Archives of Current Research International

8(2): 1-13, 2017; Article no.ACRI.34598
ISSN: 2454-7077

Behaviour of Acoustic Emission in Deformation and Microcracking Processes of Mg Alloys Matrix Composites Subjected to Compression Tests

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Authors' contributions

This work was carried out in collaboration between all authors. Author AP designed the study, interpreted the mechanical and AE results and wrote the first draft of the manuscript. Author ZR described basic procedures of AE methods, executed measurements and performed the statistical analysis of the AE results. Author AP executed measurements of mechanical parameters in compression tests. Author PR managed the investigations of ceramic materials and processed results of compression tests. Authors SK and SKJ prepared and fabricated of the Mg Alloys Matrix Composites. Author WO managed the literaturę searches, cooperated in discussion and the analysis of the results of mechanical and acoustic measurements. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/ACRI/2017/34598
Editor(s):
(1) M. A. Elbagermi, Chemistry Department, Misurata University, Libya.
Reviewers:
(1) Md. Salim Kaiser, Bangladesh University of Engineering and Technology, Bangladesh.
(2) Azuddin Mamat, University of Malaya, Malaysia.
(3) Subramaniam Jahanadan, Labuan Matriculation College, Malaysia.
(4) Sunipa Roy, Guru Nanak Institute of Technology, India.
Complete Peer review History: http://www.sciencedomain.org/review-history/19874

Original Research Article

Received 1st June 2017
Accepted 22nd June 2017
Published 5th July 2017

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ABSTRACT

Research results on both mechanical and acoustic emission (AE) behavior of Mg-Li and Mg-Al alloys matrix composites (AMC) reinforced with ceramic δ-Al_2O_3 or carbon fibers subjected to the channel-die compression at room and elevated temperatures are presented in this paper. The AE measurements at room temperature showed that, the effect of anisotropy of the fibres distribution (random planar distribution) with respect to the compression axis appeared in the most investigated composites, whereas the AE activity at 140℃ revealed a two- range character and the rate of AE events at 140℃ was higher than at roo m temperature. These effects are discussed in terms of both the differences in thermal expansion between the fibres and the matrix as well as the weakening of the coherency between the fibres and the matrix leading to stronger debonding effects at 140℃ than at room temperature. The spectral analysis of AE signals was performed with the Windowed Fourier Transform method, which served to plot the spectral density of AE signal as a function of frequency. The alominous and corundum ceramics types were also investigated in order to illustrate the enhanced AE, which was related to the different crack paths in the final stages of the sample degradation. The results were also discussed on the basis of SEM images, including the in-situ observations of microcracking as well as the dislocation strain mechanisms and microcracking ones during the channel-die compression of the Mg-Li-Al AMC.

Keywords: Composites; fibres microcracking; acoustic emission; strain mechanisms; dislocations.

1. INTRODUCTION

Composites based on Mg-Li-Al alloys reinforced with ceramic δ-Al_2O_3 fibres promote light and fairly strong construction materials in the automotive, aircraft and aerospace industries. Mg-Li alloys can appear in the form of three different phases. The hexagonal α phase of *hcp* structure occurs in the concentration range of Li up to 4 wt.%, whereas the alloys containing more than 12 wt.% consist of the β phase of *bcc* structure. The mechanical properties of the α phase are worse than these of the β phase compensated however by considerably higher plasticity, very good machine and weld abilities [1]. The alloys with Li content from 4 wt.% up to 12 wt.% occur as a mixture of the α+β phases. The alloying additions, e.g. in the amount of 3 to 5% Al, slightly increase the density of the composites, thus considerably improving their strength.

The acoustic emission (AE) method is helpful in many aspects of physical phenomena associated with deformation mechanisms, in comparison with the alloys and composites subjected to compression tests, including processing with intensive strain methods [2-5]. Systematically upgraded software (see section 2.1) allows the spectral analysis of recorded AE signals as far as the determination of spectrum characteristics and the distribution function of rate and energy of AE are considered. Some of the first major tests of the AE phenomenon, which occurs during plastic deformation of metals, were carried out in the second half of the sixties by Fisher and Lally [6]. The results of such a research work so far are the starting and comparative point to examine the relationships of AE descriptors as function of deformation degree. They put forward an idea [6] that the AE activity, related to the yield point, is the result of a rapid and collective motion of large number of dislocations generated in Frank-Read sources.

The AE method has been applied to the investigations of poly- and single crystals of fcc metals and alloys by the authors of the present paper for many years [7-12]. The investigations concentrated on explaining the correlations between AE descriptors and the mechanisms of deformation of the materials subjected to the channel-die compression tests. The results are mainly related with the application of the AE method to the investigations of plastic flow instability connected with twinning processes and Portevin – Le Chatelier effect as well as the formation of shear bands [8-11]. In consequence, the following thesis was put forward: 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 on the free surface of the deformed material.

The purpose of the present work was the documentation and the explanation of the current and previously [2-4,12-15] observed correlations between the behaviour of AE and the mechanisms of plastic deformation of alloys and AMC. The possible occurrence of superplasticity phenomenon in such materials, connected with the AE decrease due to the microstructure refinement [3,13] after processing by intensive strain methods, was also observed. However, the main objective is to confirm that the increase of strength and plasticity, corresponding to the evident AE decrease, is also observed in the Mg2Al AMC reinforced with carbon fibres as well as in Mg8Li AMC reinforced with ceramic fibres.

The investigations of Mg8Li and Mg8Li3Al AMC were performed also at the elevated temperature of 140℃. They were carried out to study the possible anisotropy of the fibre distribution with respect to the compression direction. Moreover, since the AE analyser of new generation was used for these investigations, the spectral density of AE signal as function of frequency was plotted applying the Windowed Fourier Transform analysis of the registered AE signals.

