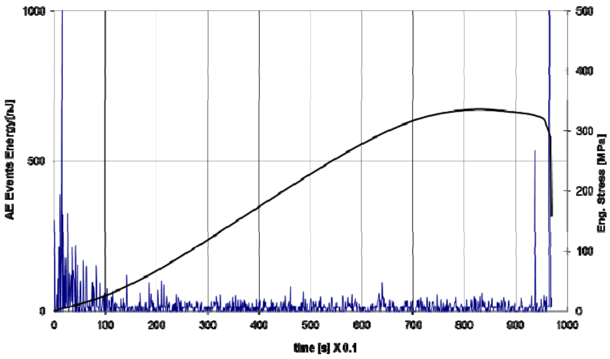
Figure 7: The TEM pictures of Mg4Li5Al alloy after tensile test at 200ºC: (a) inclusions and microcracks at the region of grain boundary, (b) microtwins and inclusions at twin boundaries.

Mg4Li5Al – compression tests

The relation between the plastic instability and the fracture was investigated first for the Mg4Li5Al compressed at room temperature. Fig.8a shows the AE behaviour to the break up of the sample and the corresponding SEM image of its fracture (Fig.8c). The high AE peak is accompanied with the sample disruption. In this case the fracture of cubic sample occurred along the diagonal surface of the cube (the normal of which lies in the ND-ED plane, see Fig.1). This diagonal plane is generally parallel to the plane of maximal stresses leading to the strain localization and shear band formation, that will be more detail discussed further, in the context of optical images presented in Fig.9.

The essence of idea of the next experiment (Fig.8b) was based on a precise compression of another sample up to the moment just before the previous sample had broken. The fracture surface for the SEM observation was obtained in this case by manual breakdown of the sample. The corresponding SEM image of the fracture (Fig.8d) shows, that the length of crack path is visibly shorter than in the case observed in Fig.8a. It means that the final break of the sample is a result of successive growth of the length of crack path.

On the other hand Fig.9a shows a fully developed shear band, which is observed on the side wall (parallel to ND-ED plane) of cubic sample compressed up to adequate external loading. Additionally, Fig.9b shows that the fully formed shear bands may be realized by the development of slip bands and next microshear bands which cut cross many grains, what means that the final fracture in this case is of transcrystalline character.



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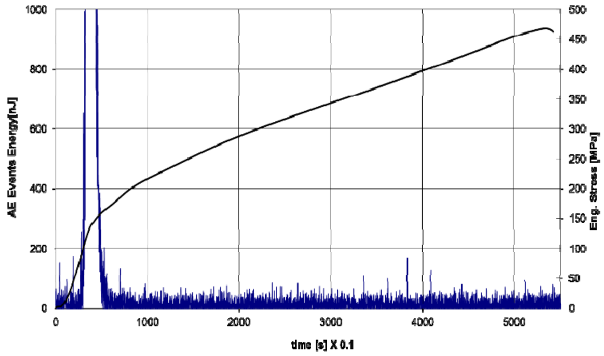

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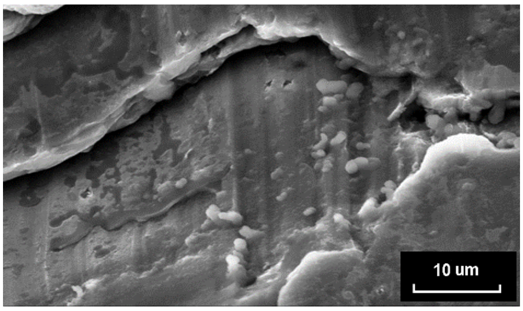

