ARCHIVES OF METALLURGY AND MATERIALS
Volume 51 2006 Issue 2

A. PAWEŁEK*, A. PIĄTKOWSKI*, S. KÚDELA**, Z. JASIEŃSKI*

# ACOUSTIC EMISSION IN Mg-Li-Al ALLOYS AND RELATED COMPOSITES BASED ON DIPHASE $\alpha+\beta$ MATRIX SUBJECTED TO CHANNEL-DIE COMPRESSION AT ELEVATED TEMPERATURE 140°C

# EMISJA AKUSTYCZNA W STOPACH I KOMPOZYTACH Mg-Li-Al NA OSNOWIE DWUFAZOWEJ $\alpha+\beta$ PODDANYCH NIESWOBODNEMU ŚCISKANIU W PODWYŻSZONEJ TEMPERATURZE 140°C

The study is a continuation of the new research direction, started a few years ago in cooperation with the Institute of Materials and Machine Mechanics of the Slovak Academy of Sciences in Bratislava, in which the acoustic emission (AE) method is used to investigate channel-die compressed Mg-Li-Al based alloys and related composites reinforced with short $\delta - Al_2O_3$ ceramic fibres. The aim of the studies is further documentation and explanation of the AE phenomenon in Mg-Li-Al alloys and composites based on $\alpha+\beta$ diphase matrix subjected to channel-die compression at ambient and elevated temperatures 140°C. Attempt has been made to explain the correlations occurring between AE intensity, AE activity and the plastic deformation characteristics as well as the microstructure observed before and after deformation using the optical and the scanning microscopy. The effect of anisotropy of the fibres distribution with respect to the compression direction has been observed in Mg8Li/$\delta$ composites at room temperature, whereas in Mg8Li3Al/$\delta$ composites compressed at elevated temperature 140°C it has been observed that AE activity occurs in two ranges and that AE intensity is higher than that observed at room temperature. The results are discussed on the basis of the dislocation mechanisms of plastic flow and the mechanisms related with the microcracking processes in $\alpha+\beta$ diphase based alloys and composites.

*Keywords*: acoustic emission, Mg-Li-Al alloys, composites with short $Al_2O_3$ fibres, channel-die compression, microstructure, dislocations

Temat stanowi kontynuację, zapoczątkowanego kilka lat temu – w ramach współpracy z Instytutem Materiałów i Mechaniki Maszyn Słowackiej Akademii Nauk w Bratysławie – nowego kierunku badań wykorzystujących metodę emisji akustycznej (EA) w testach nieswobodnego ściskania stopów na bazie Mg-Li-Al oraz kompozytów na osnowie tych stopów wzmocnionych krótkimi włóknami ceramicznymi $\delta - Al_2O_3$. Celem badań jest dalsza dokumentacja i wyjaśnienie zjawiska EA w stopach i kompozytach Mg-Li-Al na osnowie dwufazowej $\alpha+\beta$, poddanych nieswobodnemu ściskaniu w temperaturze otoczenia i podwyższonej 140°C. Podjęto próbę wyjaśnienia korelacji zachodzących pomiędzy przebiegiem intensywności i aktywności EA a charakterystykami deformacji plastycznej oraz mikrostrukturą obserwowaną przed i po odkształceniu przy zastosowaniu mikroskopu optycznego i skaningowego. W kompozytach Mg8Li/$\delta$ zaobserwowano występowanie efektu anizotropii rozkładu włókien względem kierunku ściskania w temperaturze otoczenia. Natomiast w kompozytach Mg8Li3Al ściskanych w podwyższonej temperaturze 140°C zaobserwowano występowanie dwóch zakresów aktywności EA i wyższą intensywność EA niż w temperaturze pokojowej. Wyniki przedyskutowano w oparciu o dyslokacyjne mechanizmy plastycznego płynięcia oraz mechanizmy związane z procesami mikropękania w stopach i kompozytach na osnowie dwufazowej $\alpha+\beta$.

## 1. Introduction

Production of new engineering materials of low density and high mechanical strength for the use in automotive (e.g. housings of motor-car engines), air- and spacecraft (e.g. light computer housings) industry is one of the important topics of the contemporary materials science and engineering. Magnesium-lithium alloys, as the lightest of the known metallic constructional materials (the density in the range of $1300 \div 1600$ kg/m$^3$ is comparable to that of polymers), and particularly the composites based on these alloys reinforced with ceramic fibres – are the very attractive materials for such practical applications.

Mg-Li alloys occur in the form of three different phases. In the range of Li concentration below 4wt.% they present a hexagonal phase of *hcp* structure, whereas the alloys with Li content higher than 12wt.% occur

---

* INSTITUTE OF METALLURGY AND MATERIALS SCIENCES, POLISH ACADEMY OF SCIENCES, 30-059 KRAKÓW, REYMONTA 25
** INSTITUTE OF MATERIALS AND MACHINE MECHANICS, SLOVAK ACADEMY OF SCIENCES, 831 02 BRATISLAVA 3, RAČIANSKA 75, SLOVAKIA

in the form of $\beta$ phase with *bcc* structure. Mechanical properties of $\alpha$ phase are worse than those of $\beta$ phase, which is characterized by very good machinability and weldability [1, 2]. The alloys of Li concentration between 4 and 12wt.% are diphase and occur as a mixture of $\alpha+\beta$ phases. Alloy additions, e.g. from 3 to 5% Al, indeed, slightly increase the alloy or the composite density, but distinctly improve their mechanical strength.

