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ACOUSTIC EMISSION AND STRAIN MECHANISMS DURING COMPRESSION AT ELEVATED TEMPERATURE OF β PHASE Mg-Li-Al COMPOSITES REINFORCED WITH CERAMIC FIBRES

EMISJA AKUSTYCZNA I MECHANIZMY ODKSZTAŁCENIA PODCZAS ŚCISKANIA W PODWYŻSZONEJ TEMPERATURZE KOMPOZYTÓW β-Mg-Li-Al WZMOCNIONYCH WŁÓKNAMI CERAMICZNYMI

The object of investigations was the behaviour of acoustic emission (AE) during channel-die compression at room and elevated (140°C) temperatures of the composites based on Mg12Li3Al and Mg12Li5Al alloys containing the β phase as well as of the composites based on Mg8Li3Al and Mg8Li5Al alloys also containing the β phase. The results of AE measurements at room temperature show that the effect of anisotropy of the fibres distribution with respect to compression axis occurs also in composites based on Mg8Li3Al alloys. However, the results of AE measurements at 140°C show that the course of AE activity was a two-range character and that the level of the rate of AE events is higher than that at room temperature. These effects are attributed to the thermal weakening of the fibres strength at 140°C.

Moreover, using an AE analyser of new generation, the investigations of the composites based on Mg8Li5Al and Mg12Li3Al alloys have been carried out at room temperature. On the basis of the constructed acoustograms and spectral characteristics the preliminary wavelet analysis of AE signals generated in the microcracking process of ceramic fibres has been carried out. The results obtained using the old and the new AE analysers are compared and discussed also on the basis of scanning microstructure observations and on the basis of the dislocation mechanisms of deformation and microcracking processes in the composites containing the β phase of Mg-Li system.

Badania dotyczyły zachowania się emisji akustycznej (EA) podczas testów nieswobodnego ściskania w temperaturze otoczenia i podwyższonej (140°C) kompozytów na osnowie stopów Mg12Li3Al oraz Mg12Li5Al, zawierających fazę β, a także kompozytów na osnowie stopów Mg8Li3Al i Mg8Li5Al, również zawierających fazę β. Wyniki pomiarów EA w temperaturze pokojowej wskazują, że efekt anizotropii rozkładu włókien względem osi ściskania występuje również w kompozytach na osnowie stopów Mg8Li3Al. Natomiast wyniki pomiarów EA w temperaturze 140°C wskazują na dwuzakresowy przebieg aktywności EA oraz większy poziom tempa zdarzeń EA niż w temperaturze pokojowej. Wytłumaczono to osłabieniem termicznym wytrzymałości włókien w 140°C.

Ponadto, wykorzystując analizator EA nowej generacji, wykonano badania w temperaturze pokojowej kompozytów na osnowie stopów Mg8Li5Al i Mg12Li3Al. Przeprowadzono analizę porównawczą wyników uzyskanych przy użyciu nowego i dotychczas stosowanego analizatora EA. W oparciu o skonstruowane akustogramy i charakterystyki widmowe, przeprowadzono analizę falkową sygnałów EA generowanych w procesie mikropękania włókien ceramicznych. Wyniki przedyskutowano również w oparciu o obserwacje mikrostruktur skaningowych oraz w oparciu o dyslokacyjne mechanizmy odkształcenia i mechanizmy mikropękania w kompozytach zawierających fazę β układu Mg-Li.

1. Introduction

Composites based on Mg-Li-Al alloys reinforced with ceramic fibres δ-Al_2O_3 have found increasingly wide application as light and strong construction materials in the automotive, aircraft and cosmic industries. Mg-Li alloys can occur in the form of three different phases. In the concentration range of Li up to 4 wt% there occurs the hexagonal phase α of *hcp* structure, while the alloys containing more than 12 wt% occur in the form of β phase of a regular *bcc* structure. The mechanical properties of α phase are worse than those of the β phase, which is characterized by considerably higher plasticity, very good machinability and weldability [1]. Alloys with Li content from 4 wt% up to 12 wt% are two-phase alloys and they occur as a mixture of α + β

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phases. Alloy addition, e.g. 3% or 5% Al, although they slightly increase the density of the composites, they distinctly improve their strength properties.

