Micro cracking of Ceramic and Carbon and Fibres Acoustic Emission in Channel-Die Compressed Mg-Li and Mg-Al Alloys Matrix Composites

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ABSTRACT. *In this paper there are presented the results of the investigation of 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. The results of AE measurements at room temperature show that in the most investigated composites there appears the effect of anisotropy of the fibers distribution (planar random distribution) with respect to the compression axis, whereas the AE activity at 140^0 C revealed a two-range character and that the level of the rate of AE events is higher than at room temperature. These effects are discussed in terms of both the differences in thermal expansion between the fibers and the matrix as well as the weakening of the coherency between the fibers and the matrix leading to stronger debonding effects at 140^0 C than in the room temperature. The spectral analysis of AE signals was performed with the Windowed Fourier Transform method, what served to plot the spectral density of AE signal as a function of frequency. The ceramics of corundum and 130 porcelain types were also investigated in order to illustrate the enhanced AE which is related with the different crack paths in the final stages of the sample degradation. The results are also discussed on the basis of SEM images including these in-situ observations of micro cracking fibers as well as in the context of the dislocation strain mechanisms and micro cracking ones during the channel-die compression of the Mg-Li-Al AMC.*

INTRODUCTION

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

The performed investigations were intended to determine the relations between the AE and the strain mechanisms in Mg8Li and Mg8Li3Al AMC subjected to channel-die compression at room temperature and at 140^{o}C. The latter investigations were carried out to study the possible anisotropy of the fibre distribution with respect to the compression direction. Moreover, since an AE analyser of a new generation has been applied in these investigations and on the basis of the qualitatively new results the spectral density of AE signal as a function of frequency have been plotted applying the Windowed Fourier Transform analysis of registered AE signals.

The results have been discussed on the basis of these obtained so far [2-4] which are related to the dislocation mechanisms of plastic flow and the mechanisms of micro cracking in the AMC materials. The conceptions of collective acceleration and surface annihilation of dislocations as the main reasons for AE in metals reported e.g. in [4-7] have been also considered.

EXPERIMENTAL

Composites based on Mg-Li and Mg-Li-Al alloys were prepared in cooperation with the Institute of Materials and Machine Mechanics of the Slovak Academy of Sciences, Bratislava. They were produced from a fibrous skeleton of commercial Saffil® - subjected to infiltration under pressure in a bath of liquid alloy in a laboratory autoclave. The volume fraction of in the skeleton amounted to 20%, and their contribution in the composite was 10%. The obtained composites revealed a planar random distribution of the ceramic phases, whose mean length oscillated from 100 to 500μm, and the mean size of the diameter was 3÷4 μm. Samples of the alloys and composites intended for compression tests had the shape of cubes of side 10 mm.

The compression tests were carried out using the 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 in the direction perpendicular to the channel walls (transverse direction – TD) the deformation was impossible. The velocity of the traverse of the testing machine was 0.05 mm/min. Simultaneously with the registration of the external force F, the AE parameters, mainly *AE events rate and RMS* were measured. A broad-band piezoelectric sensor enabled the registration of acoustic pulses in the frequency range from 10 kHz to 1 MHz. The contact between the sensor with the sample was maintained by means of a steel rod used as a washer in the channel-die.

Measurements at 140^{o}C were carried out using a specially profiled quartz wave-guide. The total amplification of the acoustic signals was 80 dB, and the corresponding optimal threshold voltage was 1.19V. In order to eliminate the undesired effects of friction against the channel walls each sample was covered with Teflon foil. The application of the new generation AE analyser enabled for the registration of AE source signal for its later processing to determine the AE events intensity or to create a signal spectral characteristics in limited bandwidth of 1 - 20 kHz.

Additionally, the micro structural observations of AMC reinforced with ceramic or carbon as well as ceramic materials were carried out by means of a light or scanning microscope on samples before and/or after deformation.