The realized investigations were concerned with the determination of the relations between the acoustic emission (AE) behaviour and the strain mechanisms in diphase alloys and related composites subjected to channel-die compression at ambient and elevated (140°C) temperatures. The temperature 140°C is related with the heating of the housing of moto-car engine. In order to attain this aim the results presented in our earlier papers [3–5], concerned with the investigation of AE behaviour in pure polycrystalline magnesium as well as in single phase alloys and related composites have been used in the discussion. The results obtained in the present work are discussed also on the basis of concepts well-known in literature [6, 7] as well as on the basis of the authors' own suggestions [8] that the main causes of AE in metals are the collective processes of acceleration and surface annihilation of many dislocations.

## 2. Research methods

The Mg-Li based alloys and composites were prepared at the Institute of Materials and Machine Mechanics of the Slovak Academy of Sciences in Bratislava within the scheme of international cooperation. The basic Mg-Li alloys were obtained by casting of raw materials – magnesium (3N purity) and lithium (N5 purity) metals – in a steel crucible at 800°C with subsequent pouring into a cooled steel mould in a chamber of vacuum induction furnace (Balzers) under low argon pressure (1000 Pa, of 99.999% purity) after previous evacuation ($10^{-2}$ Pa). This procedure was similar to that reported in [2].

The composites were fabricated by infiltration of a fibrous preform with metallic melts in the laboratory autoclave by the vacuum-pressure method. The preform containing ~20 vol.% of short $\delta - Al_2O_3$ fibres was prepared by ultrasonic stimulated dispersion of commercial Saffil® RF product. The obtained composites, containing ~10 vol.% of fibres, were characterized by the planar-random distribution, whereas individual fibres had the mean diameter of ~3 $\mu$m and the mean length of ~100 $\mu$m.

The composites samples, in the form cubes with 10 mm edge, were subjected to compression tests in a testing machine INSTRON-6025, equipped with a channel-die, which ensured plastic flow only in the compression axis (ND – normal direction) and in the direction parallel to the channel axis (ED – elongation direction). The plane state of strain was realized in this way since there was no deformation in the direction perpendicular to the channel walls (TD – transverse direction). The traverse speed of the testing machine in each test was always the same and was equal to 0.05 mm/min.

Simultaneously with the registration of the external compressive force F, there was measured the AE parameter in the form of the rate of AE events, $dN_z/dt$. A broad-band piezoelectric sensor enabled to record the acoustic signals in the frequency range from 100 kHz to 1000 kHz. The contact between the sensor and the sample was maintained by means of a steel rail in the channel-die being this way a natural wave-guide. In the compression tests at 140°C there was additionally used a quartz wave-guide of a special form. The number of AE events was recorded usually within the time interval $\Delta t$ = 4s or 6s. The total amplification of the apparatus was 86 dB, and the threshold voltage of the discriminator was in the range 1.17÷1.20 V. In order to minimize the undesired effect of friction against the channel walls, each sample was covered with a Teflon foil.

Moreover, before and after each test, there have been carried out microstructure observations using the standard technique of optical and scanning microscopy. This way, in many cases, besides of both acoustic (time dependence of the rate of AE events) and mechanical (work-hardening curve in the version force-time) characteristics there are presented micrographs which reflect the most essential elements of the microstructure being the result of deformation and/or the microcracking mechanisms. The force-time relationship corresponds, with a rough approximation, to the work-hardening curve in the version stress-strain since the elongation of the sample increases linearly with time due to the constant traverse speed of the testing machine.

## 3. Results and discussion

The starting point for the discussion of the deformation mechanisms in Mg-Li-Al/$\delta$ composites on diphase $\alpha + \beta$ basis, subjected to channel-die compression at elevated temperature 140°C, were the results obtained for pure alloys on diphase $\alpha + \beta$ matrix, i.e. Mg8Li, Mg8Li3Al and Mg8Li5Al alloys and related composites subjected to channel-die compression at room temperature.

