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MATERIALS DEGRADATION RESEARCH ON THE BASIS OF MECHANOACOUSTIC AND MICROSCOPIC METHODS

MECHANOAKUSTYCZNA I MIKROSKOPOWA METODA BADANIA DEGRADACJI MATERIAŁÓW

The paper presents the concept of mechanoacoustic testing of degradation processes of ceramic electrotechnical materials. Samples of small dimensions are subjected to slowly increasing compressive loading with simultaneous recording of acoustic emission descriptors. The process is continued to the destruction or is stopped at various stages of degradation of the material structure. Microscopic analysis of samples enables determining the effects of stress action.
Keywords: acoustic emission (AE), material degradation, electrotechnical ceramics

W pracy została przedstawiona koncepcja mechaniczno-akustycznych badań procesów degradacji ceramicznych tworzyw elektrotechnicznych. Małogabarytowe próbki poddawane są wolno narastającemu obciążeniu ściskającemu z jednoczesną rejestracją deskryptorów emisji akustycznej. Proces prowadzi się do zniszczenia lub zatrzymuje na różnych etapach degradacji struktury. Analiza mikroskopowa próbek pozwala określić efekty oddziaływania naprężeń.

1. Introduction

At present ceramic materials, especially electrotechnical porcelains, are widely applied to fabricate reliable electroinsulating elements. Porcelain is used to produce line, post and traction insulators as well as hollow insulators of high parameters [1-3]. For these products, besides high mechanical strength, a long period of exploitation without breakdown is required. For several years there has been observed increasing tendency of production aluminous porcelain of high mechanical strength (C 130 type). This is result not only of the present requirements concerning the short-term mechanical strength of the electroinsulating elements. This material is suitable to guarantee the reliability of power supply, which is determined by the durability, i.e. by the long-term mechanical strength of the ceramic material. Considered problem is closely connected with resistance to degradation processes – formation and development of ageing effects in the structure of ceramic body.

The essence of ageing degradation is gradual expansion of already existing microcracks and formation of the new ones under the influence of the mechanical, structural and thermal stresses, occurring in the material body. They consist of the internal stresses and stresses induced by the external factors – working load [4,5]. The internal stresses are induced during technological production processes, in particular formation and deairing as well as in the last stage of firing – cooling. Then significant mechanical stresses are formed: on micro scale – on the grain boundaries of quartz and the glassy matrix; on semi-macro scale – resulting from textural anisotropy as well as stresses on the macro scale between the internal and the external areas of the insulator rod - induced by the temperature gradient at cooling. Only compressive stresses on the boundary between ceramic body and glaze are intentional and increase the strength of the element. An insulator in operation is subjected to considerable exploitation static stresses, as well as additionally – especially dangerous – dynamic loads deriving from the cable vibrations. These stresses, when added to the intrinsic ones, accelerate the ageing processes. An additional factor contributing to the propagation of microcracks are the temperature changes in the body, attaining within 24 hours even as much as 45 °C. In case of the insulators of the older generation, long lasting periods of severe frost had a particularly destructive influence on the material. They were responsible for sudden in-

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crease of failures on domestic power lines during the sharp winter of 1986/87 [4].

The most important factor responsible for gradual degradation of the parameters of electrotechnical ceramic materials, are the local stresses occurring at the grains and the interfacial boundaries as well as the alien inclusions. The surface defects, which have essential influence on the strength of the samples, after correctly realized glazing are no longer important. Internal stresses in the micro-areas are located in the brittle medium. The only way of their relaxation is an increase of already existing or initiation of the new microcracks. Relaxation of stresses is thus connected with gradual decrease of mechanical strength of the material. However, the insulator in operation is under constant external load. As a consequence, the progressive development of microcracks causes gradual reduction of the cross-section area of the rod, which actually keeps up this stress. Thus in the material under load there are present the internal stresses inducing constant increase of microcracks. Development of microcracks causes the degradation of the parameters of the material in process of time. From reports concerning an older type insulator porcelain, it is known that about 35 years long period of exploitation causes over 30 % decrease of the mean mechanical strength of insulating material. Besides that, the dispersion of the strength of the exploited insulators is about 2.5 times greater than that of the new elements [6]. In instance of ceramic insulators, degradation of the mechanical and electric parameters is of great importance because it decreases reliability of power supply.