The performed investigations were intended to determine the relations between the behaviour of acoustic emission and the strain mechanisms, first of all, in composites based on the alloys Mg12Li3Al and Mg12Li5Al – containing β phase – and, less so, in composites based on the alloys Mg8Li3Al and Mg8Li5Al – containing a mixture of $\alpha + \beta$ phases – subjected to channel-die compression at ambient temperature and at elevated temperature 140°C. The latter investigations were carried out also with respect to the possibility of the occurrence of the effect of the anisotropy of the fibres distribution with respect to the compression direction, observed earlier at ambient temperature in the composites Mg/δ-Al_2O_3 and Mg8Li/δ-Al_2O_3.

Moreover, since an AE analyser of a new generation has been recently installed at the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences, there have been carried out preliminary investigations, at room temperature, of composites based on Mg8Li5Al and Mg12Li3Al alloys, as well as a comparable analysis of the results obtained when using the new AE analyser and the one used so far. On the basis of the qualitatively new results there have been prepared preliminary acoustic maps (acoustograms) and the spectral characteristics (spectral densities as a function of frequency), on the basis of which there has been carried out the first wavelet 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 and microstructure observed before and after deformation by means of a scanning microscope. The results have been discussed on the basis of the dislocation mechanisms of plastic flow and the mechanisms connected with the processes of microcracking in composites containing the β phase. There have been also considered the conceptions known from literature [5, 6], believing the main reasons of AE in metals to be the processes of collective acceleration and surface annihilation of many dislocations.

2. Investigation methods

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 in Bratislava. They were produced from a preliminary material – a fibrous skeleton obtained from 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% of the volume, and their proportion in the composite was equal to 10% of the volume. The obtained composites were characterized by the planar random distribution of the ceramic fibres δ-Al_2O_3, the mean length of which oscillated from 100 to 500 μm, and the mean size of the diameter was within the limits 3÷4 μm. Samples of the alloys and composites intended for channel-die compression tests had the shape of cubes with 10 mm edges.

The compression tests were carried out using INSTRON-6025 tensile testing machine, additionally equipped with a specially constructed channel-die which guarantees 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 strain is realized, since in the direction perpendicular to the channel walls (transverse direction – TD) no deformation takes place. The velocity of the traverse of the testing machine was 0.05 mm/min.

Simultaneously with the registration of the external force F, there were measured the AE parameters, mainly AE events rate, labelled as $\Delta N_z/\Delta t$, and in some cases also of the energy of AE events so as to enable a comparison with the results obtained using the AE analyser of the new generation. A broad band piezoelectric sensor enabled to register acoustic pulses in the frequency range from 100 kHz to 1 MHz. The contact between the sensor with the sample was maintained by means of a steel rod used as a pad in the channel-die. Measurements at the temperature 140°C were carried out using a specially profiled quartz wave-guide. In each test of channel-die compression the number of AE events was recorded in the time interval Δt = 6s. The total amplification of the acoustic signals was 80 dB, and the corresponding optimal threshold voltage was 1.19 V. In order to eliminate the undesired effects of friction against the channel walls each sample was covered with Teflon foil.

Application of a new AE analyser offers additional possibilities of the form of the registration of AE events by recording their duration, amplitude and the root mean square (RMS) value of an AE signal in the course of each of the registered events. Thus it offers the possibility of analysis of single AE bursts. Not less important and useful is also the possibility of drawing the distribution of the population of the registered AE events as a function of their energy.

Additionally, in the case of Mg12Li3Al/δ composites there were carried out microstructural observations by means of a scanning microscope on samples prior to their deformation (primary state) and on samples after deformation at room temperature and on samples after deformation at the temperature 140°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 dependence 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°C) temperatures, are the results obtained so far, in the years 2001–2003, especially those presented in the Report of the Institute of Metallurgy and Materials Science for the year 2003 [2] and those presented in the publications [3, 4, 7, 8].

3.1. Acoustic emission in Mg8Li3Al/δ and Mg8Li5Al/δ composites

Figure 1 shows the courses of the rate of AE events and of the external compressive force as a function of the duration of the test of channel-die compression of composites based on two-phase Mg8Li3Al. The compression tests were carried out at room temperature both for composites 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 is a repetition of a test from the previous year, as 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 fibres distribution, observed earlier in Mg8Li/δ composites [2], is observed also at ambient temperature in composites based on two-phase Mg8Li3Al alloys.