### 3.1. Acoustic emission in Mg8Li, Mg8Li3Al and Mg8Li5Al alloys at ambient temperature

Fig.1 shows the graph of the rate of AE events and of the external compressive force as a function of the

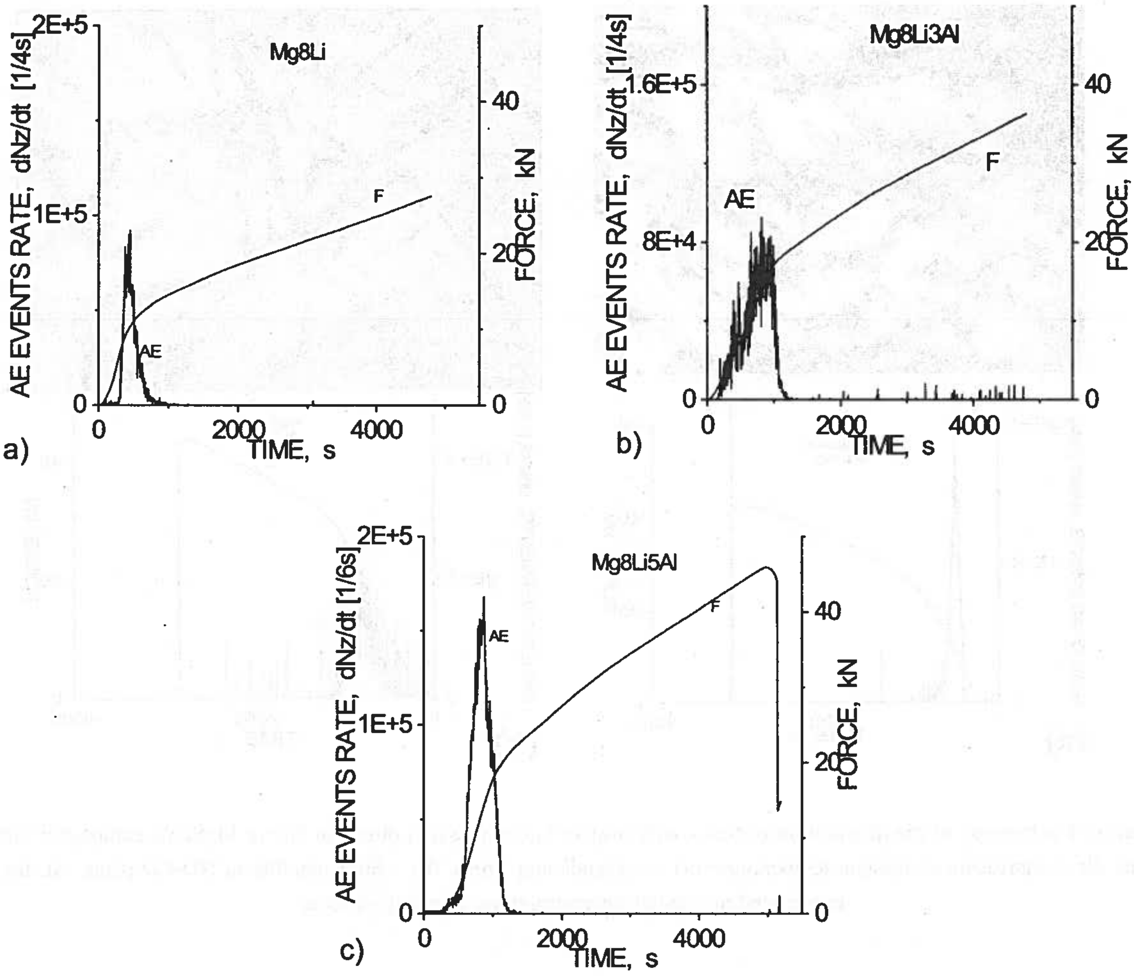

Fig. 1. The course of AE events rate and of the compressing force in alloys on diphase $\alpha+\beta$ basis: (a) – Mg8Li, (b) – Mg8Li3Al and (c) – Mg8Li5Al, subjected to channel-die compression at ambient temperature

duration of the channel-die compression test of Mg8Li alloys.

It is characteristic fact that the maximal level of the AE events rate, not exceeding the value $3\times10^4$/s in the alloy Mg8Li5Al (Fig. 1c), is here almost by one and a half order of magnitude smaller than in the case of single-phase alloys $\alpha$-Mg4Li, where it attains the value of the order $1.25\times10^6$/s [3–5]. This fact has been attributed to a considerable drop of the volume fraction of $\alpha$ phase, acoustically very efficient, at the expense of the increase of the fraction of $\beta$ phase of very poor acoustic efficiency. The poor acoustic efficiency in Mg12Li based single-phase $\beta$ alloys has been documented in the studies [3–5], where it has been demonstrated that the highest mean value of the AE events rate, observed in Mg12Li alloy, were on the level of $10^3$/s. The considerable drop of AE intensity in these alloys is attributed to the very high diffusivity of lithium in $\beta$ phase which considerably limits the possibility of a collective behaviour of the dislocations. Alloy additions 3% Al (Fig. 1b) and 5% Al (Fig. 1c) evidently increase the strength properties of these alloys in comparison with pure alloy Mg8Li (Fig. 1a). However, their possible effect on AE is of less importance here and will not be discussed in detail.