Experience obtained during exploitation of older type insulators, made of aluminous porcelain (C 120 type), has revealed a relatively quick development of the ageing processes [4-6]. This refers to the objects being in operation for some decades of years on domestic lines and power stations. Progress of ageing processes in electroporcelain structure has been also confirmed by foreign publications [6,7]. The factor which has essential influence on the degradation of the material of older type, in the process of time, is high content of quartz, exceeding 20 %. This component, present often in the form of large grains, causes serious internal stresses in the porcelain body. Quartz phase sometimes shows also weak joint with precipitates of needle-shaped mullite. An additional problem is the dispersion of the properties resulting from insufficient repeatability of parameters of technological processes, which was observed still in the nineteen-eighties.

For aluminous material C 130 kind there is not enough experience obtained during a longer period of exploitation of the products. Although production of this porcelain in the domestic industry began in the year 1979 (material denoted E-15), it became widely applied only in the nineteen-nineties [8]. This was one of the main reasons to develop a method that in short-term laboratory test make it possible to get results similar to the long lasting degradation effects. As it has been shown by the initial investigation carried out on the material C 120, obtained processes have a character close to the effects of ageing degradation. The similar structural effects of operational load acting on the C 120 porcelain material, in case of many years long exploitation of insulators, and of the compressive stresses in a laboratory tests of relatively short duration, has been observed [9]. This paper presents the concept of mechanoacoustic testing of degradation processes of ceramic electrotechnical materials.

2. Idea of mechanoacoustic method

The method of acoustic emission (AE) is a valuable scientific tool when used for monitoring internal structural changes in ceramic materials. This technique allows to obtain numerous data concerning the dynamic processes occurring during change of mechanical, thermal or thermo-mechanical stresses in the materials. The initiation and growth of microcracks is one of the main sources of the acoustic events in brittle bodies, which include ceramic materials. It should be emphasized that AE signals appear already at the threshold stresses, when the generation of microcracks in the material cannot be in practice detected by other methods [10].

Basis of used method is examination of samples, which are subjected to mechanoacoustic measurements using the technique of acoustic emission on a special two-channel measuring system. Specimens of small dimensions are submitted to slowly increasing compressive stress. Geometry of samples has significant influence on obtained results. Surface of specimens should be free from defects, which can initiate cracks development. Top and bottom surfaces, being acted by compressive force, ought to be plane and parallel to each other. If this condition is not satisfied enough, there can occur local fracture and splitting off corners and even wall of the sample. There is performed simultaneous registration of the force, and in consequence acting stress in one channel, along with AE descriptors in the second one. This investigation enables recording and description correlation between the increasing external load and the processes of structure degradation. Changes of the material structure are mainly connected with formation and growth of microcracks, which is reflected in the acoustic activity. In consequence, acoustic method is effective for the investigation of the destruction of brittle materials, where growth of microcracks belongs to the main sources of

AE signals. Examination of different ceramic materials enabled to state that the sum of AE events during the loading period is a good descriptor of the intensity of the processes of cracking, and in consequence degradation of the material. There exists a correlation between the rate of the increase of cracks and the rate of AE events (number of AE events per unit of time) [11].