The results have been discussed on the basis of these ones obtained so far [2-4], which are related to the dislocation mechanisms of plastic flow and the mechanisms of fibre microcracking in the AMC materials. The concepts of collective acceleration and surface annihilation of dislocations as the main reasons for AE in metals reported e.g. in [4,5,9,16] have been also discussed. Moreover, some recent our publications [17-20] are related also to above mentioned questions, though especially they are referred to the problems of the influence of plastic instabilities on the fracture mechanisms in deformed metallic materials.

The studies of AE in the Mg-Li alloys subjected to tests of channel-die compression show that the AE effects can be quite satisfactorily explained on the basis of collective and synchronized behaviour of many groups of dislocations generally associated with the acceleration and annihilation of dislocations. For example, the increase of AE activity in the Mg4Li alloy in comparison with the AE in pure Mg, as it is reported in [4], is the result of the activity of new slip systems due to the presence of lithium which reduces the c/a ratio in the hexagonal cell unit, and it facilitates, in this way, the activation of prismatic and pyramidal slip systems. Such an explanation is consistent with the interpretation of a significant decrease of the rate of AE events in the Mg 12Li alloy, where the strong interaction of lithium atoms with dislocations, due to the extremely high diffusivity of Li in the bcc structure of β phase, clearly impedes both, the collective behaviour and synchronized surface annihilation of dislocations, decreasing their contributions to the registered AE signals [4]. The downward trend of AE was also observed for the two-phase Mg8Li alloys, in which the AE activity, compared with that of single α phase alloys, was weakened due to the presence of the β phase, whose AE activity was very low [4]. In this context, the topics related to the application of AE methods to channel-die compression tests of alloys and composites including these of ultrafine microstructure obtained using the methods of intensive deformation are unique and innovative. A significant result of the present paper consists in the conclusion, that the AE decrease in the reinforced AMC is in good accordance with the AE decrease observed in the alloys processed with intensive strain methods [3,13].

2. EXPERIMENTAL AND THEORETICAL BACKGROUND

2.1 Compression Tests

Composites based on Mg-Li and Mg-Li-Al alloys were produced of a fibrous skeleton of commercial Saffil® - subjected to infiltration under pressure in bath of liquid alloy in a laboratory autoclave. The volume fraction of fibres in the skeleton amounted to 20%, while their weight contribution in the composite was ca. 10%. The composites revealed a planar random distribution of ceramic fibres, whose mean length ranged from 100 to 500 μm, and their mean size of diameter was 3 to 4 μm. Samples of the alloys and composites for compression tests had the cubic shape of side 10 mm.

The compression tests were carried out using INSTRON-3382 tensile testing machine, additionally equipped with a specially constructed channel-die which ensured 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 velocity of the testing machine traverse was 0.05 mm/min. The overall look on the testing arrangement and the instrumental details are presented in Fig. 1.

The AE analyzer was equipped with an additional measuring channel that allows simultaneous recording of the forces that load the sample and the AE descriptors: AE event duration, its maximum signal amplitude and energy. In this way, the map of distribution of the population of registered events in function of their energy can be obtained. The contact between the sensor and the sample is maintained by means of a steel rod used as a washer in the channel-die. The measurements at 140℃ were carried out using a specially profiled quartz wave-guide, placed between the heated sample and the AE sensor. In order to eliminate the undesired effects of friction against the channel walls, each sample was covered with Teflon foil.

2.2 AE Technique – Procedures

The measuring system of AE parameters is functionally coupled with the force measuring system, and they are described in more details in Refs [12,21]. The idea of the AE signal measurements consists in the following: an acoustic wave generated in the sample during its deformation stimulates vibrations of the attached piezoelectric sensor. At the output of the sensor, these vibrations are transformed into a voltage signal, which is amplified using an amplifier of high sensitivity to electrical loads. The amplified AE voltage signal is then passed to the signal processing system, at whose output the impulse in the form of an envelope of the surface proportional to the input signal energy appears. The device has also a discriminator of voltage containing a comparator with a threshold voltage regulator to suppress undesired environmental noises accompanying the measured system. As a result, acoustic signals are transmitted to the voltage discriminator and each of them, which exceeds its threshold level, is counted only once, corresponding to a single registered AE event.

The effective value of the noise at the input of preamplifier is about 20-30 microvolts, depending on the selected frequency band. In the course of the signal processing, this value is about four times lower due to the use of filtration in the frequency domain. The active high-culvert filter of the 8^{th} order of cut-off frequency 5 kHz is connected to the preamplifier. Additionally, the filter of cut-off frequency 20 kHz of the fourth order can be switched on. Thus, the vibroacoustic background signals which do not originate from the sample loading are eliminated from further processing. Then the signal is fed to the low-pass filter of cut-off frequency 1000 kHz.

The amplification of the signal at the output device is 60 dB. The block diagram presenting the system for recording the AE signal is presented in Fig. 2. AE signal processing unit was realized with application of the 9812 ADLINK type card hosted in a PC computer.