Figure 2 shows the behaviour of the rate of AE events and of the external force in Mg8Li5Al/δ composites, with their fibres situated parallel to ND, compressed at ambient temperature (Fig. 2a) and at elevated temperature 140°C. The course of the events rate (Fig. 2b) confirms clearly the occurrence of two ranges of AE activity and a considerably higher level of the rate of AE events in comparison with identical composites deformed at room temperature (Fig. 2a). These effects were observed earlier [2] in Mg8Li3Al/δ composites compressed at 140°C. Then they were attributed to thermal weakening of the ceramic fibres at 140°C. Maybe, there should also be taken into consideration both the processes of microcracking of the fibres and the generation of dislocations due to stresses resulting from differences in the coefficient of thermal expansion of the ceramic fibres and the metallic matrix.

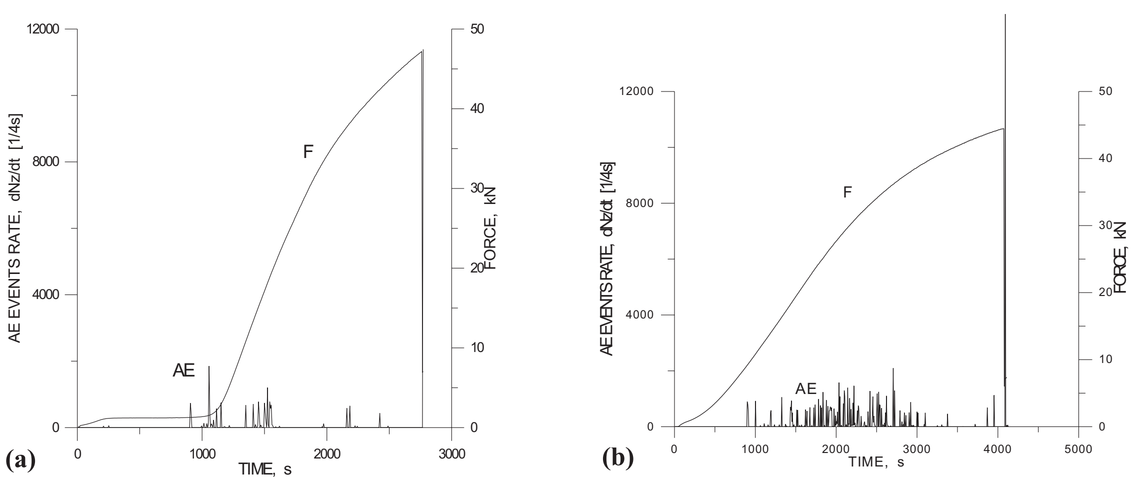

Fig. 1. AE and the external force F in Mg8Li3Al/δ composites compressed at room temperature:
(a) – perpendicular fibres, (b) – fibres parallel to the compression direction ND

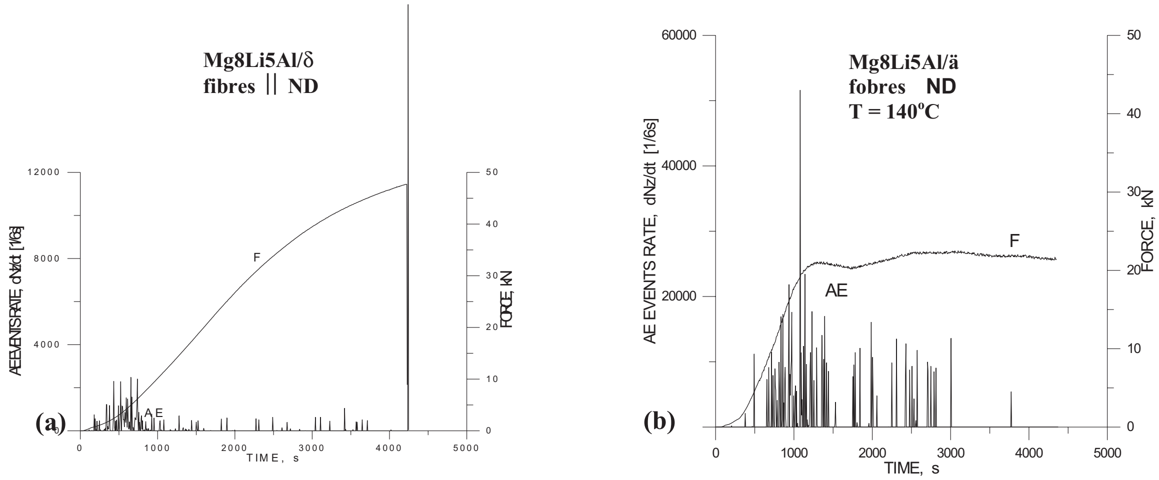

Fig. 2. The AE and F courses in Mg8Li5Al/δ composites compressed at room temperature (a) and at elevated temperature 140°C (b)

