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MICROCRACKING OF CERAMIC FIBRES AND ACOUSTIC EMISSION IN CHANNEL-DIE COMPRESSED Mg-Li-Al ALLOYS MATRIX COMPOSITES

Andrzej Pawełek, Andrzej Piątkowski, Zdzisław Jasieński

Aleksander Krupkowski Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Reymonta 25, 30-059 Cracow, Poland, e-mail: nmpawele@imim-pan.krakow.pl

Stanislav Kúdela, Stanislav Kúdela, Jr

Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Račianska 75, 831 02 Bratislava 3, Slovakia

Zbigniew Ranachowski, Przemysław Ranachowski

Institute of Fundamental Technological Research, Polish Academy of Sciences, Pawińskiego 5B, 02-106 Warsaw, Poland

The object of investigation was the behaviour of acoustic emission (AE) during channel-die compression of the Mg-Li-Al alloys matrix composites (AMC) reinforced with ceramic δ-Al_2O_3 fibres at room and at elevated temperature. The results of AE measurements at room temperature showed that in the majority of the investigated compositions the effect of anisotropy of the fibres distribution (planar random distribution) appeared with respect to the compression axis, whereas the AE activity at 140^0C revealed a two-range character and the level of the rate of AE events was higher than that at room temperature. These effects are discussed in terms of both, the differences in thermal expansion as well as weakening of the coherency between the fibres and the matrix. The results of AE measurements served to plot the spectral characteristics of the registered AE signals, i.e. the spectral density of AE signal as a function of its frequency. The spectral analysis of AE signals generated during the microcracking process of ceramic fibres in channel-die compressed Mg-Li-Al AMC was performed with the Windowed Fourier Transform method. The results are also discussed on the basis of optical and scanning microstructure images including these *in-situ* observations of microcracking fibres as well as in the context of the dislocation strain mechanisms and microcracking ones during the channel-die compression of the Mg-Li-Al AMC.

1. Introduction

Composites based on Mg-Li-Al alloys reinforced with ceramic fibres 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 4wt.% the hexagonal phase α of *hcp* structure occurs, while the alloys containing more than 12wt.% 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 4wt.% up to 12wt.% 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 composites based on the Mg12Li3Al and Mg12Li5Al alloys – containing the β phase – and also in those based on the Mg8Li3Al and Mg8Li5Al alloys – containing the α+β phases - subjected to channel-die compression at ambient 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, observed earlier at ambient temperature in the Mg/δ-Al_2O_3 and Mg8Li/δ-Al_2O_3 composites [2,3].

Moreover, since an AE analyser of a new generation has been recently available preliminary investigations of composites based on Mg8Li5Al and Mg12Li3Al alloys have been carried out at room temperature, along with the comparative analysis on the one used so far. On the basis of the qualitatively new results spectral characteristics (spectral densities of AE signal as a function of frequency) have been plotted applying the Windowed Fourier Transform analysis of AE signals generated in the process of microcracking of the ceramic fibres.

Using the results obtained so far [2-4] an attempt has been made to explain the correlations between the course of the intensity and activity of AE and the strength properties as well as the microstructure observed before and after deformation. The results have been discussed on the basis of the dislocation mechanisms of plastic flow and the mechanisms of microcracking in the composites containing the β phase. The conceptions of collective acceleration and surface annihilation of dislocations as the main reasons for AE in metals reported by [5-7] have been also considered.

2. Experimental

Composites based on 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 fibres 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 δ-Al_2O_3 fibres, whose mean length oscillated from 100 to 500μm, and the mean size of the diameter was 3÷4μm. Samples of the alloys and composites intended for channel-die compression tests had the shape of cubes of side 10mm.

The compression tests were carried out using INSTRON 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 0.05mm/min. Simultaneously with the registration of the external force F, the AE parameters, mainly *AE events rate*, denoted as $\Delta N_z/\Delta t$, and in some cases also the energy of AE events were measured. A broad-band piezoelectric sensor enabled the registration of acoustic pulses in the frequency range from 10kHz to 1MHz. 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 bandwith.

Additionally, the microstructural observations of Mg12Li3Al/δ composite were carried out by means of a scanning microscope on samples prior to their deformation (primary state) and on samples deformed at room temperature and at 140^{0}C. In this way, besides the acoustic characteristics (rate of AE events and the energy of AE events as a function of time) and the mechanical characteristics (work-hardening curves in the force-time version) the scanning microstructures – reflecting the effects of the operation of the strain and the microcracking mechanisms – represents essential elements in the discussion of the results. The external force-time curve corresponds, with good approximation, to the work-hardening curve in the stress-strain version, since the elongation of the sample changed linearly with time and the traverse speed of the testing machine was constant.