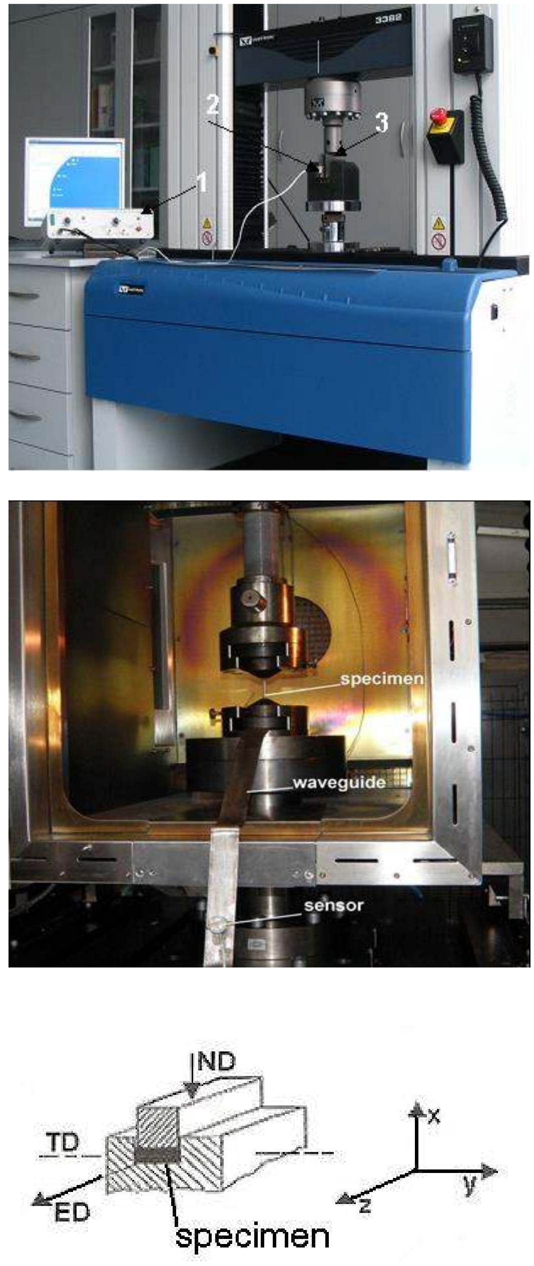

Fig. 1. The overall (top), detailed (middle) and schematic (bottom) look on the testing arrangement; (1) - Acoustic Emission analyser (2) - sample under test (3) - channel – die

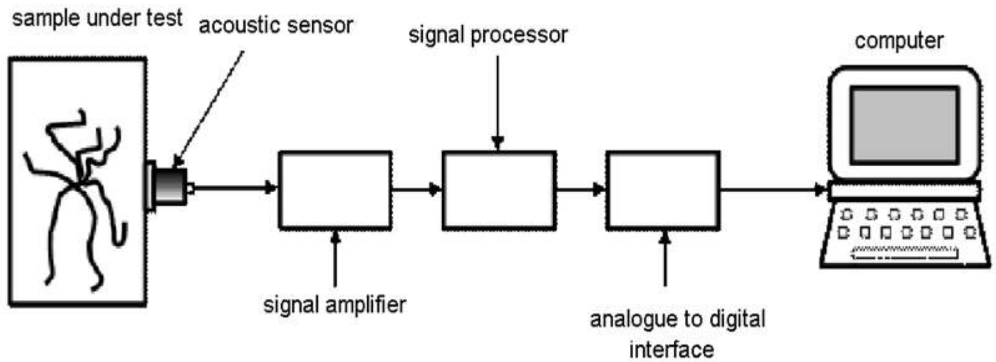

Fig. 2. Block diagram of the arrangement for processing and recording of the AE signal

2.3 AE – Theoretical

The differences in acoustic emission signal generated by different sources in the examined object can be analyzed through changes of its spectral characteristics. Thanks to the suitable software, the registered indexes of start and end of AE event in a software registry table can be used to determine the duration Δt of the AE event. The energy E of the AE event is then calculated from an approximate formula:

$$E = 0.5v^2_{max}\,\Delta t, \tag{1}$$

where v_{max} is the maximum value of AE signal in the course of the event, and Δt is its duration.

Continuous acoustic emission signal $v(t)$ in a finite time interval can be plotted as function of its spectral characteristics $A(\omega)$, where $\omega=2\pi f$ is the pulsation and f is the frequency. Assuming the infinite integrability, function $v(t)$ is linearly transformed into the spectral density function, $A(\omega)$, in accordance with the following well-known formula for the Fourier transform:

$$v(t) = \frac{1}{\pi}\int_0^\infty A(\omega)\exp(j\omega)\,d\omega. \tag{2}$$

Consequently, a procedure has been developed for the determination of spectral density function, $A(\omega)$, for subsequent segments of the discrete set of samples of the AE signal along with a graphic presentation of the results of the AE plots in the form of maps, i.e. spectrograms using the method of Windowed Fourier Transform.

3. RESULTS AND DISCUSSION

3.1 AE in Mg-Li AMC Reinforced with Ceramic Fibres

The use of AE method in the study of strength properties of composite materials, both before and after the operations of intensive deformation, is much less documented than in the case of metals and alloys. The exemplary results in Fig. 3 illustrate that the level of AE intensity in the composites based on pure Mg was significantly reduced in comparison with that in pure Mg, as was shown in Ref. [4]. Moreover, Fig. 4 presents light microscope images of microstructure of the composite subjected to tests, whose AE behavior is presented in Fig. 3. Contrary to the AE in Mg [4], the AE activity in the composites continues almost all the time during the compression test, which means that the individual AE peaks are generated as a result of microcracking processes. The decrease of the AE level in composites is caused by the presence of ceramic δAl_2O_3 fibres, which significantly limit the possibility of collective behaviour of dislocations. The effect of the anisotropy of fibres along the compression axis ND (an apparent difference in the level of AE intensity), which is caused by a different localization of plain distribution of fibres, is significant. This comes from the fact that the fibres parallel to the ND compression axis intersect active slip planes, whereas a half of fibers perpendicular to the ND is statistically parallel to these planes.

