Rev.Adv.Mater.Sci. 18(2008) 583-589

MECHANICAL AND ACOUSTIC EMISSION BEHAVIOUR INDUCED BY CHANNEL-DIE COMPRESSION OF Mg-Li NANOCRYSTALLINE ALLOYS OBTAINED BY ECAP TECHNIQUE

J. Kuśnierz[1], A. Pawełek[1], Z. Ranachowski[2], A. Piątkowski[1], Z. Jasieński[1], S. Kúdela[3] and S. Kúdela, Jr.[3]

[1]Aleksander Krupkowski Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Reymonta 25, 30-059 Kraków, Poland
[2]Institute of Fundamental Technological Research, Polish Academy of Sciences, Świętokrzyska 21, 00-049 Warszawa, Poland
[3]Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Račianska 75, 831 02 Bratislava, Slovakia

Received: March 29, 2008

Abstract. In the paper there are presented the results of investigations of the relations between the plastic strain mechanisms and the acoustic emission (AE) induced channel-die compression of magnesium and Mg8Li and Mg12Li alloys of ultra fine-grained (UFG) (nanocrystalline) structure, obtained using the technique of equal channel angular pressing (ECAP). The results are compared with those obtained for similar materials not subjected to ECAP operation. Moreover, the results obtained using of a new generation AE analyser are compared with those obtained earlier using an AE analyser of older generation. The spectral analysis of the detected AE signals has been carried out and the AE behaviour discussed in the context of both the dislocation strain and the superplastic flow mechanisms in UFG materials.

1. INTRODUCTION

The magnesium-lithium based alloys, being the lightest of the known construction materials, especially composites on the basis of these alloys, reinforced with ceramic fibres – are very attractive materials from the point of view of practical application, e.g. in the automobile industry as casings of the engines. Mg-Li alloys may occur in the form of three different phases. In Li concentration up to 4 wt.% there occurs the hexagonal phase α with A3 lattice (*hcp*). Alloys containing more than 12 wt.% appear in the form of β phase with regular A2 lattice (*bcc*). The mechanical properties of a phase are worse than those of β phase which is characterized by good machinability and weldability [1]. Alloys containing from 4 wt.% to 12 wt.% of Li are diphase alloys and they occur as a mixture of the phases α+β. Alloys additions, e.g. 3÷5% Al increase slightly the density of the alloy or of the composite, but improve their strength.

In the last decade more and more attention has been given to obtaining nanocrystalline materials on account of their excellent mechanical properties, such as great strength and plasticity or even superplasticity occurring under conditions of relatively moderate temperatures [2].

The ECAP (Equal Channel Angular Pressing) technique is one of the method of producing mate-

Corresponding author: J. Kusnierz, e-mail: nmkusnie@imim-pan.krakow.pl

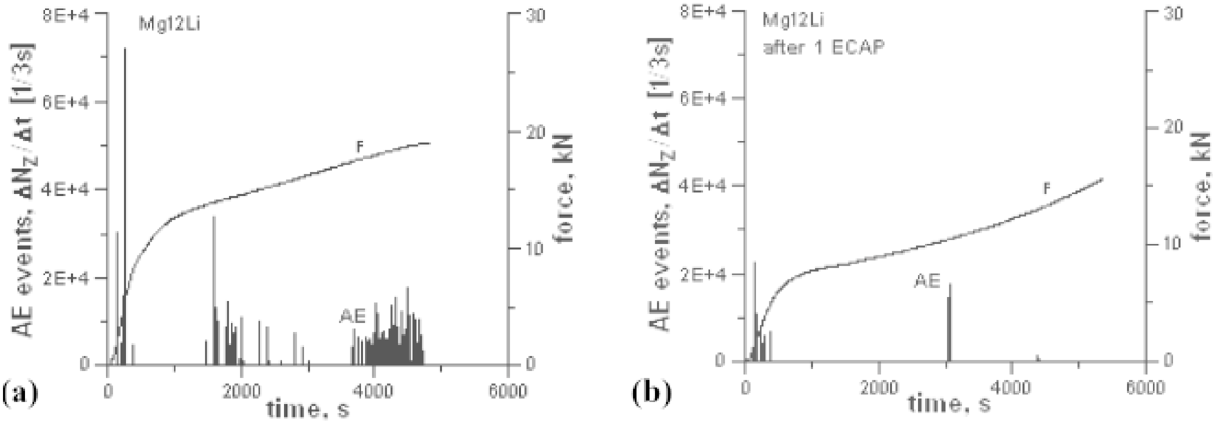

Fig. 1. AE in Mg12Li alloy before (a) and after fourfold (b) ECAP operation.

rials of nanocrystalline structure. The ECAP method offers the possibility to obtain a solid material of relatively big dimensions and free from porosity, which distinguishes it from the traditional methods, such as rolling and drawing. Investigations carried out at the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences (IMIM PAN) within the range of obtaining nanocrystalline materials by means of ECAP technique [3-5] have found response in the world literature. The obtained size of the grains was of the order from 100 nm to 1000 nm.