Measuring set applied in examination of the ceramic materials was specially designed and constructed. The most important feature of this system, was that it has been composed of two independent channels. The mechanical channel contained testing machine INSTRON 3382, controlled by the computer. Stressed sample was placed on specially prepared base of elongated shape. This element was made of hardened steel and functioned additionally as a waveguide of acoustic signals. The range of the velocities of the traverse of the machine, possible to apply, was from 10^{-3} to 10^{1} mm/min. Taking into account geometry of the specimens, acting force was converted into stress. Parallel to the measurement of the load acting on the sample, AE descriptors were recorded. The acoustic registration path contained a broad band transducer WD PAC type, placed on the steel base of the testing machine. The passband of transducer ranged from 80 to 1000 kHz. The transducer was connected to standardized AE analyser and a computer registering acoustic data. Amplification in acoustic channel was equal to 60 dB, one second time interval of summing up the signals was applied. This choice was due to the fact that the force increase registration was performed every one second. Sampling frequency of AE signal equaled 44.1 kHz. Final analysis of registered AE signals was carried out using standard software made in Institue of Fundamental Technological Research PAS in Warsaw [12]. The measuring set is presented in figure 1.

Velocity of stress growth is important factor affecting results of the mechanoacoustic tests. The AE descriptors of appearing signals are not a linear function of changes of the mechanical or thermal stresses. The velocity of these changes is one of the factors influencing the acoustic activity. This dependence is additionally difficult to define quantitatively. The measurement of AE descriptors, at very slow increase of mechanical load, of the order of 10^{-2} mm/min allows, however, to make the mechanoacoustic tests almost independent of the influence of the experimental factors. At very low velocity of stress increase the process of structure degradation has quasi-static character, which better reflects operational conditions, when ceramic element is under working load. Velocity of stress changes has essential influence on the structure degradation processes.

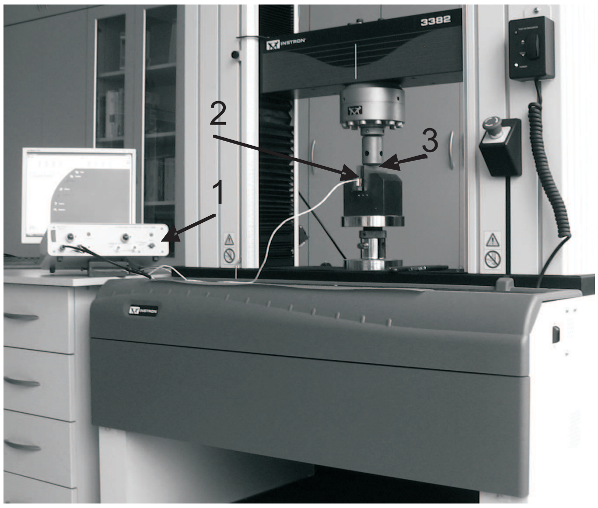

Fig. 1. Two-channel measuring system applied to mechanoacoustic tests of the ceramic samples. There are marked: 1 – AE analyser, 2 – acoustic transducer, 3 – enclosure containing the sample

Higher velocities, of the order of 10^{0} mm/min, favour initiation and growth of transglanular cracks. While lower velocities, of the order of 10^{-2} mm/min favour interglanular cracks increase. In the structure of the aged ceramic material of the objects, especially insulators after long period of exploitation, there are observed mainly interglanular cracks in the matrix [5,6,9]. This fact confirms necessity of application of lower velocities of stress growth in degradation processes investigation. In case of short-term mechanical strength high velocity can be used to obtain proper result. Moreover, the velocity of loading increase has important influence on mechanical strength of tested samples and to smaller extend on dispersion of the results – table 1 [13]. This is the result of different mechanisms of structure degradation at different experimental conditions. During investigation of degradation effects in ceramic material there was used the displacement velocity of traverse of Instron machine equal to 0.02 mm/min. This value allowed effective performance of the mechanoacoustic tests of sufficiently numerous groups of samples. Part of them was loaded up to destruction, while in case of specimens for microscopic study – increase of stress was stopped at different levels of structure degradation.