Fig. 5 shows the acoustic emission in Mg8Li/δ composites, in which the fibres are perpendicular (Fig. 5a) or parallel (Fig. 5b) to the compression axis ND. It can be seen, that in the case of parallel fibres, the AE course against the background of the main broad-band maximum of the AE event rates, connected with pure dislocation processes is more violent, much more jerky and is characterized by a distinctly longer period of activity (AE peaks are observed after the main maximum almost to the end of the compression test) in comparison with its course in the composite with fibres perpendicular to the compression direction. Such behaviour of AE is typical for the effect of fibre anisotropy, and can be attributed to the fact that, in the case of perpendicular fibres, a considerable number of fibres is nearly parallel to the active slip systems connected with the direction of the maximal shear stresses. In this way the number of effective shears of fibres by dislocations, leading to the formation of microcracks generating jump-like AE events, is statistically lower than that in the case of the composite with parallel fibres. In fact, such microcracking of fibres may occur e.g. under the influence of very high concentration of internal stresses at the front of dislocation pile-ups, which exceed hundred times the external stresses.

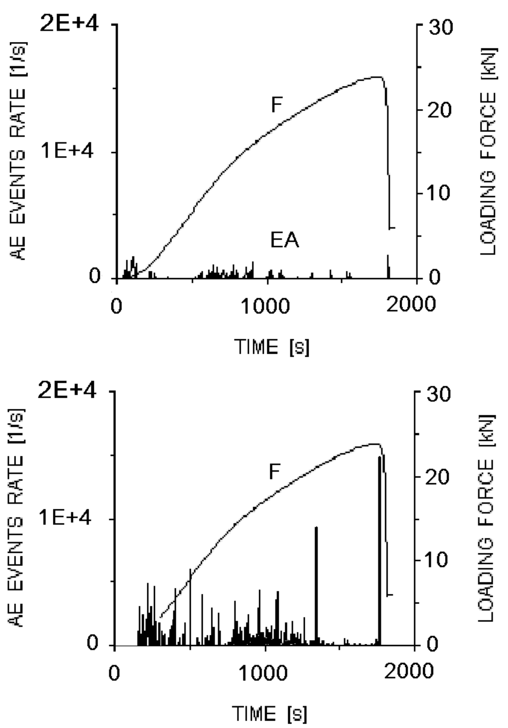

Fig. 3. AE in composites based on pure magnesium reinforced with ceramic fibers compressed at ambient temperature: for perpendicular (top) and for parallel fibers to ND (bottom)

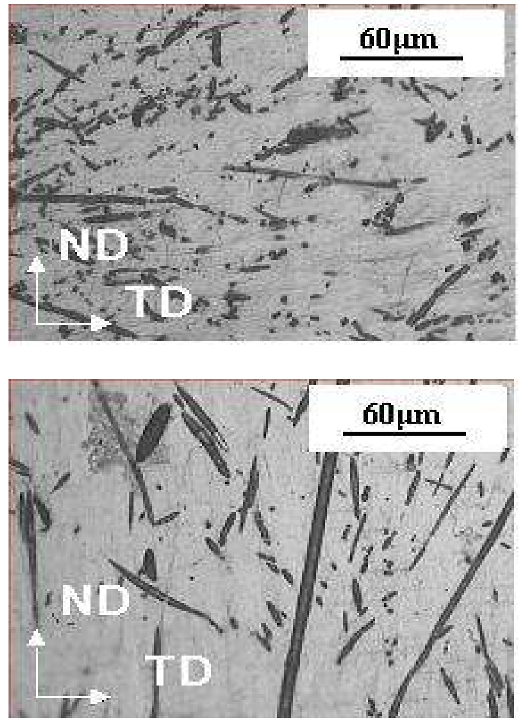

Fig. 4. Light microscope picture of microstructure of Mg based composites

On the other hand, the presence of fibres, as obstacles for the movement of dislocations, reduces the tendency to collective behaviour and both internal and surface annihilation of many dislocations, responsible for the contribution to the registered AE signals [9,16].

Fig. 6 presents the behaviour of AE in Mg8Li3Al/δ AMC with lower volume fractions of fibres compared with that presented in Fig. 5. Although a somewhat greater AE activity in the case of parallel fibres can be noticed (Fig. 6b), this is not a satisfactory confirmation of the phenomenon of anisotropy. Instead, very high level of AE in comparison with that at room temperature (Fig. 5) is observed after about 1500s, apart from the second range of AE activity. Such unexpected behaviour of AE at 140℃, observed in Mg8Li3Al/ δ AMC, allows the assumption, that it is due to the processes which are so dominating that the anisotropy effect is almost invisible. In fact, the second range of AE activity would be connected with the enhanced microcracking of fibres at 140℃, though the initial fibre distribution does not change with the increasing temperature. This suggestion has been strongly confirmed by microstructures shown in Fig. 7 obtained using a SEM. These images have revealed that the microcracks of

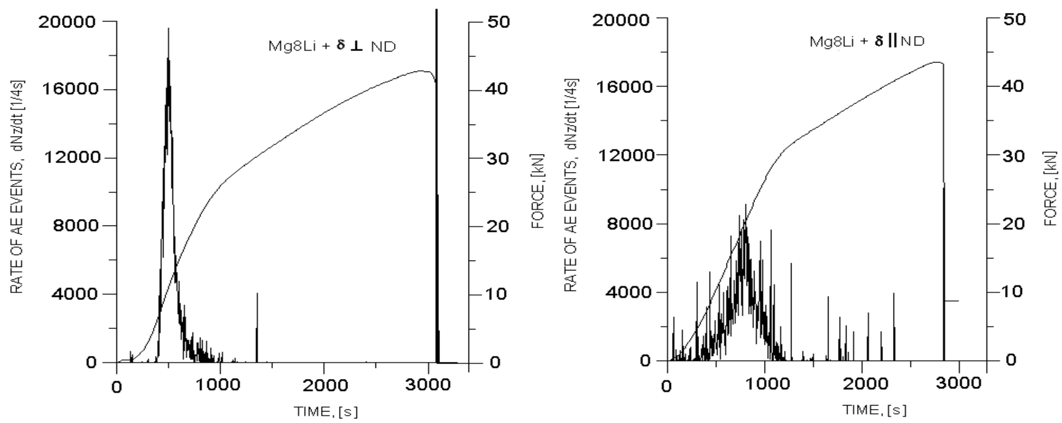

Fig. 5. Effect of anisotropy of the distribution of fibers with respect to compression direction ND in Mg8Li/δ composites subjected to channel-die compression at ambient temperature: – perpendicular fibers (at the left), – fibers parallel to ND (at the right)