The aim of the present study is the initiation of a new direction of research in which, for the first time, the AE method is applied in channel-die compression tests of nanocrystalline materials obtained by means of ECAP technique. This concerns especially the documentation and the determination and explanation of the relation between the AE phenomenon and the mechanisms of plastic and superplastic deformation in ultra fine-grained (UFG) Mg-Li and Mg-Li-Al alloys subjected to channel-die compression tests. First attempt have also been made to determine the conditions which must be satisfied so that after angular pressing of composites based on these alloys there will be obtained materials of UFG structure.

The aim of preliminary AE investigations carried out by means of an AE analyser of a new generation was to compare them with the results obtained by means of an analyser used until now, in which the computer measurement system has been modified. A new program was tested which enables to carry out the spectral analysis of AE signals including the wavelet analysis of single AE signals and to construct acoustic maps (acoustograms) and spectral characteristics, i.e. spectral densities as a function of frequency. In future it is planned to carry out the wavelet analysis in the case of compressing nanocrystalline composites obtained by means of ECAP operations. The theoretical basis of the wavelet analysis in a shortened version has been discussed in earlier works [6,7], where as a preliminary step it has been applied in the case of compressing composites not subjected to ECAP operation.

2. INVESTIGATION METHODS

Alloys and composites based on Mg-Li alloy prepared in cooperation with the Institute of Materials and Machine Mechanics of the Slovak Academy of Sciences in Bratislava. The basic mg-Li alloys were obtained by the method of induction melting of magnesium of 99.99% purity and lithium of 99.5% purity. The composites were produced from a preliminary material – a fibrous skeleton, obtained from commercial Saffil – subjected to infiltration process in a bath of liquid alloy in a laboratory autoclave [1]. Samples of alloys and composites to be used in the ECAP tests had the form of rectangular prisms with square basis and 10 mm edge, and the height of 30-40 mm, while the samples intended for compression tests had the shape of

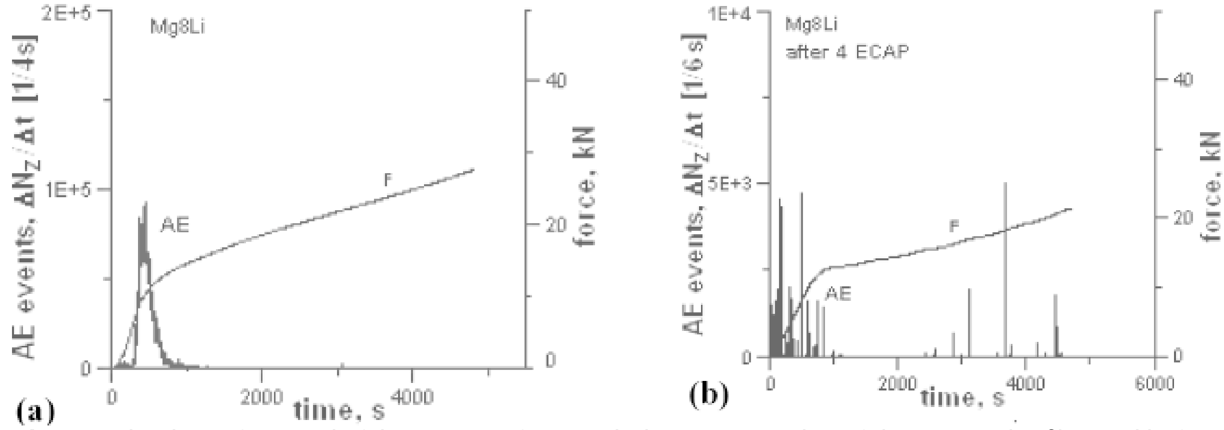

Fig. 2. Behaviour of AE and of the external force F during compression of dual alloys (α+β)Mg8Li before (a) and after fourfold (b) ECAP operation.

cubes with the edge not exceeding 10 mm. ECAP operation and the channel-die compression tests were realized using the INSTRON-6025 tensile testing machine. The displacement velocity of the machine in the compression tests was 0.05 mm/min, and in the ECAP tests it was 10 mm/min.