TABLE 1

Results of measurements of mechanical strength of porcelain samples during three-point bending tests for various velocities of stress growth [13]

Velocity of stress growth [mm/min]	0.001	0.01	0.1	1
Number of samples	20	20	20	20
Range of stress value [MPa]	79.2 ÷ 121.6	80.4 ÷ 148.5	100.4 ÷ 150.2	102.3 ÷ 150.9
Average strength value [MPa]	104.6	111.7	124.0	127.4
Standard deviation [MPa]	10.6	22.6	16.1	12.6
Relative dispersion [%]	40.5	61.0	40.2	38.1

Relative dispersion = 100 % $(\text{value}_{max}\text{-value}_{min})/\text{value}_{average}$

Descriptors of acoustic emission are registered parallel to mechanical parameters in the second channel of measuring set. In ceramic materials, as typically brittle, recorded acoustic emission is mainly associated with the growth of already existing or initiation of the new microcracks. Therefore, the starting point of technical applications of acoustic emission method was a statement of the fact, that each microcrack generates a AE pulse defined as the term "event" [14]. Assuming that the ceramic material is macroscopically homogeneous, it can be supposed that the elongation of the every microcrack is roughly equal, and thus the number of AE events N is proportional to the elongation of the crack a, and hence [15]:

$$\frac{da}{dt} = B\frac{dN}{dt}. \tag{1}$$

There should be taken into account only the pulses of energy exceeding a certain threshold value, which is defined as discrimination level. The number of events during the examination period is called the sum of AE events. Number of events per time unit is called the events rate The event consists of the group of signals and is usually characterized by an envelope of amplitudes on the output of acoustic emission sensor. For the sample of finite dimensions, it is also necessary to take into account the secondary signals, originating from multiple reflections of the waves inside the sample. Then the AE event should be treated as statistical phenomenon. Measurements of acoustic emission have always a comparative nature, because they concern a particular object which is the subject of research. Obtaining of the absolute value of energy of AE event is practically impossible [16]. Acoustic emission analyzers, besides determining of the signals amplitude, are equipped with functional blocks calculating the energy of measured signals or the energy-related functions. The most commonly used solution is electronic conversion of a group of measured signals amplitudes $V(t)$ into the effective value V_{RMS} over the time period T, according to the formula:

$$V_{RMS} = \sqrt{\frac{1}{T}\int_0^T V^2(t)dt}, \tag{2}$$

with energy of AE signal proportional to the square of V_{RMS}.

The essential factor in AE measurements is to use such descriptors of signals that contain the most relevant information for the evaluation of the tested process [17]. The choice of the optimal descriptor for the definite measurement is determined by the following criteria:

– linear dependence between descriptor and measured physical parameter in time;
– maximal sensitivity of descriptor concerning changes of measured parameter;
– good repeatability of measurements – small dispersion of results during testing high number of samples of the same material (the lowest standard deviation);
– the lowest influence of external conditions such as e.g. discrimination level of AE signals;
– minimizing of influence caused by the signals of the background.

Besides the above criteria, there exist additional, very important factor. Acoustic emission descriptor, selected at the research should be optimal for tested material. As it was stated experimentally by the authors, choice of AE descriptor should be different in case of aluminosilicate materials and oxide ceramics. Aluminosilicate materials such as porcelain, steatite or cordierite, are characterized by numerous signals of lower energy. These signals can be effectively registered using AE events rate. It is specially important during preliminary and subcritical stages of structure degradation. During registration descriptors based on energy of signals, even at low discrimination level, substantial part of them can be lost. Illustration of this effect are the mechanoacustic characteristics of the samples of aluminous porcelain, cut off from long-rod insulator for 400 kV power lines. In figures 2 and 3 there are presented recorded courses of the rate of AE events and of the effective value of signal (RMS) for the same sample.