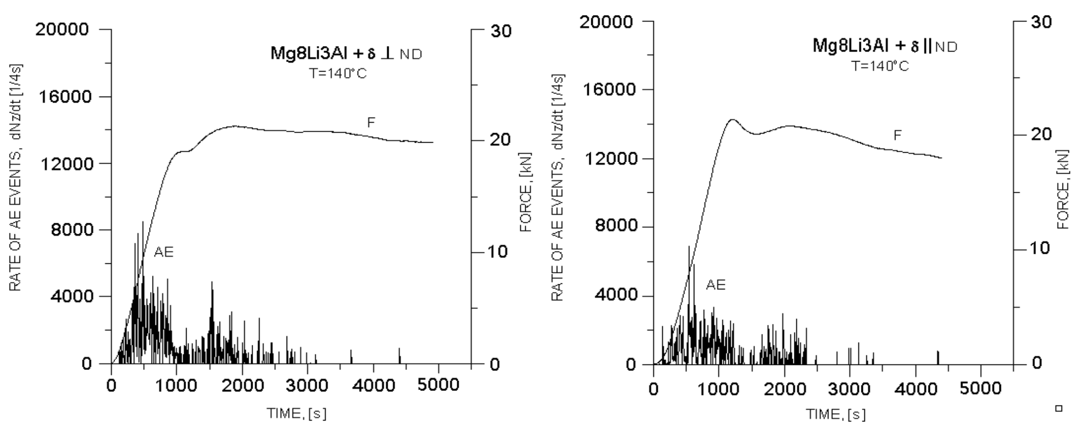

Fig. 6. AE and the external force F in Mg8Li3Al/δ AMC compressed at elevated temperature (140℃): – perpendicular fibers (at the left), – fi bers parallel to ND (at the right)

fibres may occur due to the following effects: by dislocation shear, e.g. as a result of relaxation of high internal stresses formed in the front of a dislocation pile-up, by decohesion of fibres from the matrix, so-called debonding, by the processes occurring along the phase boundaries or due to the differences in the coefficients of thermal expansion of the ceramic fibres and the metallic matrix. The question remains, however, which of these processes are dominant, though the results presented here suggest that the fibre microcracking seems to be the most efficient.

3.2 In situ SEM Investigation

The processes of ceramic fibre microcracking were observed also using *in situ* technique. In-situ SEM tensile experiments of Mg-Li matrix composites were carried out using a specially adapted SEM apparatus equipped with a tensile device [22]. A flat composite specimen, metal ographically prepared, (thickness of 2 mm,) was inserted into the SEM chamber to be tensile strained parallel to the fibre array planes with simultaneous SEM observation of the gauge section (length of 10 mm) of the tested specimen. Gradual increase of load was stopped several times to take SEM micrographs. The goal of in- situ SEM observations was to monitor the failure evolution of individual fibers and/or the fiber/matrix decohesion as a function of the load applied. It was found that Mg-Li composites failed usually via decohesion of fibers oriented transversely to the tensile axis without exhibiting any fiber pull-out. Multiple cross-cracking of individual fibres into shorter segments occurred during the elongation of the composite. It was observed that fiber cross-breakage did not propagate into adjacent matrix region and it did not initiate the overall composite failure. This may be attributed to the extraordinary Mg-Li matrix plasticity absorbing the fracture energy near the crack tip. The absence of fiber pull-out and multiple fiber cross-breakage indicated a strong interfacial bond promoting the effective stress transfer from the matrix to the fiber under external loading. According to simple shear lag model [23], only the fibers longer than critical fiber length l_C can be stressed to their failure strength. The fiber fragments of sub- critical length ($L < l_C$) practically do not contribute to overall composite strength and their previously carried load is redistributed through the matrix to neighboring fibers. This implies that multiple fiber cross-breakage is stopped if the segments are shorter than l_C. The results of these observations are presented in Fig. 8 as an example of sequence of fiber fracturing and/or failure evolution in tensile strained Mg8Li/δ AMC.

3.3 AE in Ceramic Materials

Large cracks in the matrix were the principal effect of ceramic material degradation. In the case of aluminous ceramics, cracks were often branched and curved. Fig. 9 (top) shows critical cracks in the central part of the sample loaded up by compression in channel-die to about 400 MPa. The smaller cracks joined and formed larger and more branched cracks. Their propagation was facilitated by destroyed structure elements, in particular grains of quartz. Large cracks were subjected to strong branching inside the sample. Big cascades of splitting cracks were also observed. The top of Fig. 9 shows light microscope picture of aluminous ceramics microstructure along with the typical course of EA as a function of load for this type of porcelain (below). A characteristic strong increase of AE activity in the critical (i.e. next to failure) load range is seen in the figure.

In the case of corundum ceramic, the most distinctive effect of the critical stage of the structure destruction was the propagation of large cracks, starting from the central part of the samples (Fig. 10). Large cracks were at first of intergranular type (intercrystalline cracking), but at the stage of rapid crack growth, they also passed through the grains, especially these of larger diameter (transcrystalline cracking). The growth of these cracks was the source of a series of strong AE signals (Fig. 10).