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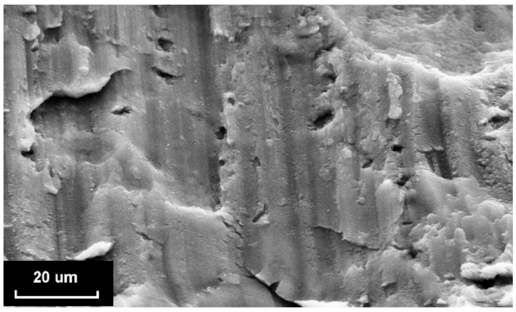
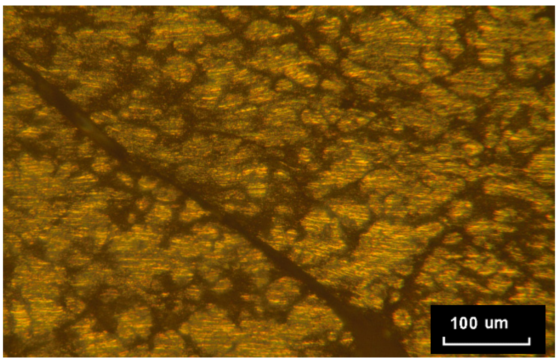
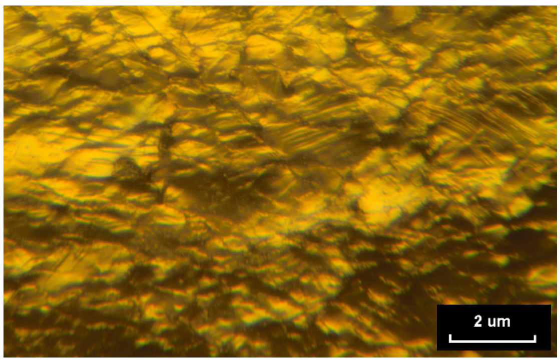


(d)

Figure 8: AE behavior and the course of external load: (a) up to all-out breaking and (b) exactly just before breaking of the Mg4Li5Al sample; (c) and (d) – corresponding SEM images.



(a)



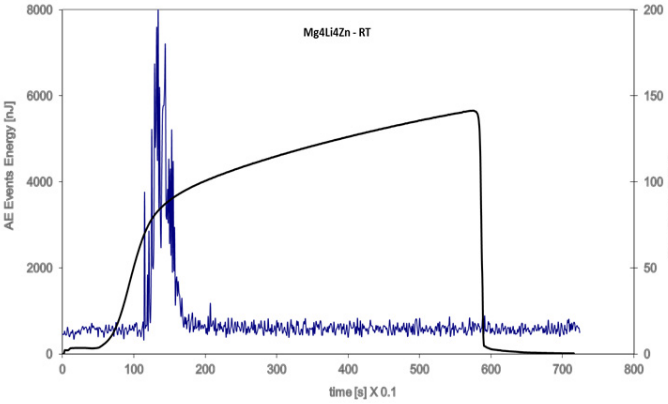
(b)

Figure 9: Optical images illustrating the fully developed shear band (a) and the slip bands and microshear bands crossing over many grains (b).

Mg4Li4Zn alloys – tensile tests

In order to investigate the role of alloy addition, e.g. of zinc instead aluminium, a Mg4Li4Zn alloy was chosen, especially because the plastic instability connected with the PL effect in these alloys is known in literature [19]. The results of the preliminary studies for these alloys are presented in Fig.10. The AE behavior and external stress are shown in Fig.10a for room temperature and in Fig.10b for 200ºC. The SEM images are presented respectively in Fig.10c,d, whereas in Fig.10e,f the SEM pictures are placed as additional observations and they correspond to the fracture at 400ºC.

Unfortunately, when analysing Fig.10a,b it seems that the effect PL in Mg4Li4Zn alloys did not occur or it was not visible. There is by the reason that these alloys were prepared by casting of raw materials, whereas in work [19], where it was clearly visible, those alloys were prepared by extrusion followed with homogenization at 623K. On the other hand, the SEM pictures (Fig.10c,d,e and f) show that the character of fracture did not substantially change and it was mostly of intergranular type. Thus, it is necessary to carry on the research improving the technical aspects of the experiments (better solution for sensor-sample contact at elevated temperatures) and the preparation of the Mg4Li4Zn alloys, following the indications of work [19], so that the plastic instability related to the PL phenomenon could be observed.



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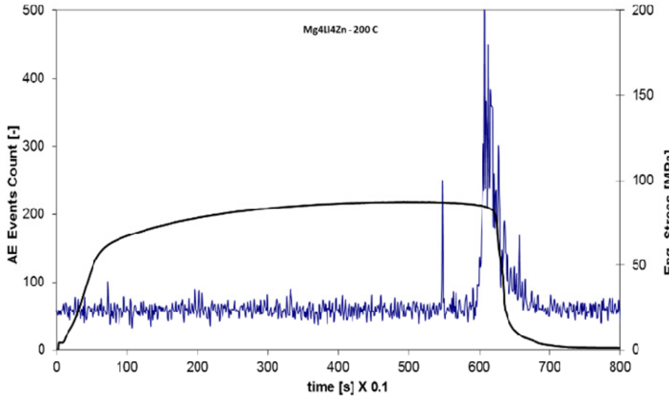