Simultaneously with the registration of the external force F, the AE parameter was measured as the events rate $\Delta N_z/\Delta t$. 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 and the sample was maintained by means of a steel plate in the channel-die. In each compression test the number of AE events was recorded within the time interval Δt = 4 s or Δt = 6 s. The total amplification of the acoustic signal was 86 dB, and the threshold voltage of the discriminator was 1.20 V. In order to eliminate the effects of friction each sample was covered with a Teflon foil.

Using an AE analyser of the new generation it is possible to register the elementary, single AE events with simultaneous recording of their duration, amplitude and the root mean square (RMS) of the AE voltage signal, and as a consequence to determine the number of events and the number of AE counts (amplitudes) both as a function of energy or the threshold voltage of the discriminator as well as a function of the duration of the deformation test. Moreover, before applying and after carrying out the last, fourth ECAP operation, there were carried out microstructural investigations by means of an optical microscope for approximate estimation of the degree of refinement of the microstructure.

3. INVESTIGATION RESULTS AND DISCUSSION

Fig. 1 shows the results of the first, preliminary test of the application of the AE method in the compression tests of single phase alloys (β)Mg12Li before (Fig. 1a) and after four successive pressing (Fig. 1b) by the ECAP method. It can be seen that AE after ECAP operation is clearly decreasing, and the decrease of force (e.g. after about 5000 s) from the level of about 20 kN (Fig. 1a) to the level of about 15 kN in the alloy after ECAP operation (Fig. 1b) is evident indication of increased plasticity.

Similar tendencies are observed also in the results of compressive tests of dual alloys (α+β)Mg8Li. Fig. 2 shows the behaviour of AE and of the external force *F* during compression of these alloys before (Fig. 2a) and after fourfold (Fig. 2b) ECAP operation. It can be noticed, moreover, that the graphs of AE and of force in (β)Mg12Li alloy after ECAP operation (Fig. 1b) are additionally characterized by the occurrence of fairly distinct correlations between the AE peaks and rather small, almost regular kinks on the graph of force. The optical microstructures before (Fig. 3 on the left) and after (Fig. 3 on the right) show that after fourfold ECAP operation there take place distinct re-

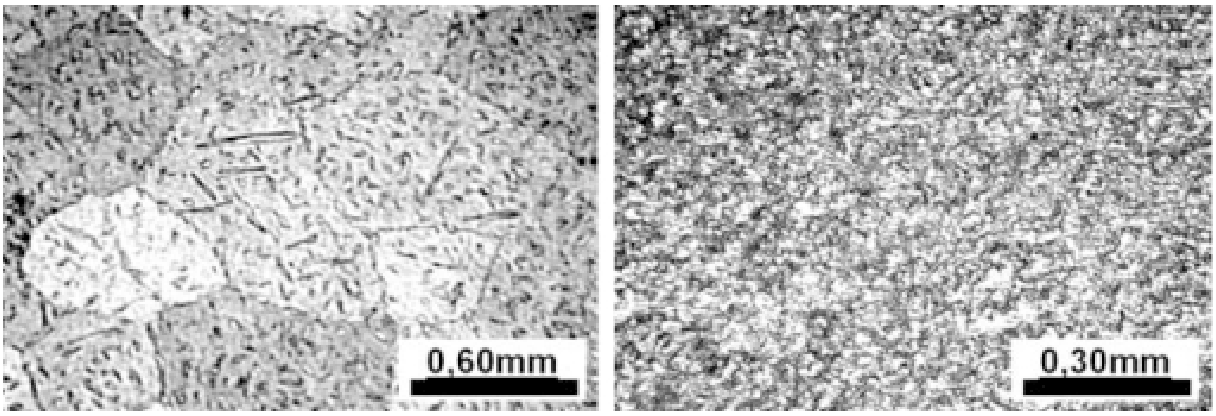

Fig. 3. Optical microstructures of Mg8Li alloy before (on the left) and after fourfold (on the right) ECAP operation.

finement of the structure: from the grain size of the order of about some hundreds of micrometers in the initial state (Fig. 3 on the left) to the size of the order of a few, ten to twenty micrometers in the state after ECAP operation (Fig. 3 on the right).