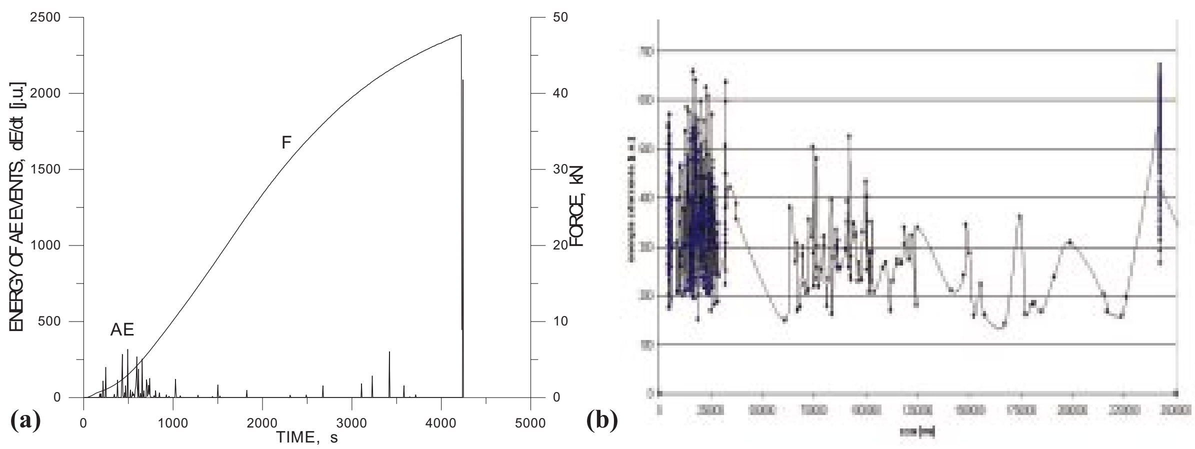

Fig. 3. Conventional energy of EA events in Mg8Li5Al/δ composite compressed at room temperature, measured by means: (a) – analyser used so far, and (b) – new AE analyser

When comparing the courses of AE activity (Fig. 2a) and of the energy of AE events (in arbitrary units, Fig. 3a) it can be seen that they are qualitatively similar. This proportionality indicates that in the AE spectrum there does not occur even a very small population of the number of events of very great energy, which might disturb such a proportionality. The graph of the energy of AE events has been quoted here considering the possibility of a first, preliminary comparison with the results (Fig. 3b) obtained using the AE analyser of the new generation. When analyzing the courses in Figs 3a and 3b it can be said that they are similar enough in terms of quality. However, the results obtained using the new analyser have provided additional elements. In the range up to 1000s (in Fig. 3b time is given in ms) two different ranges of AE activity are much better visible than in Fig. 3a.

Moreover, when comparing Figs 2a and 1b, it can be seen that the increase in Al content causes a small, however visible increase of the level of the rate of AE events, which confirms the earlier suggested explanation of this fact [2] based on the increase in the volume fraction of the very acoustically effective phase α.

3.2. AE in Mg12Li3Al/δ composites

Figure 4 shows AE in Mg12Li3Al/δ composites with perpendicular fibres and the scanning microstructures before (Fig. 4a, initial state) and after deformation (Fig. 4c) at room temperature. The course of the rate of AE events (Fig. 4b) confirms the earlier established fact [2–4] of the very weak acoustic efficiency of the β phase, and the initial microstructure (Fig. 4a) 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 microcracks of the fibres (Fig. 4c) have been formed as a result of the deformation process of the composite.