Another example, where the choice of AE descriptor enables more detailed analysis of the material structure degradation mechanics, may be the results obtained during mechanoacoustic investigation of corundum ceramics. Measurements have shown a large difference between the AE signal energy registered for various stages of degradation. The first - preliminary stage of degradation process is characterized by occurrence of numerous, but very weak and short in duration intergranular cracks. Acoustic effects of this process are the large number of signals of small-amplitude, both for recording AE events and RMS descriptors. Therefore, detailed analysis of the effects of the initial phase is difficult. These descriptors illustrate well the strong and long-term effects of destruction, particularly the critical stage. For the analysis of subcritical stage and in particular the preliminary one, the most useful is AE event energy. This parameter represents well the energy of particular events, which correspond to weak intergranular cracking of microseconds duration. Recording of AE event energy enables a more detailed analysis of all stages of sample degradation, and presentation of all three stages in one diagram. Mechanoacoustic courses of the AE event energy and the rate of events for the same corundum sample are presented in figures 4 and 5.

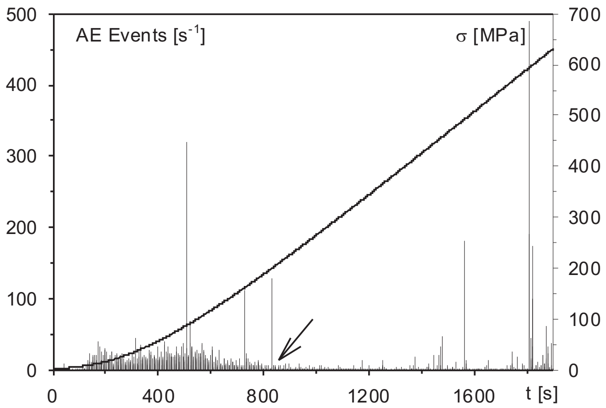

Fig. 2. Typical course of the rate of AE events versus increase of compressive stress for the porcelain sample of the strength 635 MPa. Displayed are only preliminary and subcritical stages of degradation in the stress range equaled 0 ÷ 631 MPa. Arrow points out approximate boundary between stages

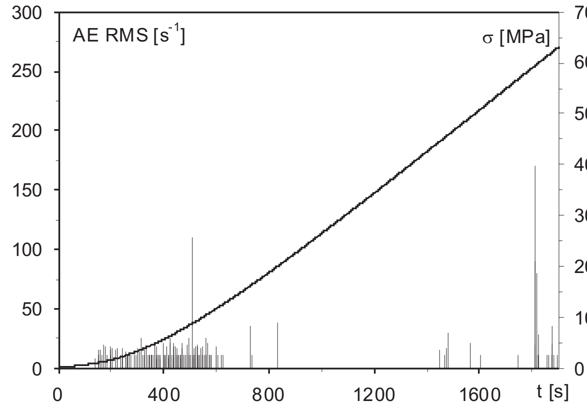

Fig. 3. Course of the rate of AE RMS versus compressive stress for the same porcelain sample as in figure 2. Important part of signals is lost because of low energy at preliminary and subcritical stages

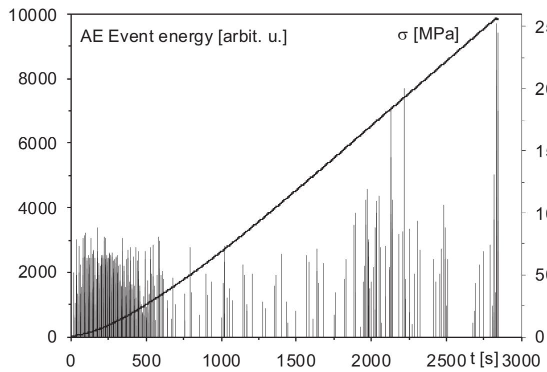

Fig. 4. Typical course of the rate of the AE event energy versus compressive stress for corundum sample of the strength 2572 MPa. The whole range of degradation process is presented