## 3.2. EA in Mg8Li/$\delta$, Mg8Li3Al/$\delta$ and Mg8Li5Al/$\delta$ composites at ambient temperature

AE investigations, carried out last year [4], have shown that in composites based on pure Mg, there occurs the effect of anisotropy of the fibres distribution with respect to the compression direction. However, on the other hand, although the ultrasound measurements have confirmed sufficiently the occurrence of this anisotropy [5], earlier AE investigations in Mg4Li3Al/$\delta$ and Mg4Li5Al/$\delta$ composites based on $\alpha$ phase [3, 4] have not fully confirmed the occurrence of this phenomenon. For this reason the investigations of AE in Mg8Li/$\delta$ composites on diphase $(\alpha+\beta)$ matrix have been

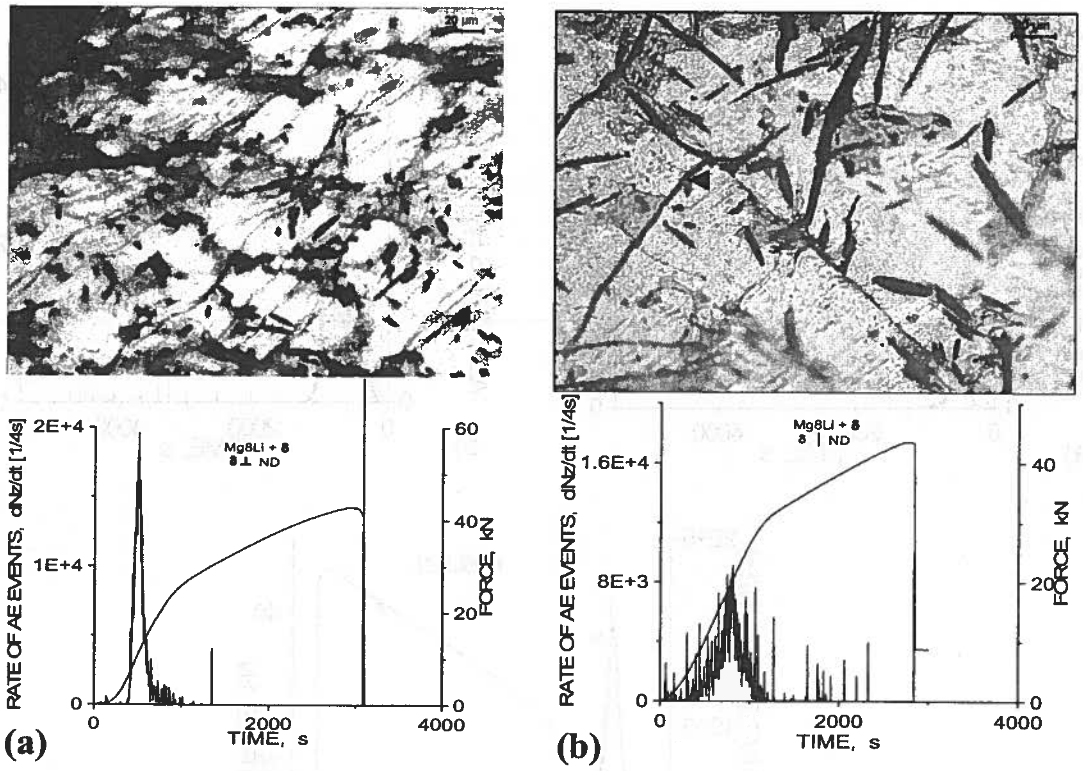

Fig. 2. Effect of anisotropy of the distribution of fibres with respect to compression direction ND in Mg8Li/δ composites subjected to channel-die compression at ambient temperature: (a) – perpendicular fibres, (b) – fibres parallel to ND-ED plane. At the top – corresponding optical microstructures after deformation

carried out also with respect to the phenomenon of the anisotropy of fibres distribution.

Fig. 2 shows the behaviour of AE in Mg8Li/δ composites in which the fibres are situated perpendicular (Fig. 2a) and parallel (Fig. 2b) 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 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 fibres perpendicular to the compression direction. Such behaviour of AE is typical for the effect of fibres anisotropy and it evidently confirms its occurrence. The effect of anisotropy can be attributed to the fact that in case of perpendicular fibres a considerable number of the fibres is parallel (or nearly parallel) or at a small angle to the active slip systems connected with the direction of the maximal shear stresses. In this way the number of effective intersections of dislocations with the fibres, leading to microcracks, generating jump-like AE events, is statistically lower in the same time interval than in the case of a composite with fibres situated in parallel.

Moreover, microcracking of fibres, occurring 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) is much probable in the case of parallel fibres on account of higher values of the angles between the active slip planes and the axes of fibres.

It can be said that the optical microstructure (Fig. 2a, at the top) in the case of fibres perpendicular to ND, is more "point-like", i.e. a greater number of fibres intersect the side plane of observation ND-ED (in cases discussed here and further on it is always the plane of the figure), than in the case of fibres parallel to ND (Fig. 2b, at the top), where a greater number of fibres are parallel to the observation plane ND-ED. The latter optical microstructure suggests that the microcracking of fibres may occur as a result of a dislocation interaction with shear microbands. This suggestion will be later ultimately confirmed by observations using a scanning microscope. Fig. 2 shows additionally that the AE level in composites is at least by one order of magnitude smaller than that in pure alloys (Fig. 1), as a result of the presence of fibres which are obstacle for the dislocation motion and reduce their collective behaviour.