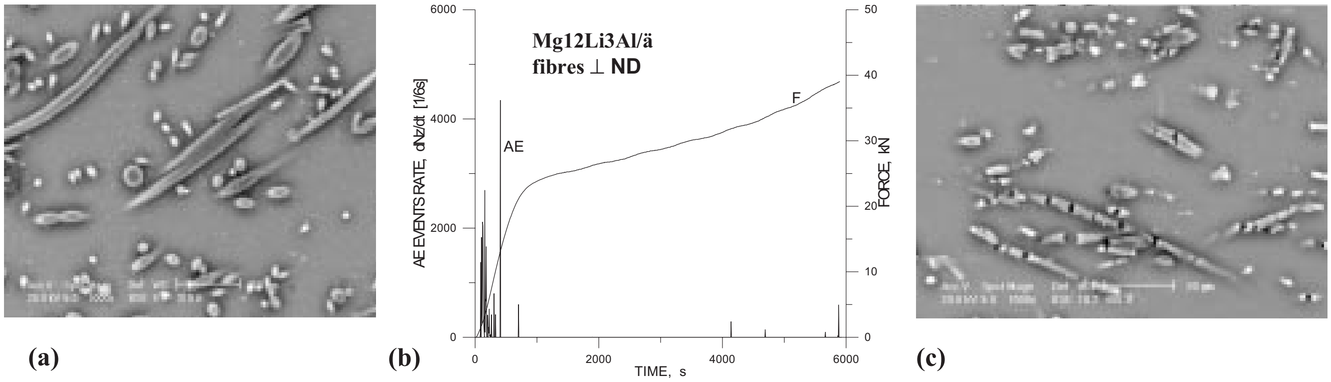

Fig. 4. AE and F in Mg12Li3Al/δ composite with fibres perpendicular to the compression direction ND (b) and its scanning microstructures before (a) and after deformation (c) at room temperature

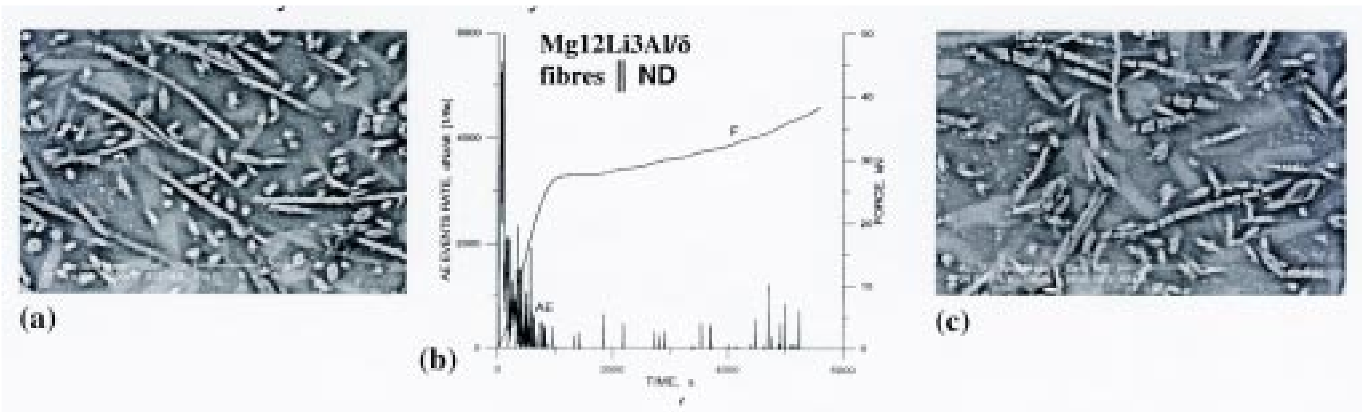

Fig. 5. AE and F in Mg12Li3Al/δ composite with fibres parallel to the compression direction ND (b) and its microstructures before (a) and after deformation (c) at room temperature

Figure 5 presents AE in Mg12Li3Al/δ composites with parallel fibres and the scanning microstructures before (Fig. 5a, initial state) and after deformation (Fig. 5c) at ambient temperature. When comparing AE activity (Fig. 5b) with AE activity in a 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. It should be emphasized here that the small difference in the fibres density (Fig. 4c and Fig. 5c) may also contributes to this effect, however the results of our earlier papers [2, 3, 8] suggest that it is rather not important.

Moreover, the initial microstructure (Fig. 5a) confirms the comments to Fig. 4a, 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.

3.3. AE in Mg12Li5Al/δ composites

Figure 6 shows the courses of the rate of AE events and of the external force F in Mg12Li5Al/δ composites with parallel fibres, tested at ambient temperature (Fig. 6a) and at elevated temperature 140°C (Fig. 6b). When comparing the AE courses in Fig. 6a and 6b it can be noticed that the increase in Al content causes here an effect reverse to that in the composites Mg8Li3Al/δ. This is attributed to blocking of the collective motion of the dislocations resulting both from increased Al contribution to work hardening and a greater contribution to the population of the particles of LiAl phase precipitates.