Moreover, there was also undertaken the first attempt at angular channel pressing of a composite based on Mg8Li alloy. However, obtaining material with refined microstructure at ambient temperature has been found impossible. The graph of the external force as a function of the punch displacement, not shown here (see e.g. [8]), indicated that the composite, already in the initial stage of pressing, is subjected to strong cracking. In order to obtain appropriate microstructure it is necessary to select and to apply other parameters of the ECAP operation.

Below there are presented and discussed by way of example some preliminary results obtained by means of the new AE analyser. This concerns mainly the value of RMS and the rate of counting Ä*N*/Ä*t*. The analyser used till now did not allow to measure these quantities. It has been found that e.g. in the case of Mg8Li alloy, the RMS values, not quoted here (see [8]), for a sample after ECAP operation are lightly, but distinctly smaller than the RMS values for the initial state. Such result should be interpreted in favour of the earlier mentioned suggestion that in the state of increased plasticity, i.e. with increasing tendency to superplastic flow – AE shows the tendency to drop, similarly as in the case presented in Fig. 2.

On the other hand, Fig. 4 shows the dependence of the number of events as a function of the duration of the compression test for Mg12Li alloy before and after fourfold ECAP operation. It is clearly seen that the AE level is evidently higher in the state after ECAP operation than in the initial state, thus contrary to the case presented in Fig. 1. It is probably connected with different conditions of extrusion in the angular channel. Figure 1b refers to alloy obtained at room temperature, and Fig. 4b – to alloy extruded at the temperature 70 °C.

However, it seems that the graphs in Fig. 5 may be found more interesting, as they present the number of registered AE events as a function of the threshold voltage of the discriminator for Mg8Li (Fig. 5a) and Mg12Li (Fig. 5b) alloys, both before and after a single and a fourfold ECAP operation. Already a preliminary analysis of these dependencies allows to state that the

ECAP process causes a change in the distribution of the energy of the measured AE events, which is more distinctly visible in the case of Mg8Li alloy (Fig. 5a) than for Mg12Li alloy (Fig. 5b). It can be seen that after fourfold ECAP process the number of events is higher and the number of fractions of AE events is considerably increasing, which may be evidence of increased refinement of the microstructure as well as of the appearance of new deformation mechanisms, which thereby determine the operation of new AE sources generating the AE events of greater energy than in the case of samples not subjected to the ECAP process, or