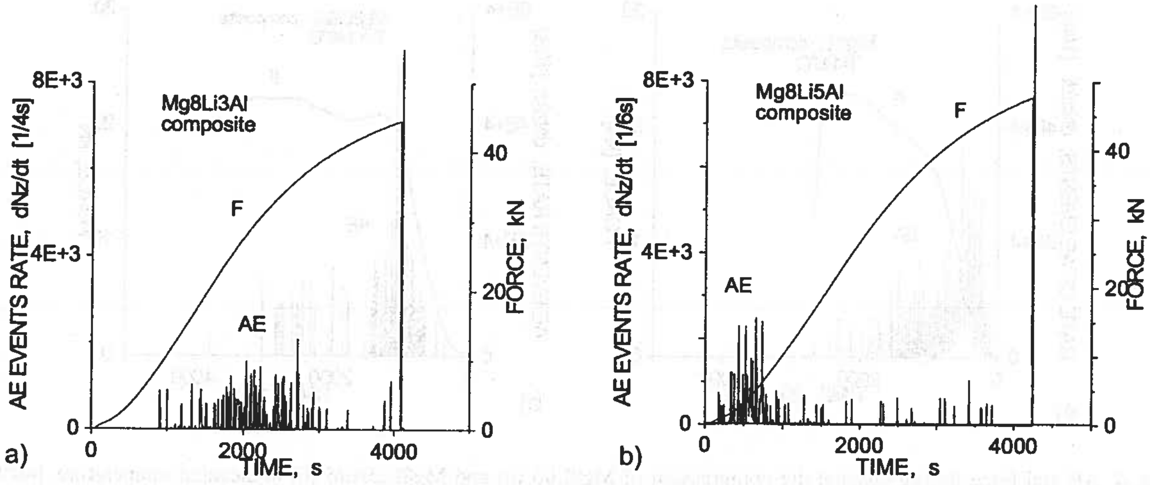

Fig. 3. EA and force during channel-die compression of Mg8Li3Al/$\delta$ (a) and Mg8Li5Al/$\delta$ (b) composites at ambient temperature

It is possible that in the processes of the limitation of the collective dislocation motion also Al participates, which is suggested by the AE graphs in the composites Mg8Li3Al (Fig. 3a) and Mg8Li5Al (Fig. 3b), with parallel location of the fibres. When comparing the AE graphs it can be observed that AE activity is retained almost throughout the whole duration of the test, which is, moreover, characteristic for composites, however it is distinctly smaller in a composite with greater Al content. This is attributed to a smaller population of the precipitation particles of LiAl phase (or even the transition $MgLi_2Al$ phase) in composites with lower Al concentration. The greater population of these particles in Mg8Li5Al composites (Fig. 3b) favours the limitation of the dislocation motion, which also causes a rather small increase of their strength in relation to Mg8Li3Al composites (Fig. 3a).

### 3.3. EA in Mg8Li/$\delta$, Mg8Li3Al/$\delta$ and Mg8Li5Al/$\delta$ composites subjected to channel-die compression at elevated temperature 140°C

An important element in the discussion of AE in composites compressed at the temperature 140°C will be also the results obtained in previous studies [3–5] for pure, polycrystalline magnesium Mg and the corresponding composites Mg/$\delta$, tested at ambient temperature. First of all, as it has been observed in these investigations [3–5], the surprise is the evidently higher AE intensity in the composite Mg/$\delta$ at 140°C than in pure Mg at ambient temperature, which, in turn, is a few time higher than in an identical composite compressed also at ambient temperature. On the other hand, AE activity at 140°C in Mg/$\delta$ composite, similarly as at ambient temperature, is retained almost throughout the whole duration of the test, which is characteristic for these composites. Moreover, the investigation results [3–5] suggested that in these Mg/$\delta$ composites, at the temperature 140°C, there occur at least two ranges of AE activity with a characteristic course of the work-hardening curve. This suggestion is confirmed by Fig. 4, which shows that the two-range character and the long duration of AE activity, remaining at a high level till the end of the test, are particularly visible in Mg8Li/$\delta$ (Fig. 4a) and Mg8Li5Al/$\delta$ (Fig. 4b) composites on diphase ($\alpha + \beta$) matrix and are actually connected with the characteristic deflection of the work-hardening curve.