It should be noted here that the course of the force and, as a consequence, the AE behaviour, shown in Fig. 6b, must be treated with great caution. The course of the force has a clearly jerked character throughout the duration of the compression test at 140°C, and it is difficult to establish whether it is due to some new physical phenomena, e.g. Portevin – Le Chatelier effect or twinning, or whether it is the result of e.g. microcracking of the surface oxide layer.

Maybe, the jerked courses of force in Mg12Li5Al/δ composites (Fig. 6b), or even, although in a much smaller degree, in Mg8Li5Al/δ composites (Fig. 2b) – thus, similarly as in Mg12Li3Al/δ composites, where these effects were observed earlier [2] – at elevated tempera-

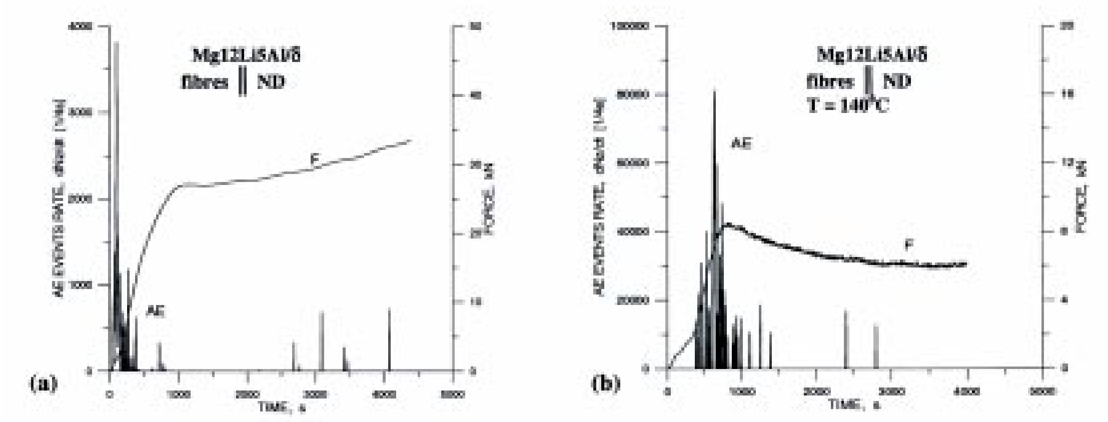

Fig. 6. The EA and F courses in Mg12Li5Al/δ composites, with parallel fibres, compressed at room temperature (a) and at elevated temperature 140°C (b)

tures they are characteristic of composites containing the β phase. Nevertheless, the jerking remains a problem the more so that jerked courses of force were observed also at ambient temperature in Mg12Li3Al/δ composite with perpendicular fibres [2]. This unexpected behaviour of AE and force was the main reason to repeat this experiment, assuming that such 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. 4b) 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 the temperature 140°C, becomes sufficiently oxidized to disturb the courses of AE and force, and thereby it does not reflect the actual processes occurring during deformation. In case of these composites, particularly susceptible to oxidizing, it is planned in future to curry out channel-die compression tests in a protective atmosphere.

3.4. Preliminary wavelet analysis of AE in the composites Mg8Li5Al and Mg12Li3Al

Using the new analyser there were carried out additional AE measurements - from the point of view of preliminary wavelet analysis of AE signals – for the following channel-die compressed composites: Mg8Li5Al/δ with parallel fibres and Mg12Li3Al/δ with the fibres parallel and perpendicular to the compression axis ND. The wavelet analysis, when treating it at this stage of the investigations briefly and without details, consists in the application of the algorithm of the decomposition of AE signal in the sense of deriving the spectral characteristics of this signal. The principle of the operation of this algorithm has its beginning in the Fourier method, describing the decomposition of integrable functions into trigonometric series. The registered AE signals are stored in a computer in a discrete form as a series of samples $\nu(mT_l)$, where $T_l = T/N$, T – length of a selected time window, N – number of samples. Using this notation we can write the algorithm of Fourier's discrete transformation in the following form:

$$C_n = (1/N) \sum_{m=0}^{N-1} \nu(mT_l) \exp\,(2\pi jnm/N). \qquad (1)$$

The coefficients C_n can be interpreted as a transformed set of information obtained from the temporal form of AE signal. The information becomes transformed from the region of time into the region of frequency, and the coefficients C_n describe the spectral components of the examined AE signal in the time window under consideration.