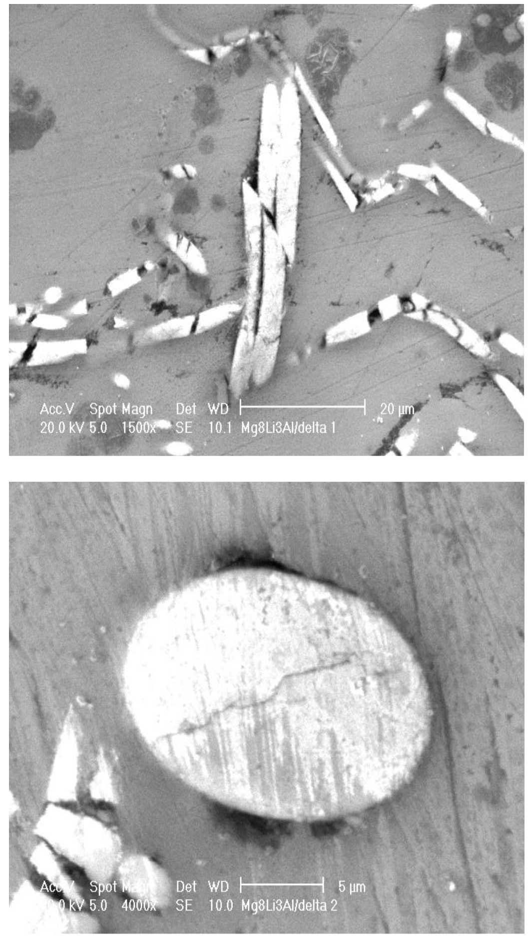

Fig. 7. Typical SEM pictures of microstructures after compression of the Mg8Li3Al/δ AMC composite

3.4 AE in Mg-Al AMC Reinforced with Carbon Fibers

Fig. 11 shows the results of compression tests and the AE behaviour in the Mg2Al AMC reinforced with a low concentration of fibres (about 5.8 vol.%). Carbon fibres were perpendicular (Fig. 11) and parallel (Fig. 12) to the ND axis. The initial microstructures of these composites are presented at top sections in figures mentioned above. A brief analysis of these results shows that the AE in the composites with parallel fibres was noticeably higher than in the composites with perpendicular fibres. Therefore, this observation indicated that the effect of anisotropy of fibre distribution with respect to the compressive axis, observed in the case of composite with ceramic fibres, took place also in the case of a composite reinforced with carbon fibres, and next it suggested that the process of carbon fibre microcracking occured also in a similar way to the process of microcracking of the δ-Al_2O_3 fibres in Mg8Li/δ composite presented in Figs. 4 and 8. On the other hand, that suggestion was confirmed by Fig. 13, which shows additionally, that also the carbon fibre microcracking occurred in the same way as in the case of ceramic fibre reinforcement illustrated in Fig. 8.

3.5 Spectral Analysis of AE Signals

The Windowed Fourier Transform, offered by the AE analyser of new generation, enables the construction of spectral characteristics of AE signal recorded during the process of fibre breaking. It can be stated that the different orientation of strained and damaged fibres results in different signal spectra of the registered AE signal. It is expected that more and more information about the mechanisms of the processes which generate the AE can be obtained applying this method.

Averaged spectral characteristics carried out on the sets of six fragments of AE signal of 1s duration taken from the region of the greatest AE activity of Mg8Li3Al/δ AMC are presented in Fig. 14. Regarding the image of presented curves, it is necessary to emphasize, that a low frequency part of the signal spectrum, i.e. 0 to 8 kHz, was affected by the operation of the Instron drive. The AE activity coming from the process of breaking fibres was situated in the region of frequencies between 8 and 16 kHz. The process of breaking the fibres parallel to the ND compression direction caused the emission of AE signals of the greatest amplitude and a relatively broad bandwidth.

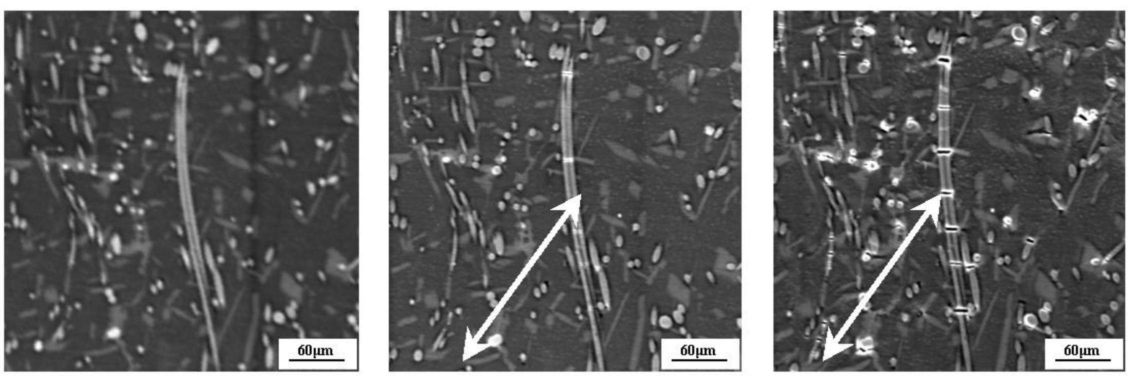

Fig. 8. An example of sequence of SEM pictures of fiber fracturing and/or failure evolution in tensile strained Mg8Li/δ AMC. At the left side: non-strained sample before test. Image in center and at the right side: results after tensile test, the arrows point to the tensile direction