3. Results and discussion

The starting point for the discussion of the AE behaviour and the strain mechanisms in composites based on single- and two-phase Mg-Li-Al alloys, subjected to channel-die compression at room and at elevated (140^{0}C) temperatures, are the results obtained so far, presented in the publications [2-4,7].

3.1 AE in Mg8Li3Al/δ and Mg8Li5Al/δ composites

Figure 1 shows the courses of the rate of AE events and the external compressive force as a function of time of the channel-die compressed composites based on the two-phase Mg8Li3Al alloy. The compression tests were carried out at room temperature both for samples in a perpendicular (Fig. 1a) and a parallel (Fig. 1b) position of the ceramic fibres with respect to the compression axis ND. The first test had been already reported but the result obtained then was not fully convincing. It can be seen that AE activity is distinctly greater in the case of parallel fibres (Fig. 1b) than in the case of perpendicular fibres (Fig. 1a). Thus it should be concluded that the effect of the anisotropy of the fibre distribution, observed earlier in the Mg8Li/δ composite [4], can be also observed at ambient temperature in the composites based on two-phase Mg8Li3Al alloys.

Figure 2 shows the behaviour of the rate of AE events and the external force in Mg8Li5Al/δ composites, with fibres situated parallel to ND, compressed at ambient temperature (Fig. 2a) and at elevated temperature. The course of the events rate (Fig. 2b) confirms clearly the occurrence of two ranges of AE activity and a significantly higher level of the rate of AE events in comparison with identical composites deformed at room temperature (Fig. 2a). These effects were observed earlier [4] in the Mg8Li3Al/δ composite compressed at 140^{0}C. It is possible, that they are related to both the processes of microcracking of the fibres and the generation of dislocations due to the stresses resulting from differences in the coefficient of thermal expansion of the ceramic fibres and the metallic matrix. When comparing left part of Figure 2 and Figure 1b, it can be seen that the increase in Al content causes a small, however visible increase in the rate of AE events, which confirms the earlier suggested explanation of this fact [4] based on the increase in the volume fraction of acoustically very effective phase α.

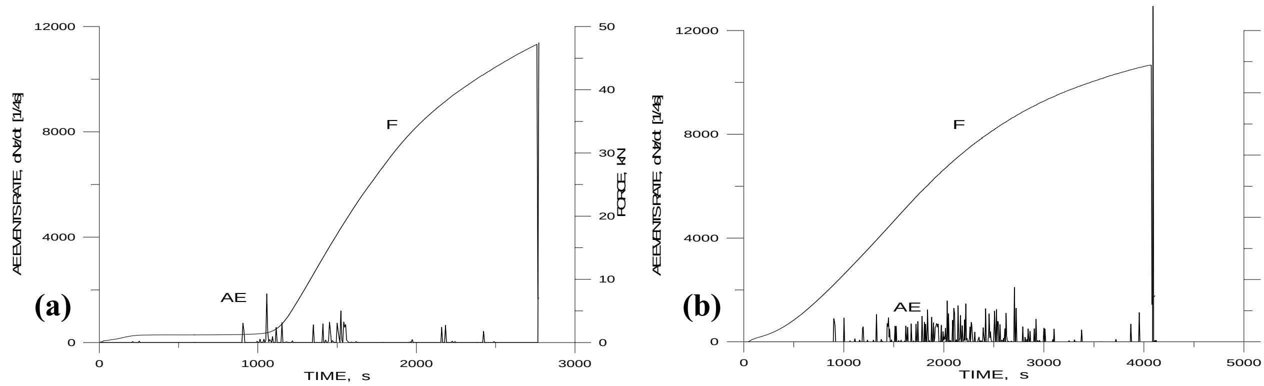

Figure 1. AE and the external force F in Mg8Li3Al/δ composites compressed at room temperature: (a) – perpendicular fibres, (b) – fibres parallel to the compression direction ND.