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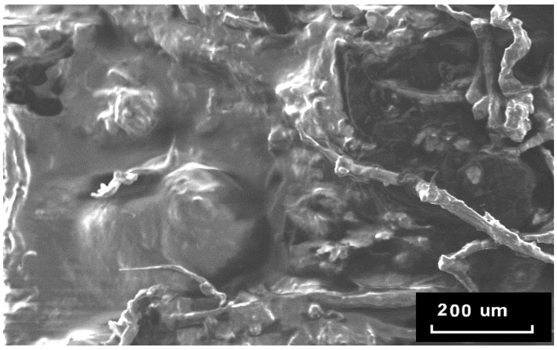

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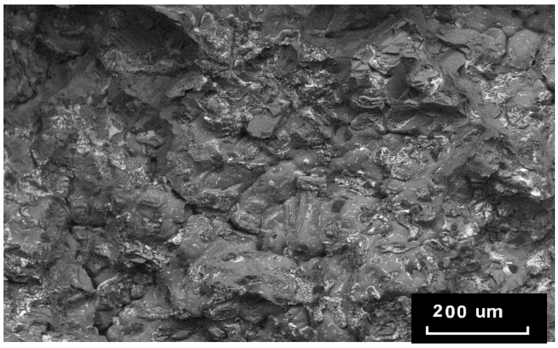

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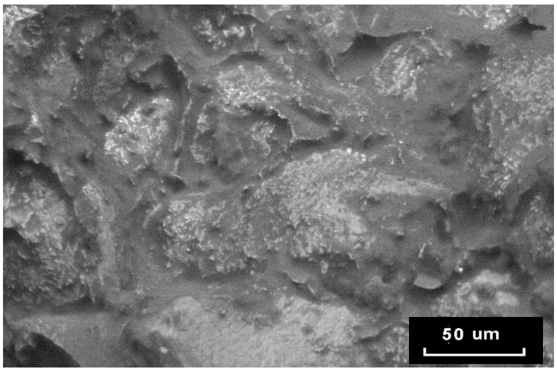

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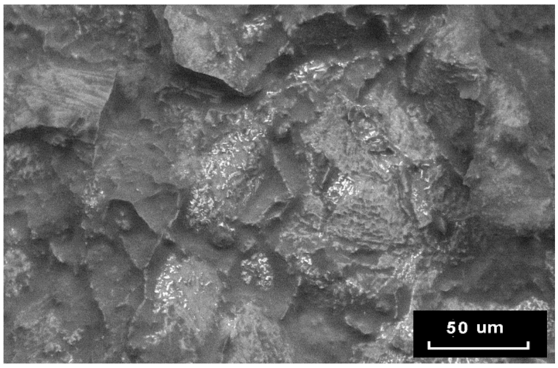

(f)

Figure 10: The AE behavior and the external stresses of tensile tested Mg4Li4Zn alloy: (a) at room temperature and (b) at 200°C; (c, d) corresponding SEM pictures of fracture, (e, f) SEM images of fracture at 400°C.

CONCLUSIONS

The work describes the preliminary research of the influence of plastic instabilities (PL effect, twinning, shear banding) on the fracture of Mg-Li based alloys in tensile and compression tests at elevated temperatures with the unique application of Acoustic Emission technique. It seems that the following conclusions are quite reasonable:

1. The fractures of tensile tested Mg4Li5Al alloys at elevated temperatures are initially of transcrystalline character but the transition to intergranular type starts already from 150°C, while at 200°C the fracture is of intercrystalline character already.

2. The transition from one to another kind of fracture is correlated with the stress and AE parameters achieve minimal values just at the same temperature 150°C.
3. The AE technique can be helpful in further research of the changes in fracture character at elevated and/or higher temperatures.
4. The fracture of compressed Mg4Li5Al alloy at room temperature is related to the strain localization in shear bands, and it is characterized by growth and development of the crack path.
5. In Mg4Li4Zn alloys tensile tested at elevated temperatures, prepared by casting of raw materials, the effect PL does not occur and the plastic flow is rather of stable character, and the fracture, being mostly of intergranular character, as well as the AE behavior are not essentially changed.

ACKNOWLEDGMENTS

The studies were financially supported by Polish National Science Centre (project in competition OPUS 4, grant No 2012/07/B/ST8/03055), and also by the Slovak Academy of Sciences - project VEGA No 2/0186/14.

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