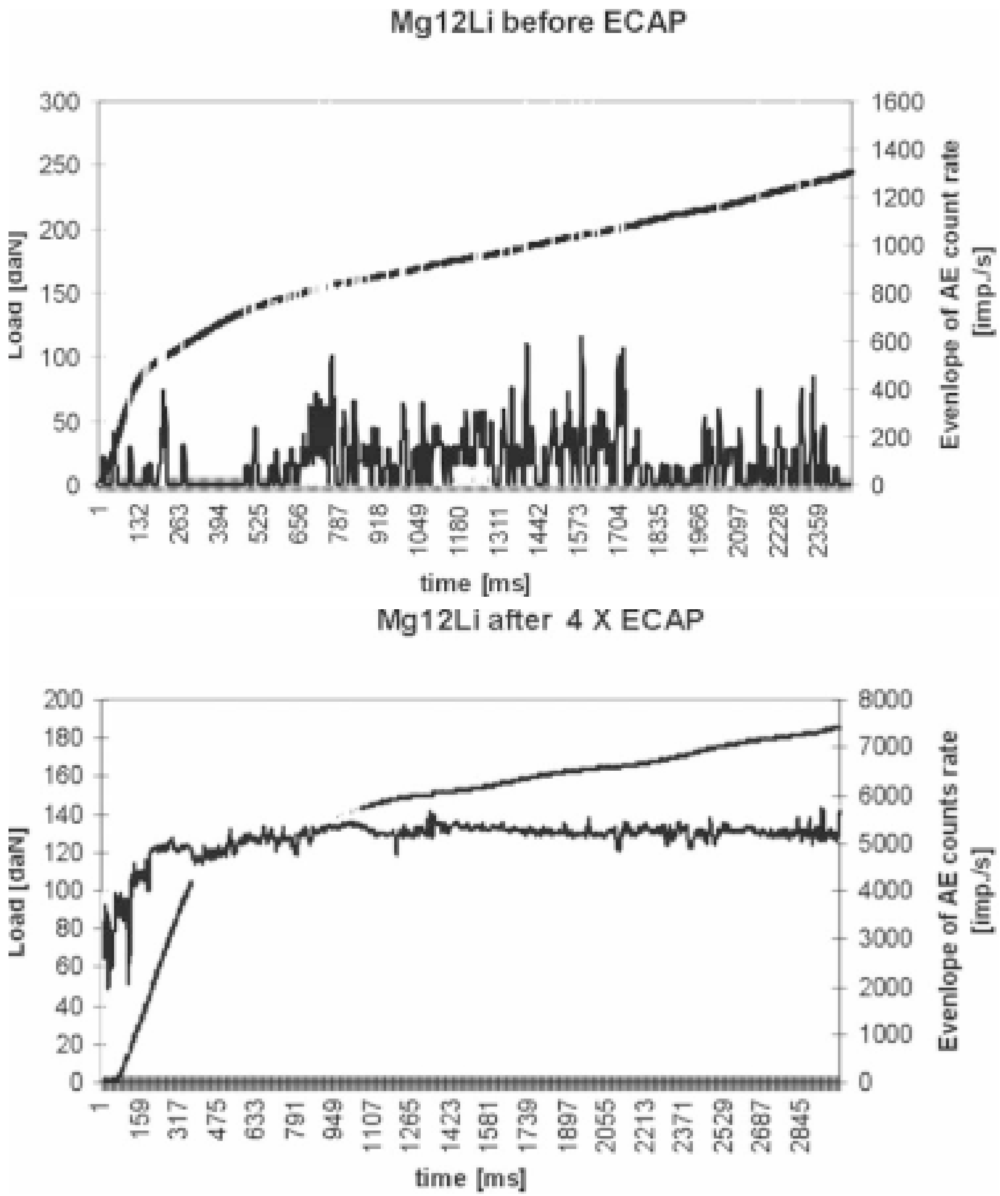

Fig. 4. AE behaviour in Mg12Li alloys before (a) and after (b) ECAP operation.

even samples after a single ECAP process. At this stage of investigations it is difficult to interpret definitely the above relations. It is necessary to obtain more information which means the continuation of systematic investigations of this subject.

When summing up the discussion it must be said that the application of AE method in compression tests of Mg-Li alloys obtained by the ECAP technique has a pioneer character. The first report from the year 1998 [2] on the subject of AE generation during plastic deformation concerned the tension test of UFG copper of 200 nm grain size. The mechanisms of plastic deformation in nanocrystalline metals have not been definitely explained until now, although it is believed [2] that the factors controlling the mechanical properties and affecting AE are the strain localization at the mesoscopic level and the slip along the grain