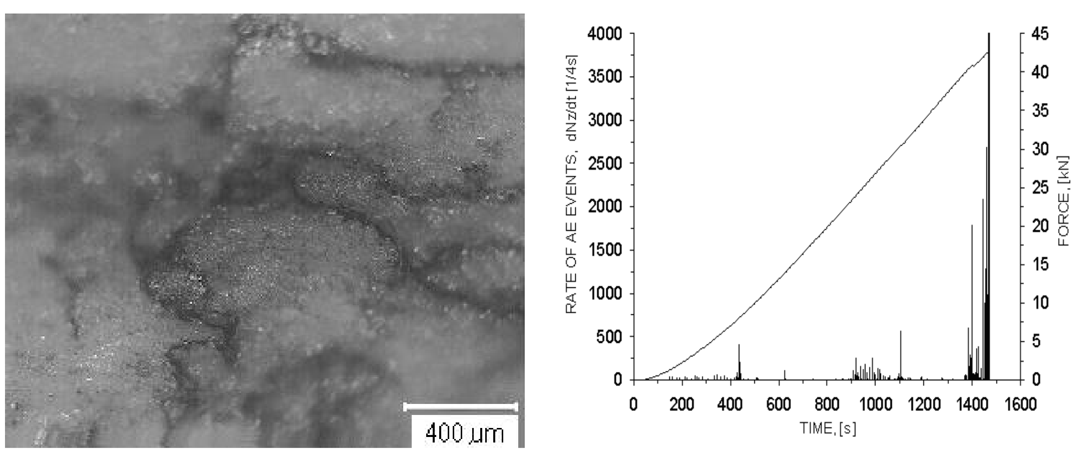

Fig. 9. Light microscope picture of microstructure of porcelain of C 120 type (at the left) together with a typical course of AE at critical stage of material degradation (at the right)

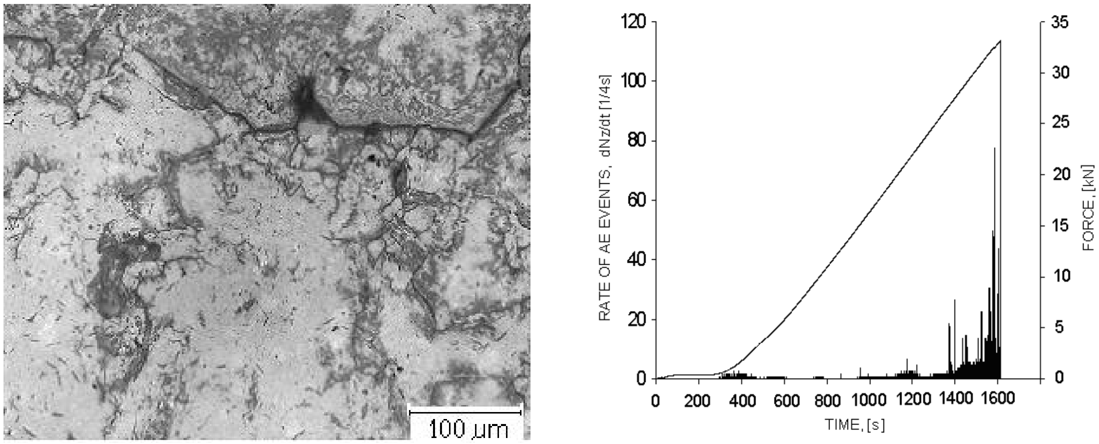

Fig. 10. Light microscope picture of microstructure of corundum ceramic (at the left) and typical course of AE (at the right) in the critical stage of degradation of the material

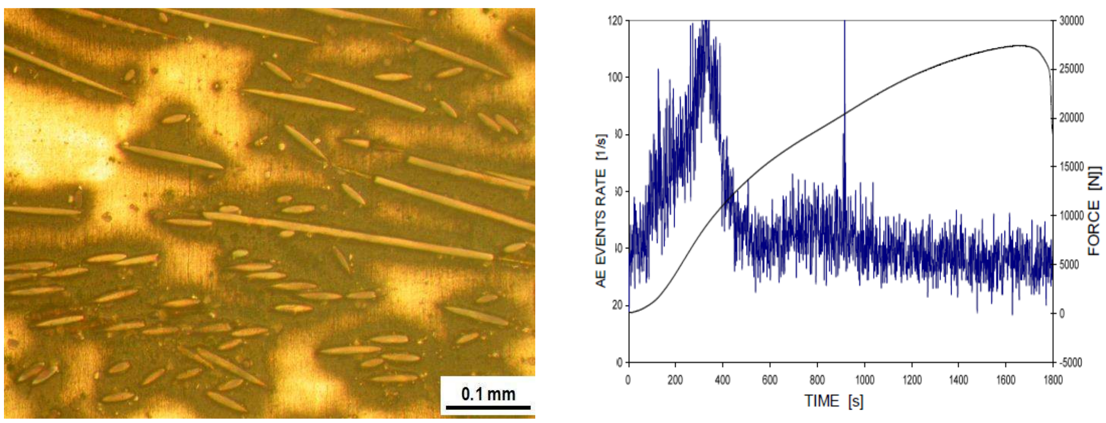

Fig. 11. The initial microstructures (at the left), and AE courses and compression force in the AMC Mg2Al reinforced with carbon fibers of low density - fibers perpendicular to the ND axis (at the right)

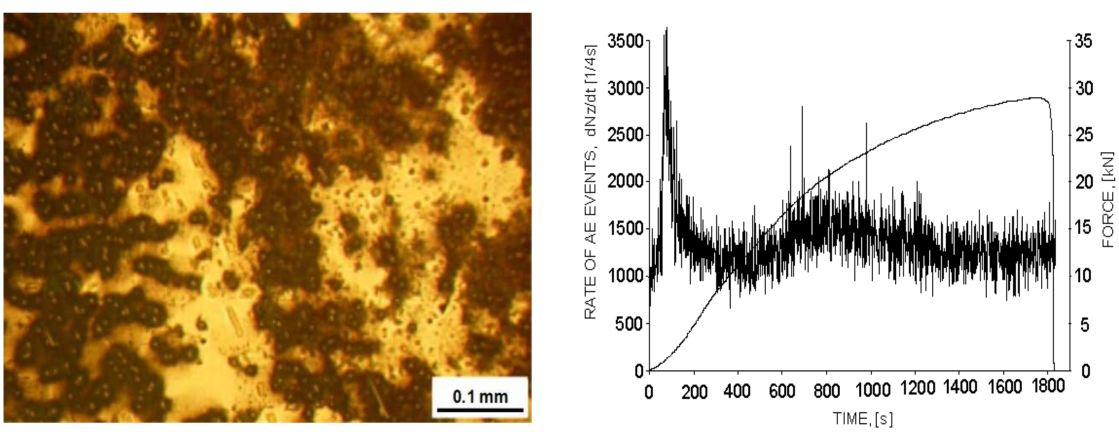

Fig. 12. The initial microstructures (at the left), and AE courses and compression force in the AMC Mg2Al reinforced with carbon fibers of low density - fibers parallel to the ND axis (at the right)