RESULTS AND DISCUSSION

AE in Mg-Li AMC reinforced with ceramic

Figure 1 shows the behaviour of AE in Mg8Li/δ composites in which the are situated perpendicular (Fig.1a) and parallel (Fig.1b) to the compression axis ND. It can be seen that in the case of parallel the AE course against the background of the main, broad-band maximum of the AE events rate, connected with purely 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 the course in a composite with perpendicular to the compression direction. Such behaviour of AE is typical for the effect of anisotropy and it can be attributed to the fact that in the case of perpendicular a considerable number of the 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 by dislocations, leading to the formation of micro cracks generating jump-like AE events, is statistically lower than in the case of a composite with situated in parallel. In fact, such micro cracking of may occur e.g. under the influence of very high concentration of internal stresses at the front of dislocation pile-ups, even some hundred times exceeding the external stresses.

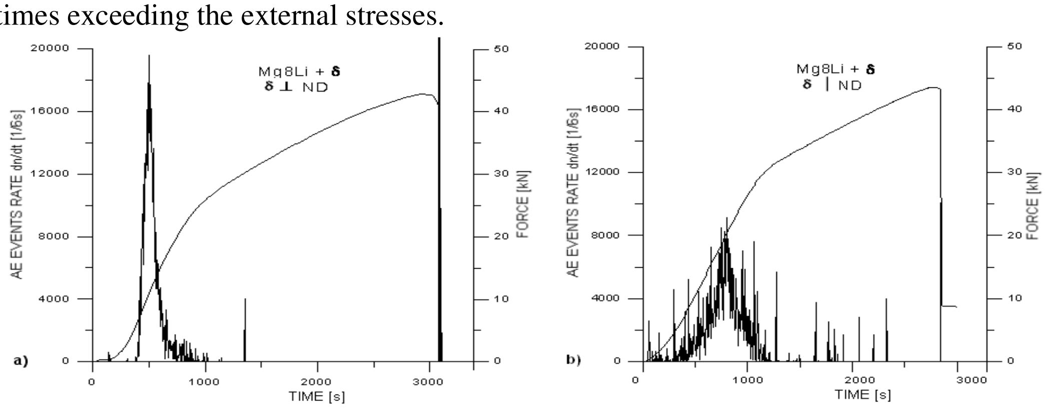

Figure 1. Effect of anisotropy of the distribution of with respect to compression direction ND in Mg8Li/δ composites subjected to channel-die compression at ambient temperature: (a) – perpendicular , (b) – parallel to ND.

On the other hand the presence of fibres, as the obstacles for moving dislocations, reduces the tendency to collective behaviour and both internal and surface annihilation of many dislocations, responsible for contribution to the registered AE signals [6,7].

Figure 2 presents the behaviour of AE in Mg8Li3Al AMC with different initial distribution of the fibres. Although a somewhat greater AE activity in the case of parallel fibres can be noticed (Fig.2b), yet this is not a satisfactory confirmation of the phenomenon of anisotropy. Instead, the very high level of AE in comparison to that at room temperature (Fig.1) and the second range of AE activity, after about 1500s, is observed. Thus the unexpected behaviour of AE at 140^0C, observed in Mg8Li3Al AMC premises to assume that it is due to the processes which are so dominating that the anisotropy effect is almost invisible. Namely, the second range of AE activity would be connected with the enhanced micro cracking of fibres at 140^0C. Owing to the application of scanning microscopy this suggestion has been strongly confirmed by microstructures presented at the bottom of Fig.2.