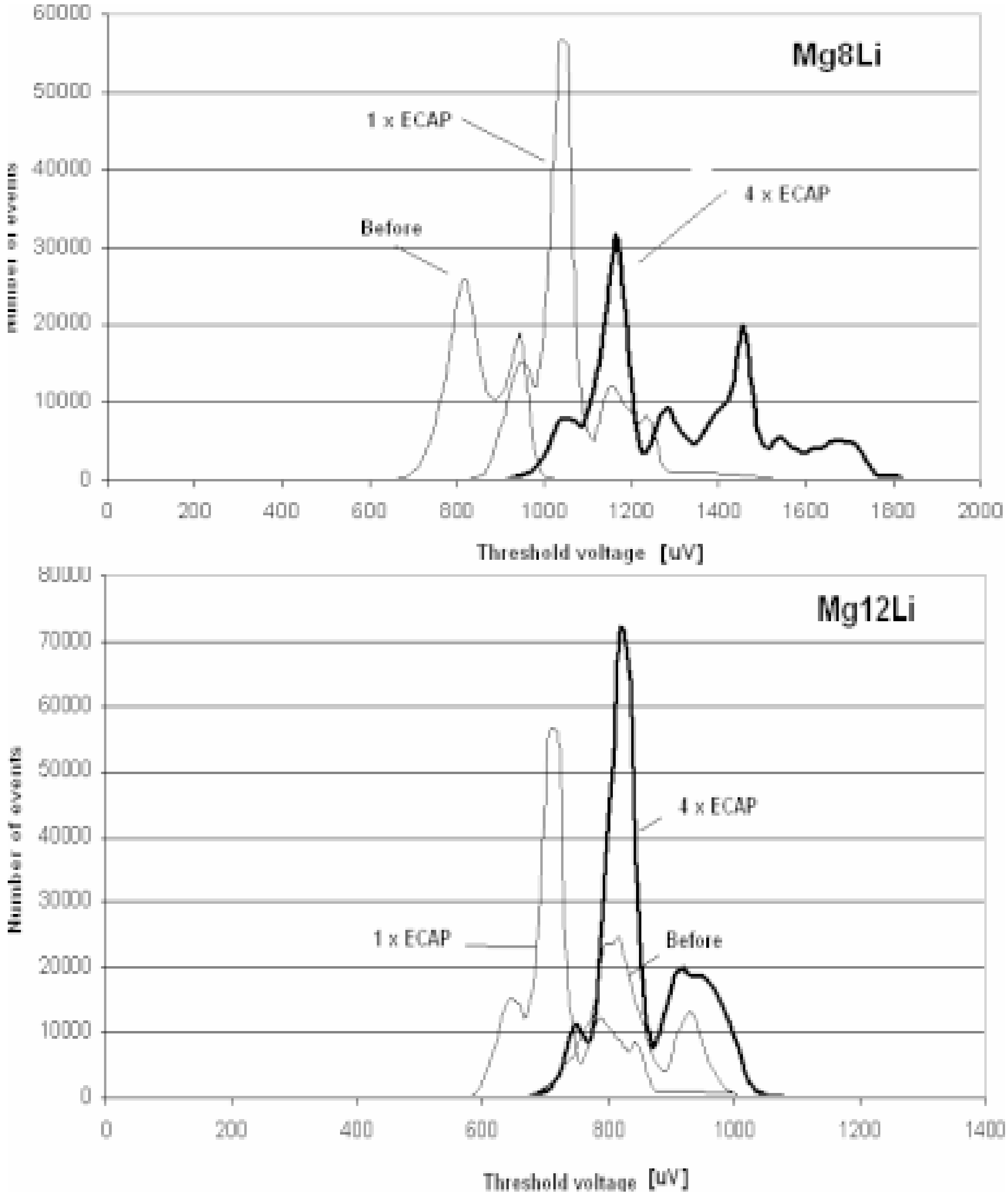

Fig. 5. Dependence of the number of AE events as a function of the threshold voltage of the discriminator for Mg8Li (a) and Mg12Li (b) alloys before, after a single and after fourfold ECAP operation.

boundaries, depending on their specific state. It is emphasized that for their further recognition the analysis of single AE events may be helpful. Thus, in the investigations of strain localization in nanocrystalline materials the new AE technique, which supplies information about the structural-dislocation processes occurring in the real time within very small amount of the material, may be helpful.

It is expected that in future the methods of spectral analysis will become a general, very helpful instrument in the discussion of the relation between the phenomenon of acoustic emission and the phenomenon of superplastic flow in nanocrystalline materials, especially in ultra fine-grained Mg-Li and Mg-Li-Al alloys, as well as composites on the basis of these alloys, reinforced with short and long ceramic fibres.

ACKNOWLEDGEMENTS

The above study was financially supported from the research project of the Committee for Scientific Research (KBN): grant No 3 T08A 032 28 and partly also from grant No N507 056 31/1289.

REFERENCES

[1] S. Kúdela, Z. Trojanová, V. Kolenčiak and P. Lucáč, In: *Proc. of the Int. Conf. Advances in Composites* (Bangalore, 24-26 August 2000), p. 679.

[2] A. Vinogradov // *Scripta Mater*. **39** (1998) 797.

[3] J. Kuśnierz // *Archives of Metallurgy* **46** (2001) 375.

[4] J. Kuśnierz, M.H. Mathon, T. Baudin, Z. Jasieński and R. Penelle // *Mat. Scie. Forum* **408-412** (2002) 703.

[5] J. Dutkiewicz, J. Kuśnierz and T.G. Nieh // *Mat. Sci. Forum* **357-359** (2001) 111.

[6] Z. Ranachowski, A. Pawełek and A. Piątkowski // *Abstract - Archives of Acoustics* **30** (2005) 382; ibid. **30** (2005) 29.

[7] A. Pawełek, Z. Ranachowski and A. Piątkowski // *Archives of Metallurgy and Materials* (2005) in press.

[8] A. Pawełek, *Annual Report of the Institute of Metallurgy and Materials Science* (Polish Academy of Sciences, Kraków, 2005).