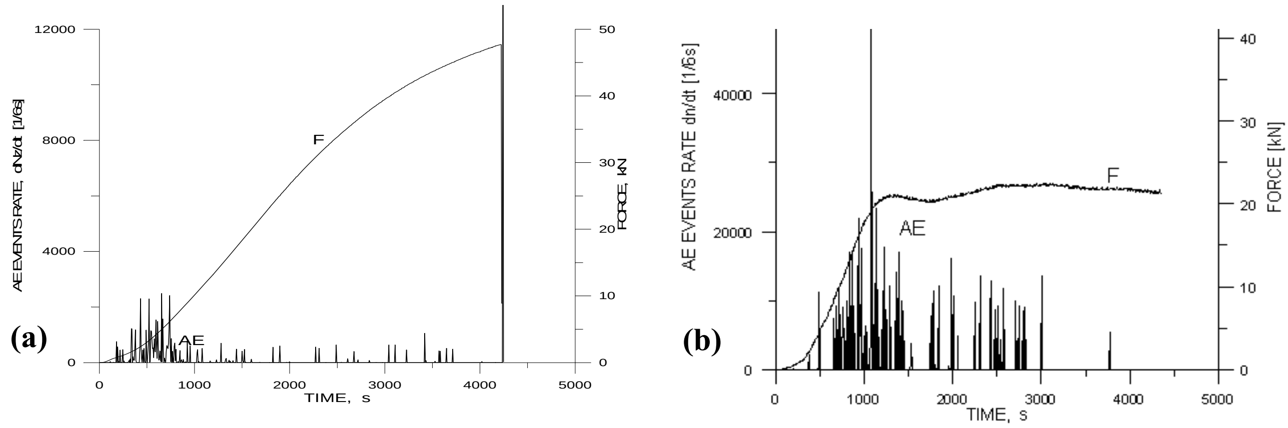

Figure 2. The EA and F courses in Mg8Li5Al/δ composites compressed at room temperature (a) and at 140^{0}C (b).

3.2 AE in Mg12Li3Al/δ and Mg12Li5Al/δ composites

Figure 3 shows AE events rate as a function of time and the course of loading force *F* in Mg12Li3Al/δ composite with perpendicular fibres and the scanning microstructures before (Fig. 3a) and after deformation (Fig. 3c) at room temperature. The course of the rate of AE events (Fig. 3b) confirms the earlier established fact [4] of the very low acoustic efficiency of the β phase. The initial microstructure (Fig. 3a) definitely solves the problem of the occurrence of microcracks of the fibres in the technological process of the production of composites. This means that the observed cracking of the fibres (Fig. 3c) occurred as a result of the deformation process of the composite.

Figure 4 presents AE events rate as a function of time and the course of loading force *F* in the Mg12Li3Al/δ composite with parallel fibres and the scanning microstructures before (Fig. 5a) and after deformation (Fig. 5c) at ambient temperature. When comparing AE activity (Fig. 5b) with AE activity in the composite with perpendicular fibres (Fig. 4b) it can be stated that the effect of the anisotropy of the fibres distribution occurs also in composites based on single-phase β alloys. Moreover, the initial microstructure (Fig. 4a) confirms the comments to Fig. 3a, although it should be noted that here it is more clearly visible that already in the initial state the fibres do not adhere closely to the matrix everywhere. The acoustic emission activity of a composite with parallel fibres presented in Fig. 4. is considerably higher than that presented in Fig. 3.

Figure 5 shows the courses of the rate of AE events and of the external force *F* in Mg12Li5Al/δ composite with parallel fibres, tested at ambient temperature (Fig. 5a) and at elevated temperature (Fig. 5b). The effects of breaking the fibres are increased at elevated temperature.