Fig. 5, in turn, presents the behaviour of AE in Mg8Li3Al/$\delta$ composites with different initial distribution of the fibres. Although a somewhat greater AE activity in the case of parallel fibres can be noticed (Fig. 5b), yet this is not a satisfactory confirmation of the phenomenon of anisotropy. On the other hand, there can be noticed here the occurrence of the second range of AE activity (after about 1500 s). Moreover, when comparing the AE graphs in Fig. 5 and Fig. 4 with the graphs for ambient temperature (Fig. 2), it is seen that AE activity at elevated temperature in diphase based Mg8Li/$\delta$ composites is several times higher – even by an order of magnitude when comparing Fig. 5a with Fig. 2b – than it is observed at room temperature. Thus the unexpected behaviour of AE at elevated temperature 140°C, observed for the first time in Mg/$\delta$ composites [3–5], is

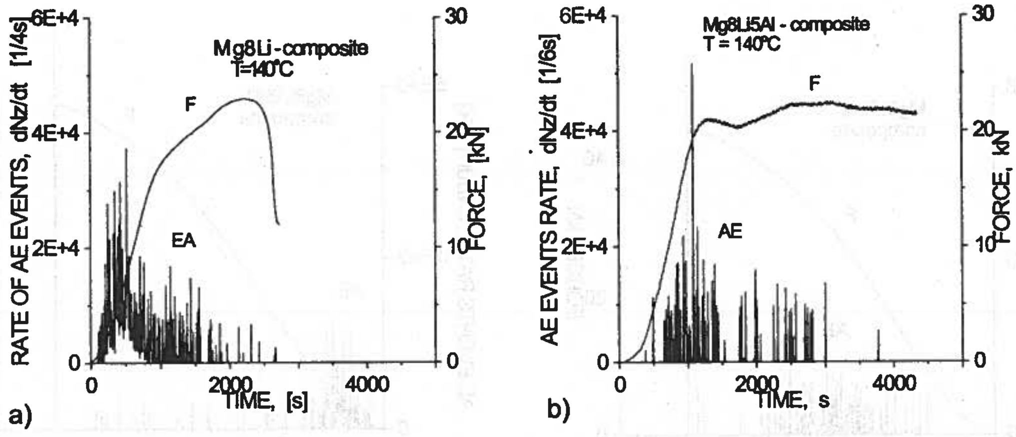

Fig. 4. AE and force during channel-die compression of Mg8Li/δ (a) and Mg8Li5Al/δ (b) at elevated temperature 140°C

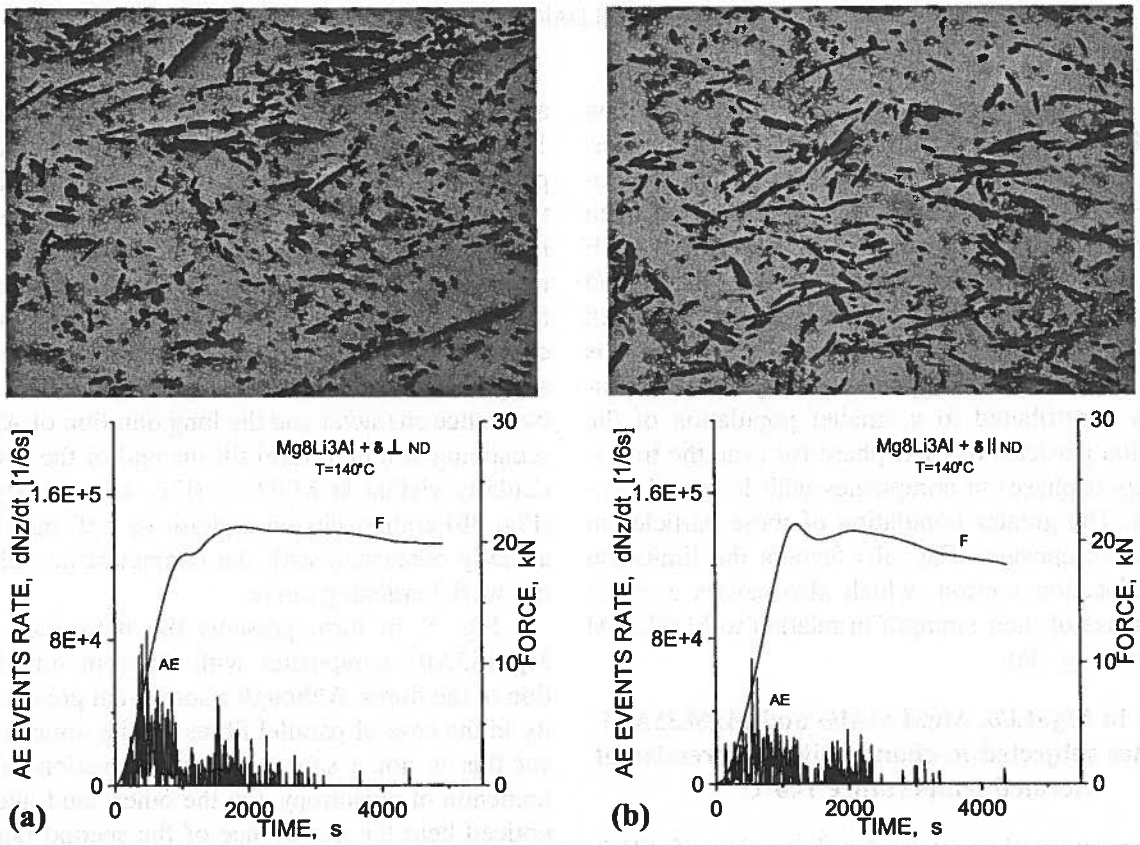

Fig. 5. AE behaviour in Mg8Li3Al/δ composites subjected to channel-die compression at 140°C: (a) – perpendicular fibres, (b) – fibres parallel to the ND-ED plane. At the top: corresponding optical microstructures after deformation