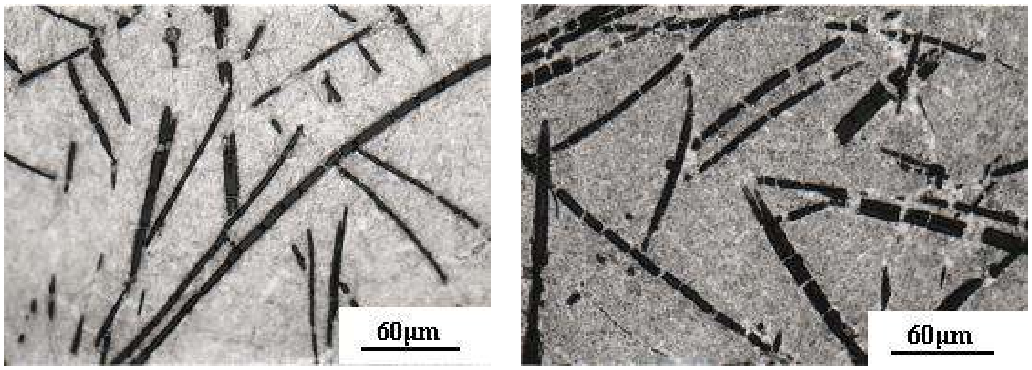

Fig. 13. Exemplary illustrations of light microscope pictures of microstructure microcracking process of carbon fibers due to small (at the left) and large plastic deformation (at the right) [23]

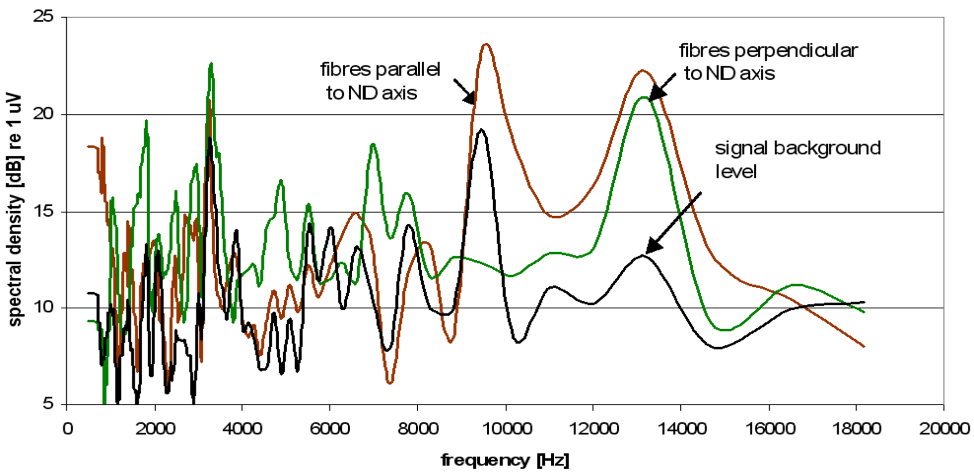

Fig. 14. Averaged spectral characteristics carried out on the sets of six fragments of AE signal of 1s duration taken from the region of the greatest AE activity of Mg8Li3Al/δ AMC

4. SUMMARY AND CONCLUSIONS

Summing up, the authors wish to express their hope, that the most important results presented in the paper will contribute to the satisfactory solution of the problems of monitoring and the identification of mechanisms of plastic deformation with the AE method and, in the case of nano crystalline materials, to the introductory explanation of the possible processes related to the mechanisms of super plasticity.

Finally, the following conclusions could be drawn:

- Acoustic emission in Mg-Li AMC occured during the whole range of compressive test, and it was mainly due to the micro cracking processes of ceramic fibres.
- AE behaviour in Mg8Li AMC reinforced with ceramic fibres channel-die compressed at room temperature revealed the occurrence of the effect of anisotropy with respect to the compression axis.
- The influence of anisotropy of fibre distribution on the AE activity was also observed in Mg2Al AMC reinforced with carbon fibres compressed at room temperature.
- The increase of AE activity in Mg8Li3Al AMC compressed at 140℃ in comparison to the results obtained at room temperature and two-range character of the AE activity at high temperature may be related to the enhanced fibre microcracking due to the shear by dislocation pile-ups, the debonding processes and the difference in thermal coefficients of fibres and matrix.
- The contribution to the observed AE, related with the presence of fibres, may

be discussed in terms of the concept of collective behavior of many dislocations described wider in [3,13].

- The AE method is very useful for the identification of mechanisms and *on line* registering the intensity changes of various microcracking processes including these observed in porcelain and corundum ceramics.

ACKNOWLEDGEMENTS

The research studies were financially supported by the research projects of the Polish Ministry of Science and Higher Education No N507 056 31/128 and No N N507 598038 as well as by the research project No 2/0196/11 of the Grant Agency VEGA of the Slovak Republic. This work was sponsored partially also by the new project of the Polish National Center of Science (NCN) – grant No 2012/07/B/ST8/03055.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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The peer review history for this paper can be accessed here:
http://sciencedomain.org/review-history/19874