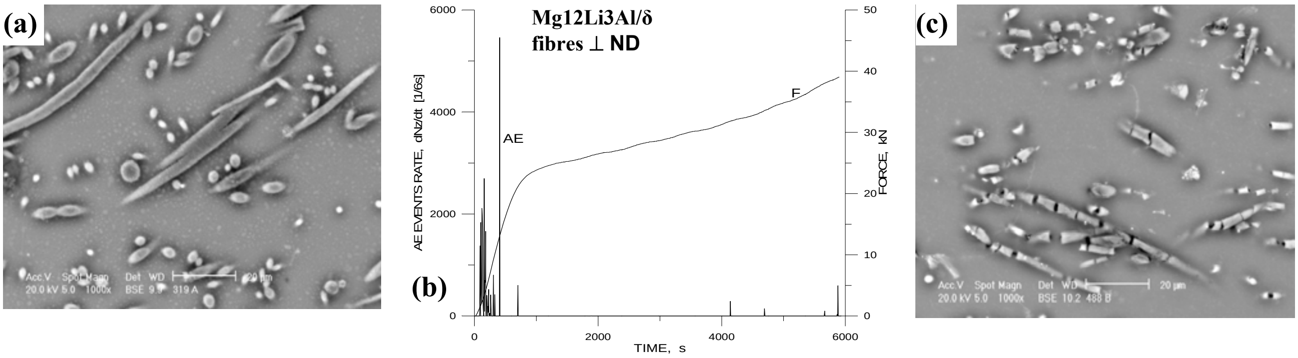

Figure 3. EA and F in Mg12Li3Al/δ composite with fibres perpendicular to the compression direction ND (b) and its scanning microstructures before (a) and after deformation (c) at room temperature.

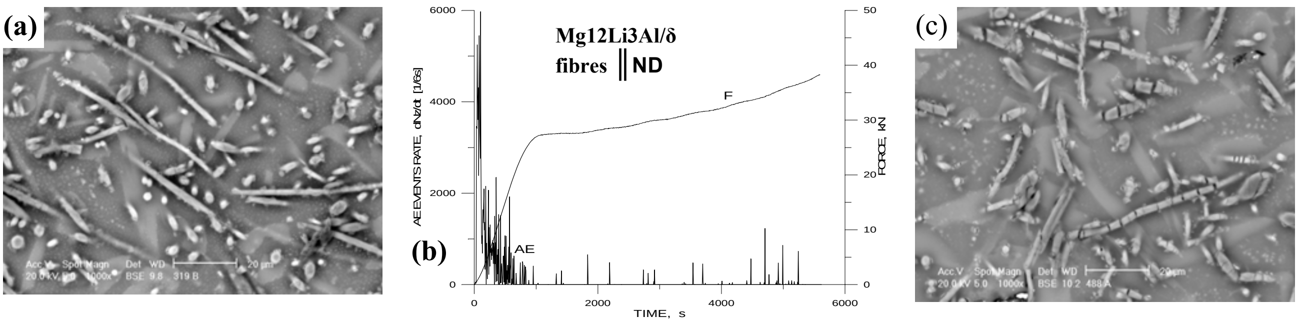

Figure 4. EA and F in Mg12Li3Al/δ composite with fibres parallel to the compression direction ND (b) and its microstructures before (a) and after deformation (c) at room temperature.

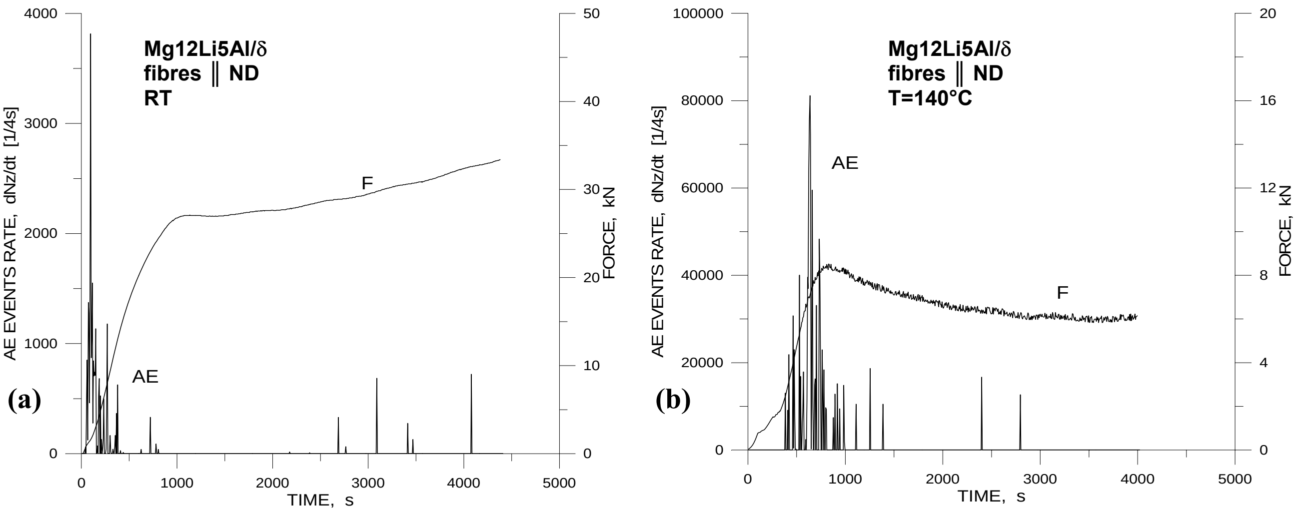

Figure 5. The EA and F courses in Mg12Li5Al/δ composites, with parallel fibres, compressed at room temperature (a) and at elevated temperature 140^{0}C (b)