As a result of the wavelet decomposition, thanks to another algorithm, the series of the signal samples becomes transformed into a two-dimensional set in the time-frequency coordinates. This algorithm is realised by the determination of scalar products of the segments of a series of AE signal samples and the sets Ψ_{mn}, called the family of wavelets. The wavelets are formed through transformation of the basic form of a wavelet. For the sake of simplification, assuming that the wavelets are described by continuous functions, this transformation can be written in the following form:

$$\psi_{mn}(t) = a_0^{-m/2}\, \psi(a_0^{-m}\, t - nb_0), \qquad (2)$$

where $\Psi(t)$ denotes the function of a basic wavelet, $\Psi_{mn}(t)$ denotes the family of wavelets generated trough m-fold elongation of the base vectors and n-fold shifting on the time axis, a_0 – scale coefficient, b_0 – coefficient of shifting on the time axis. In this way, as a result of the operation of software, the data are transformed into the form of a two-dimensional table, which in the time-frequency system describes the temporary effective values of AE signal. These data, in the graphic form, represent an acoustic map, called briefly acoustogram. To each point of the acoustogram a certain colour is assigned (i.e. from blue through yellow to red or in gray scale) which encodes the intensity of the AE signal – i.e. the temporary value of the spectral density of the registered signal. The spectral density, $\omega = \Delta E/\Delta f$, informs about the value of the energy (ΔE) of the AE signal for the given interval (Δf) of frequency. The energy E, measured in the given time interval, is thus the integral from the spectral density over the whole frequency band, i.e. according to the formulae $E = \int \omega\,(f)df$. The graph of spectral density as a function of frequency, i.e the spectral characteristics supplies much valuable information to classify different signal sources, since, speaking briefly, it indicates the frequencies which carry the maximal energy of the events, present in the AE signal.

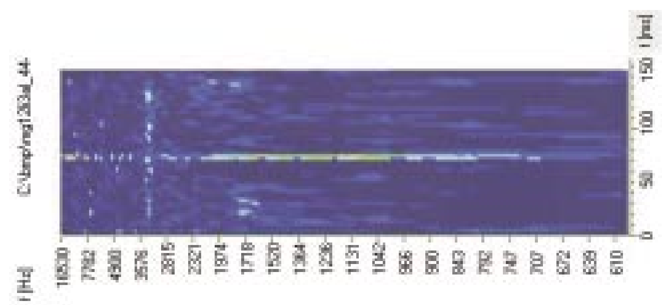

Fig. 7. Preliminary wavelet analysis of a fragment of an AE signal at the initial compression stage of Mg12Li3Al/δ composite with fibres parallel to ND axis. Acoustogram of the area of maximal energy of the EA signal

Figure 7 presents the results of a preliminary wavelet analysis for the composite Mg12Li3Al/δ with fibres parallel to ND axis. This composite has been selected on account of the effect of anisotropy of the fibres distribution. Both the frequency of the occurrence of the events (AE activity) and the number of events (AE intensity) are greater in a composite with the fibres parallel than in a composite with the fibres perpendicular to ND axis, and this offers the possibility to get more information. The acoustogram (Fig. 7) refers to the initial section of loading in which there occurs a sharp AE peak originating, as it seems, from cracking of the weakest fibres. Fig. 8. presents averaged spectral characteristics of the AE signal registered in the composite shown in Fig. 1b, averaged spectral characteristics registered in the composite shown in Fig. 1a and averaged spectral characteristics registered in the composite when no fibre breaks occurred. In the last case the spectral characteristics in the frequency range of 1–10 kHz reflects the activity of the drive of the loading machine, while the spectral components shown in two previous figures situated above 10 kHz are caused by the breaking fibres.