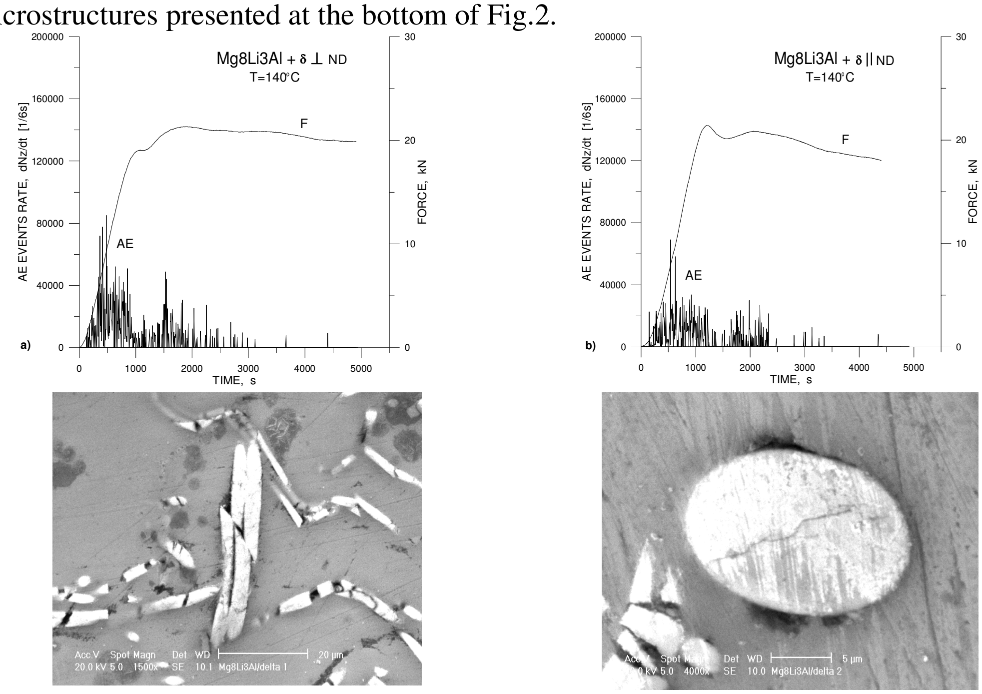

Figure 2. AE and the external force F in Mg8Li3Al AMC compressed at elevated temperature (140^0C):(a) – perpendicular , (b) – parallel to ND.
At the bottom: typical scanning microstructures after deformation.

These microstructures have revealed that the micro cracks of might 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 the decohesion of the from the matrix, the so-called debonding, at last by the processes forming along phase boundaries or due to the differences in the coefficients of thermal expansion of the ceramic and the metallic matrix. It is probable that also the process of relaxation type related with very high diffussivity of lithium in the matrix may play some role.

In situ observation

The processes of ceramic micro cracking were observed also using *in situ* technique. Figure 3 presents the results of these observations.

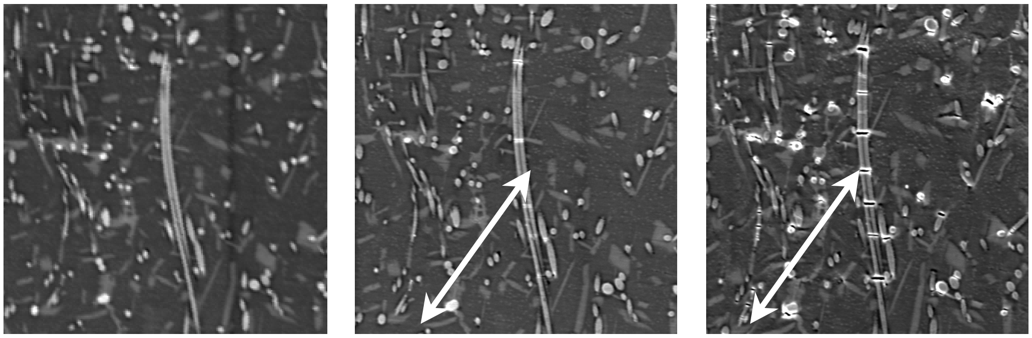

Figure 3. An example of sequence of fiber fracturing and/or failure evolution in tensile strained Mg8Li AMC. At the left side: non-strained sample before test. Image in center and at the right side: results after tensile test, the arrows point to the tensile direction.