When comparing the AE courses in Figs. 3 and 4 to those presented in Fig. 5, it can be noticed that the increase in Al content causes here an effect reverse to that in the Mg8Li3Al/δ composite. This is attributed to blocking of the collective dislocation motion resulting both from increased Al contribution to work hardening and a greater contribution of the LiAl phase particles. It should be noted that the course of the force and, as a consequence, the AE behaviour, shown in Fig. 5b, must be treated with great caution. The course of the force had a clearly jerky character throughout the duration of the compression test at 140^{0}C, and it is difficult to establish whether it was due to some new physical phenomena, e.g. Portevin – Le Chatelier effect or twinning, or whether it was the result of microcracking of the surface oxide layer.

It is also possible that the jerky courses of force observed in the Mg12Li5Al/δ and Mg8Li5Al/δ composites (Fig. 5b and Fig. 2b, respectively) are characteristic of composites containing the β phase. The jerky courses of force were observed also at ambient temperature in the Mg12Li3Al/δ composite with perpendicular fibres [4]. This unexpected behaviour of AE and force was the main reason to repeat that experiment, assuming that such a behaviour was most probably the result of microcracking of the surface oxide layer, which could not be entirely eliminated when preparing the sample for the experiment. The result of the repeated test (Fig. 3b) is reliable in the context of the discussion and comparison with other results. Thus it is very probable that even a well prepared sample of a composite containing the β phase, during its heating to 140^0C, becomes sufficiently oxidized to disturb the courses of AE and force.

3.3 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. It can be stated that different orientation of strained and damaged fibres 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