confirmed by the AE behaviour in Mg-Li/δ and Mg-Li-Al/δ composites based on diphase ($\alpha + \beta$) alloys. Hence, there exist premises to assume that the higher AE intensity and the two-range character of AE activity is due to the processes which are so dominating that the anisotropy effect is almost invisible. It is probable that such a process may be the weakening of the mechanical strength of fibres at higher temperature which increases the number of microcracks generating AE events. According to the above concept the second range of AE activity would be connected with the microcracking of "another portion" of fibres whose strength has been reduced after some time since the beginning of the test at 140°C. It should be also added that the optical microstructures after deformation (Fig. 5, at the top), which reveal distinct fragmentation of the fibres, not observed in the initial state, are a strong suggestion that the microcracks of fibres occur during deformation, and are not the result of the technological process of fabrication of the composites. Owing to the application of scanning microscopy this suggestion has been strongly confirmed in another study by the authors [9].

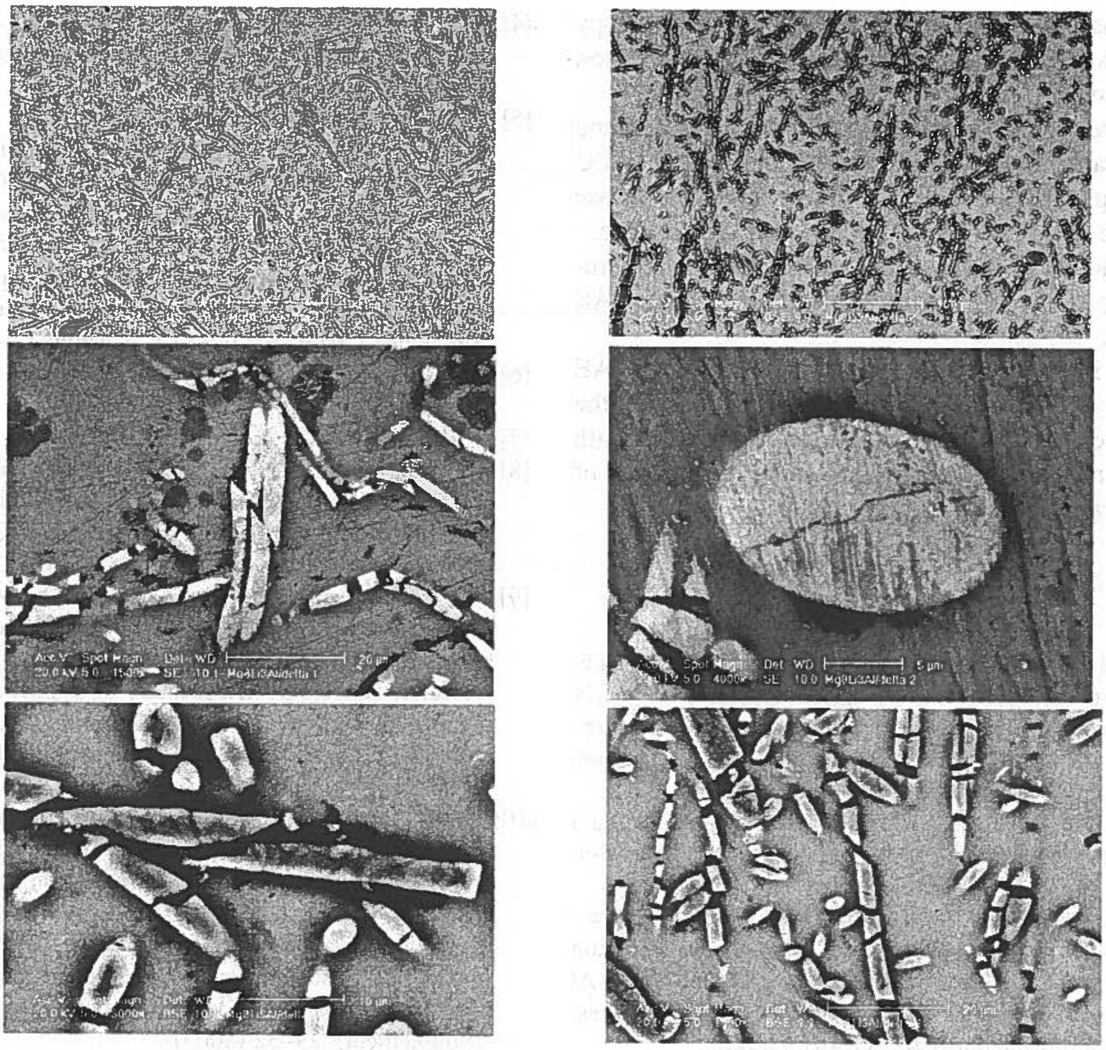

Fig. 6. Scanning microstructures of Mg8Li3Al/δ composites after deformation at 140°C. In the column on the left – fibres situated perpendicular to the compression direction ND, and in column on the right – situated parallel to the ND-ED plane (ND is perpendicular to the marker direction). There are visible the effects of the microcracks of fibres due to dislocation shear (photo in the centre of the left column and at the bottom of the right column) and the effects of debonding (photo in the centre of the right column and at the bottom of the left column)