AE in ceramic materials

The principal effect of the degradation of the ceramic materials are large cracks in the matrix. In case of C 120 technical porcelain, cracks are often branched and curved. Figure 4 (at the left) shows critical cracks in the central part of the sample loaded up by compression to about 400 MPa. The smaller ones join and form larger and more branched cracks. Their propagation is facilitated by destroyed structure elements, in particular grains of quartz. Large cracks are subjected to strong branching inside the sample. Big cascades of splitting cracks were also observed. Figure 4 (at the right) shows the typical course of EA as a function of the load for this type of porcelain. There is a characteristic strong increase of the activity of the AE in the critical load range.

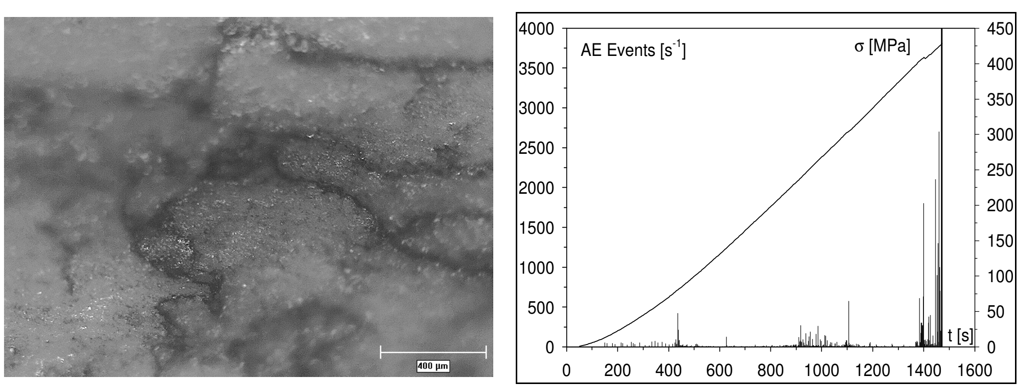

Figure 4. Microstructure of porcelain of C 120 type (at the left) together with a typical course of AE at critical stage of material degradation (at the right).

In the case of corundum ceramic an essential and the most distinctive effect of the critical stage of the structure destruction is the propagation of large cracks, starting from the central part of the samples (Fig.5 at the left). Large cracks are at first of all of intergranular type (intercrystalline cracking), but at the stage of rapid growth they also pass through the grains, especially of larger diameter (transcrystalline cracking). The growth of these cracks is the source of a series of strong AE signals (Fig.5 at the right).

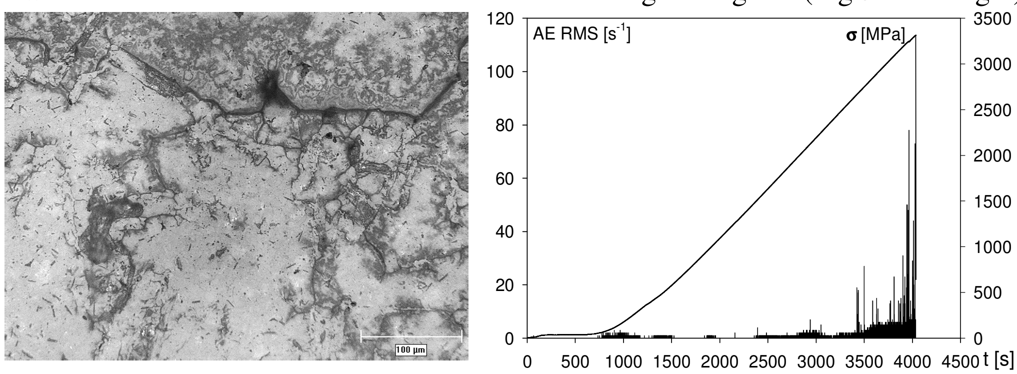

Figure 5. Microstructure of corundum ceramic (at the left) and typical course of AE (at the right) in the critical stage of degradation of the material.