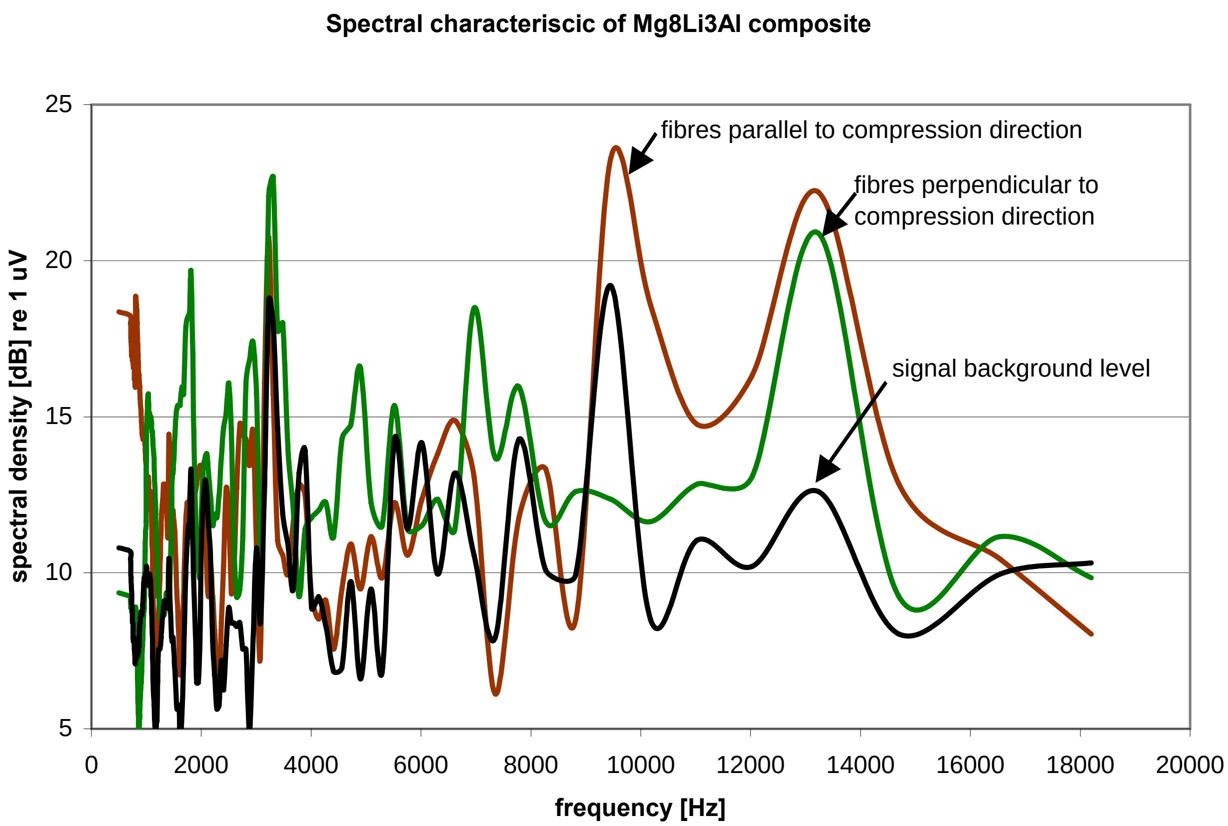

Figure 6. Averaged spectral characteristics carried out on the sets of 6 fragments of AE signal of 1s duration taken from the region of the greatest AE activity of compressed Mg8Li3Al/δ composite. The characteristics of the AE signal registered in the composite with fibres placed parallel to compression direction is compared with that registered in the composite with fibres placed perpendicular to compression direction and with the other one when no fibre breaks had occurred.

Regarding the images presented in Fig. 6 it is necessary to remember that a low frequency part of the signal spectrum, i.e. 0÷8kHz is caused by the operation of the INSTRON drive. The AE activity coming from the process of breaking fibres is situated in the region of frequencies between 8 and 16kHz. The process of breaking the fibres parallel to the compression direction ND caused the emission of AE signals of greatest amplitude and of relatively broad bandwidth. Averaged spectral characteristics registered in three Mg-Li alloys and in three composites made on the base of those alloys are presented in Fig. 7 together with some explanations placed below.