The remarks concerning the optical microstructures after deformation have been fully confirmed by observations using a scanning microscope (Fig. 6). They have additionally revealed that the microcracks of fibres occur as the effect of a dislocation shear (Fig. 6, on the left, in the centre, and on the right at the bottom), e.g. as a result of relaxation of high internal stresses formed in the front of a dislocation pile-up. Scanning microscope reveal also numerous effects of the decohesion of the fibres from the matrix, the so-called debonding (Fig. 6, on the right, in the centre and at the bottom, on the left at the bottom); this process may greatly contribute to the observed AE signals. Attempts to decide which AE peaks can derive from this process will be possible using an AE analyser of new generation, which is at present installed at the Institute. Some preliminary results are presented in [10]. Similar attempts will be made to test the possibility of the generation of AE from the microcracks forming along phase boundaries or due to the differences in the coefficients of thermal expansion of the ceramic fibres and the metallic matrix.

## 4. Conclusions

1. The maximal level of AE in Mg8Li alloys on the $(\alpha+\beta)$ diphase basis is more than one order of magnitude lower than in Mg4Li alloys on the basis of single $\alpha$ phase. The reason is the fraction of $\beta$ phase, of poor acoustic efficiency due to very high diffusivity of lithium, hampering the collective behaviour of dislocations.
2. AE behaviour in Mg8Li/δ composites based on diphase $(\alpha+\beta)$ matrix tested at ambient temperature

confirms the occurrence of the effect of anisotropy of fibres distribution with respect to the compression direction.

3. The level of AE intensity in Mg/$\delta$ composites and Mg8Li alloy based composites, compressed at 140°C is unexpectedly considerably higher than in the case of these composites tested at ambient temperature.
4. AE behaviour in composites tested at elevated temperature shows the occurrence of two ranges of AE activity.
5. Higher intensity and the two-range character of AE activity at 140°C can be explained on the basis of the processes of weakening of the mechanical strength of ceramic fibres, leading to increased number of microcracks generating AE events.

## REFERENCES

[1] S. Kúdela, Z. Trojanová, V. Kolenčiak, P. Lucáč, Short fibre reinforced Mg-8Li-xAl matrix composites - preparation, structure and properties. Proc. of the Int. Conf. Advances in Composites, Bangalore, 679–686, 24–26 August 2000.

[2] S. Kamado, Y. Kojima, Deformability and strengthening of superlight Mg-Li alloys, Met. Sci. Techn. **16**, 45-54 (1998).

[3] A. Pawełek, Z. Jasieński, A. Piątkowski, S. Kúdela, A. Litwora, Acoustic emission during channel-die compression of Mg-Li and Mg-Li-Al alloys and composites reinforced by short $\delta Al_2O_3$ fibres, Archives of Metallurgy **47**, 175–183 (2002).

[4] A. Pawełek, Annual Reports 2001–2003 of the Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Kraków.

[5] A. Pawełek, Z. Jasieński, S. Kudela, A. Piątkowski, P. Ranachowski, F. Rejmund, Acoustic Emission in Channel-Die Compressed Mg-Li-Al Alloys Reinforced with Short Ceramic Fibres, pp. 225–228, and Influence of $\beta$ Phase on Mechanical and Acoustic Behaviour of Mg-Li-Al Alloys, pp.229–234, Proc of the Int. Conf. on Advanced Metallic Materials, Smolenice Castle, SLOVAKIA, November 5–7, 2003.

[6] V.S. Boiko, R.I. Garber, L.F. Krivenko, Fiz. Tverd. Tela **16**, 1233–1235 (1974).

[7] A. Vinogradov, Scripta Mater. **39**, 797–805 (1998).

[8] A. Pawełek, A. Piątkowski, Z. Jasieński, S. Pilecki, Acoustic Emission and Strain Localization in FCC Single Crystals Compressed in Channel-Die at Low Temperature. Z. Metallkde **92**, 376–381 (2001).

[9] A. Pawełek, Z. Ranachowski, A. Piątkowski, S. Kúdela, Z. Jasieński, Acoustic emission and strain mechanisms during compression at elevated temperature of $\beta$ phase containing Mg-Li-Al composites reinforced with ceramic fibres, submitted to Archives of Metallurgy and Materials, 2006.

[10] Z. Ranachowski, A. Pawełek, A. Piątkowski, Wavelet analysis of acoustic emission signals generated in composites based on $\beta$-Mg12Li3Al alloys subjected to compression tests, Abstracts of the 52th Open Seminar on Acoustics OSA 05, Wągrowiec, 11th – 15th September, 2005, Archives of Acoustics **30**, 3, 382–383 (2005); Archives of Acoustics **30**, 4 (Supplement), 29–32 (2005).

*Received: 10 November 2005.*