AE in Mg-Al AMC reinforced with carbon

Figure 7 shows the results of compression tests and AE behavior in the Mg2Al AMC reinforced with carbon with a low concentration of order of 5.8 vol.%, situated perpendicularly (Fig.7a) and parallel (Fig.7b) to the ND axis.

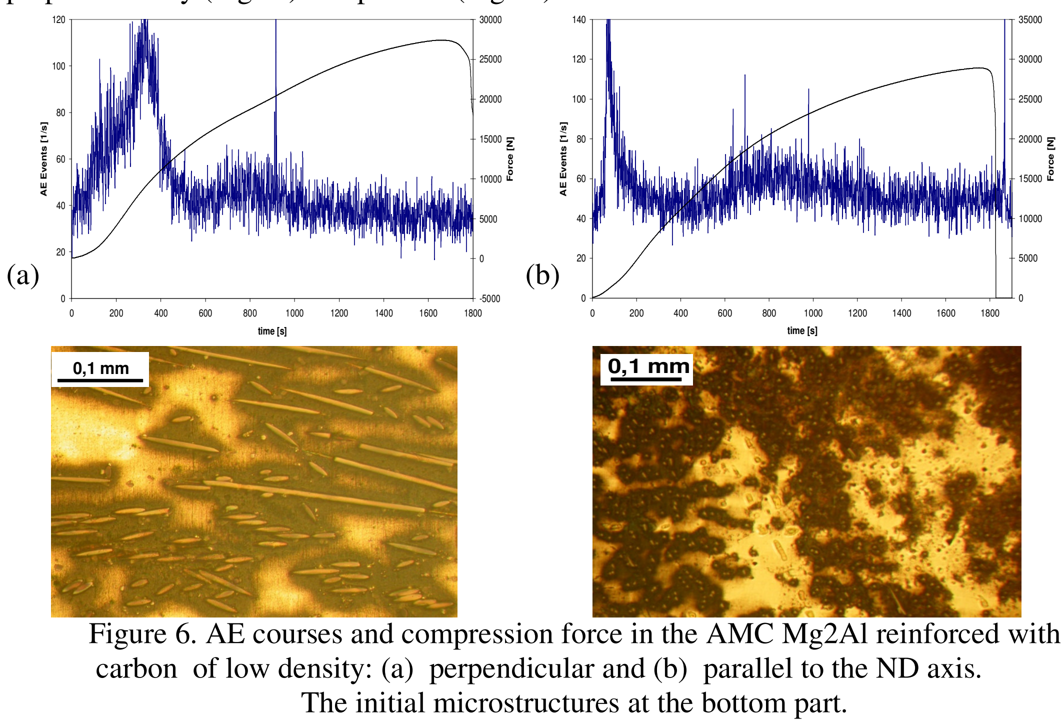

Figure 6. AE courses and compression force in the AMC Mg2Al reinforced with carbon of low density: (a) perpendicular and (b) parallel to the ND axis. The initial microstructures at the bottom part.

In addition, the Figure 6 (bottom) shows the corresponding microstructures illustrating the position of against the compression axis. Analyzing briefly these results one can admit that the AE in the composites with fibers placed parallel to ND is noticeably higher than in the composites of perpendicular fibers orientation. This new observation allows therefore to conclude that the effect of anisotropy of fiber distribution in respect to the compressive axis, observed in the case of composite with ceramic – takes place also in the case of composite reinforced with carbon phase. On the other hand, Figure 7 shows that also the process of carbon micro cracking occurs quite similar as in the case of ceramic reinforcement illustrated in Fig.3.