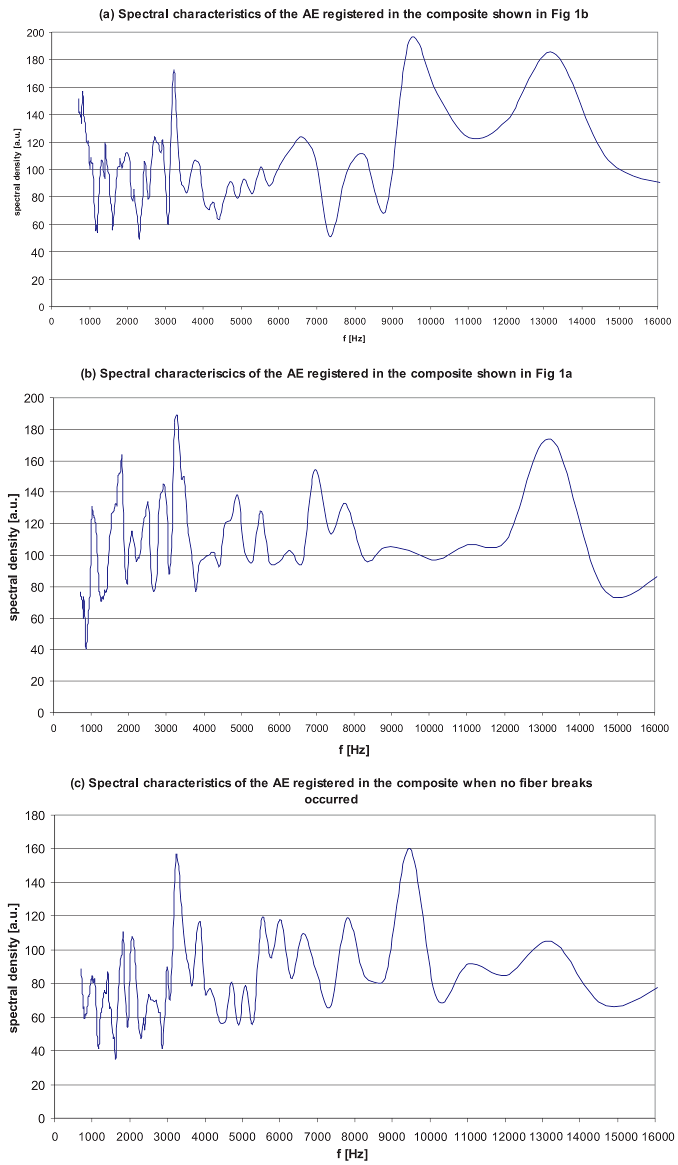

Fig. 8. Preliminary wavelet analysis of a fragment of an EA signal at the initial compression stage of Mg12Li3Al/δ composite with fibres parallel to ND axis: (a) – averaged spectral characteristics of the AE signal registered in the composite shown in Fig. 1b, (b) – averaged spectral characteristics registered in the composite shown in Fig. 1a, (c) – averaged spectral characteristics registered in the composite when no fibre breaks occurred

The wavelet analysis, presented above, is intended to demonstrate the possibilities offered by the AE analyser of new generation, first of all as regards the analysis of single AE pulses, constructing of acoustic maps (acoustograms) and spectral characteristics. It is expected that more and more information about the mechanisms of the processes generating AE will be obtained applying this method.

4. Conclusions

1. Examination of the behaviour of AE in the composites Mg8Li3Al/δ and Mg12Li3Al/δ during compression at ambient temperature have confirmed the occurrence of the effect of the anisotropy of fibres with respect to the compression direction.
2. Higher maximal level of AE in the composites Mg8Li5Al/δ than in the composites Mg8Li3Al/δ is the effect of the increased of the volume fraction of the very acoustically effective phase a with increasing Al content.
3. The reverse effect in Mg12Li3Al/δ and Mg12Li5Al/δ composites can be attributed to blocking of the collective dislocation motion as a result of higher Al contribution to work hardening and to the population of the particles of LiAl phase precipitates.
4. Higher AE level and the occurrence of two ranges of AE activity at 140°C in the composites Mg8Li5Al/δ have confirmed such behaviour, observed earlier in the composites Mg8Li3Al/δ which were explained on the basis of thermal processes of weakening the mechanical strength of the ceramic fibres.
5. The microstructures in the initial state and after deformation indicate unambigously that the microcracks of the fibres occur as a result of the deformation process, and not as a side effect in the technological production process of the composites. The starting microstructure indicate, however, that the fibres do not everywhere adhere closely to the matrix.
6. The results of testing the composites Mg12Li3Al/δ and Mg12Li5Al/δ at 140°C have not been interpreted explicitly because of a high suspection that AE may be disturbed by microcracking of the surface layer of oxides, which is particularly easily formed in composites based on single-phase alloys β.
7. From the comparison of the preliminary results of the measurements of the energy of AE events by means of the AE analyser used so far and the one of new generation it follows that the courses are similar qualitatively, and additionally, they introduce new elements, creating the possibility of a more accurate analysis of the occurrence of AE activity ranges.

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