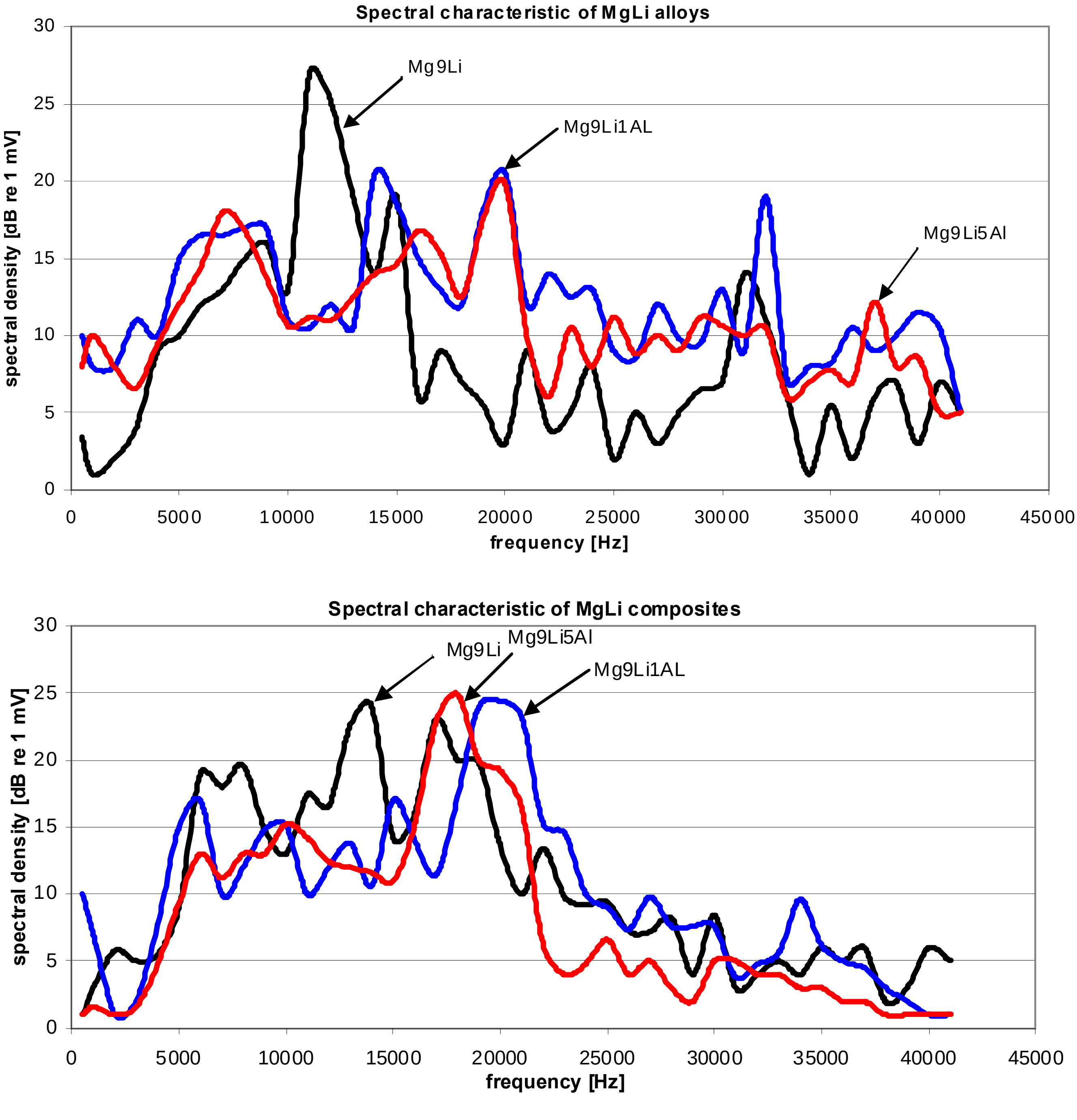

Figure 7. Averaged spectral characteristics carried out on the sets of 6 fragments of AE signal of 1s duration taken from the region of the greatest AE activity of various compressed Mg-Li alloys (above) and composites (below)The characteristics of the AE signal registered in the alloys has a more distinct broad–band character than that that registered in the composites. It is to be noted that the addition of the Al component improves the generation of the AE signal located at higher frequency region.

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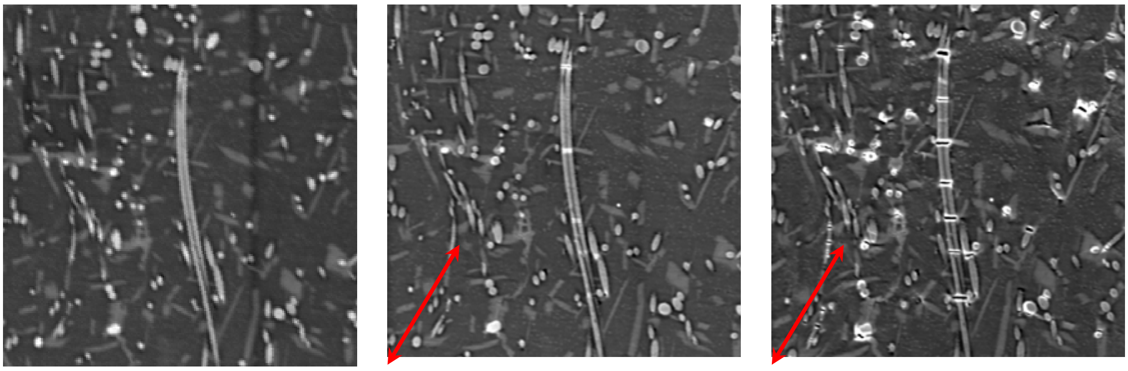

Non-strained sample   Results after tensile test

Figure 10. Sequence of fibre fracture and/or failure evolution in tensile strained of Mg8%wt.Li/δ-Al_2O_3 composites. The arrow denotes tensile direction

Conclusions

The examination of the behaviour of AE in the Mg8Li3Al/δ and Mg12Li3Al/δ composites during compression at ambient temperature have confirmed the occurrence of the effect of the anisotropy of fibres with respect to the compression direction. Higher maximal level of AE in the Mg8Li5Al/δ composite than in the Mg8Li3Al/δ one is the effect of the increased volume fraction of the very acoustically effective α phase with increasing Al content. The reverse effect in the Mg12Li3Al/δ and Mg12Li5Al/δ composites can be attributed to blocking the collective dislocation motion as a result of higher Al contribution to work hardening and to the appearance of the LiAl particles. Higher AE level and the occurrence of two ranges of AE activity at 140^0C in the Mg8Li5Al/δ composite have confirmed such a behaviour, observed earlier in the Mg8Li3Al/δ composite [3].

Acknowledgements

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