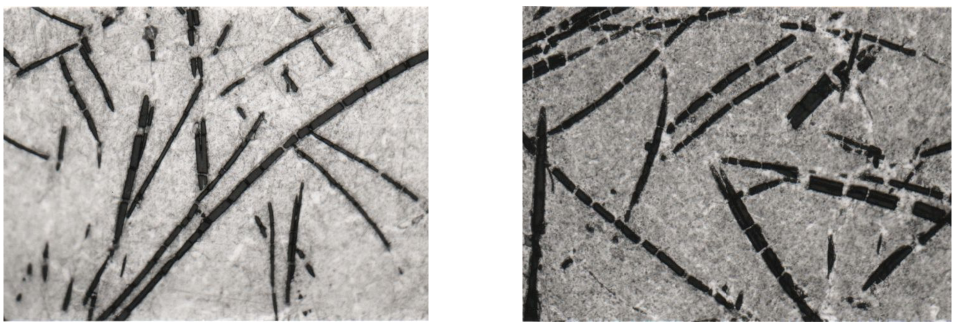

Figure 7. Example illustrations of micro cracking process of carbon due to small (at the left) and large plastic deformation (at the right).

Spectral analysis of AE signals

The Windowed Fourier Transform, offered by the AE analyser of new generation, enables for the construction of spectral characteristics of AE signal recorded during the process of fibre breaking. An example of the sequence of fibre fracturing is presented in Fig. 8. It can be stated that different orientation of strained and damaged result in different signal spectra of the registered AE signal. It is expected that more and more information about the mechanisms of the processes generating AE will be obtained applying this method.

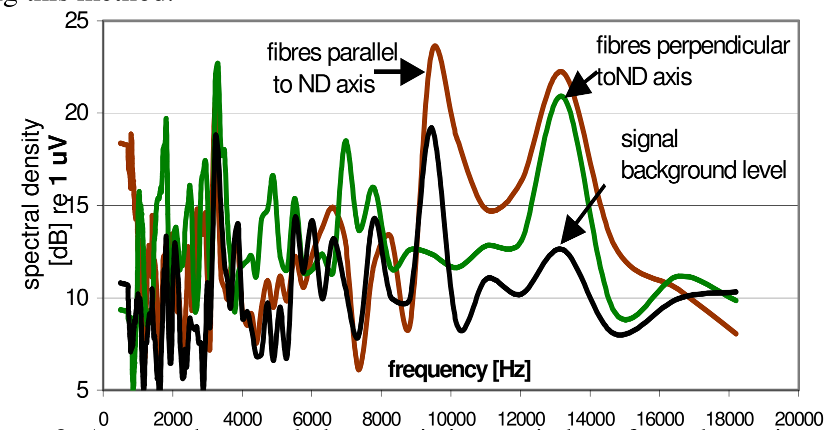

Figure 8. Averaged spectral characteristics carried out from the region of the greatest AE activity of compressed Mg8Li3Al AMC.

Regarding the images presented in Fig.8 it is necessary to emphasize that a low frequency part of the signal spectrum, i.e. 0 – 8 kHz is caused by the operation of the Instron drive. The AE activity coming from the process of breaking is situated in the region of frequencies between 8 and 16 kHz. The process of breaking the parallel to the compression direction ND caused the emission of AE signals of greatest amplitude and of relatively broad bandwidth.

CONCLUSIONS

- AE behavior in Mg8Li AMC reinforced with ceramic channel-die compressed at room temperature reveals the occurrence of the effect of anisotropy of distribution with respect to the compression axis.
- The anisotropy effect occurs also in Mg2Al AMC reinforced with carbon compressed at room temperature.
- Higher intensity and the two-range character of AE activity in Mg8Li3Al AMC compressed at 140^0C may be related to the enhanced of micro cracking due to the shear by dislocation pile-ups, the debonding processes and the difference in thermal coefficients of and matrix.
- The AE method is very useful for the identification of kinds micro cracking processes including those observed in porcelain and corundum ceramics.

ACKNOWLEDGEMENTS

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