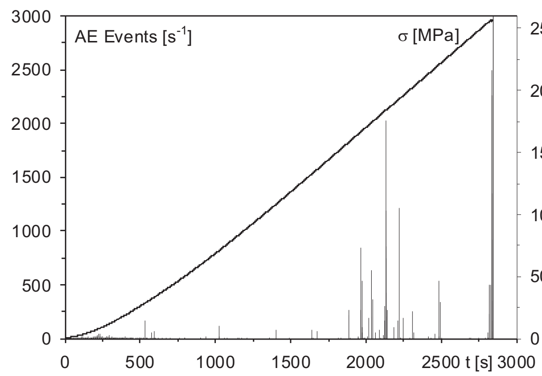

Fig. 5. Course of the rate of AE events versus compressive stress for the same corundum sample as in figure 4. Mechanics of degradation process is represented not as properly as by the AE event energy

3. Preparation of ceramic samples for microscopic study

Microscopic techniques are among the most important methods for research of ceramic materials – aluminosilicate, oxide and non-oxide. This concerns to both – materials without structural defects, containing faults, as well as samples taken from the objects after different period of work, such as insulators. In the latter case, the material is for a long time exposed to various kinds of destructive forces, there is required special procedure of sample surface preparation. It is anticipated that operational stresses can leave visible effects in the material structure. In early studies, it is difficult to assess the nature and scale of the structure degradation. As a consequence there is required special care in the treatment of sample at each stage of preparation and the study. Used procedures should not change the true image of the effects of degradation.

Microscopic analysis of ceramic materials, including the quantity and spatial distribution of particular phases and pores as well as the presence of all kinds of heterogeneities and defects, requires careful preparation of the observed surfaces. The modern method of polishing is defined as mechanochemical process. Surface microroughness obtained using this method is at the level of nanometers. Role of mechanical polishing factor performs colloidal solution of silica (SiO_2) with particle size of not more than 50 nm. As a chemically active agent is usually used sodium chlorate (NaClO). Used abrasive slurry has reaction pH equal to 13,5. The process is performed on special polyurethane pads with high porosity. The rate of polishing of ceramic materials is about 1 μm/min. Process of mechanochemical polishing depends on large reduction of forces of chemical bonds in the area of several atomic layers, and next mechanical removal of weak bounded layer. By using this polishing technique, obtained surfaces, even of highly aged ceramic materials, are very little changed (distorted). The delicate treatment during mechanochemical removal of successive layers of material enables also disclose even subtle effects of ageing process. For aluminosilicate materials, traditional grinding and polishing on diamond abrasive powders is not used now. After careful cut with saw containing internal diamond edge, a defected layer has only about 100 μm. Then, without grinding, using directly mechanochemical polishing, layer of about 200 μm is removed. Although this method is modern and considerably limits introduction of additional surface defects, in case of samples tested using mechanoacoustic method has turned out to be insufficient. During mechanochemical polishing, elements of structure weakly integrated with the matrix, undergo crushing out. A good example are particles of porcelain cullet, which are not melted in the process of firing (sintering) and are weakly connected to the glassy matrix. The other example are grains of quartz. Particles of cullet most often can not be observed on surfaces prepared using mechanochemical method. Dark fields, remaining after crushed out particles of cullet and quartz grains, occupy on average 7.0 % of the area – Figure 6. The exact analysis of images of porcelain, especially after application of compressive stress, requires a different method of preparing the sample surface.

In connection with described difficulties, special epoxy resin (e.g. Struers company production) is poured over tested samples. The two-stage process of polishing is performed next. The top layer of resin is removed in the beginning. The first coarse stage of polishing is performed on the hard polishing disc, using silica suspension with grain sizes ranging from 10 to 5 μm. The second final stage is conducted on a soft polishing cloth. Grains in suspension have sizes from 5 to 0.25 μm. Only use this procedure enables avoiding the introduction of additional defects and minimizes the effect of crushing out. Microscopic analysis of such prepared surfaces of samples showed the presence of cullet in the porcelain structure, which has not been detected after mechanochemical polishing. As a result quantity of crushed out elements of the structure has been reduced by over 40 %. Particles of cullet, remaining after polishing, constitute 2.6 % of the surface area. Losses after crushed out elements of the structure occupy on average 4.1 %, including 1.5 % of cullet, which connection with the matrix is very weak. Majority of lost elements have size less than 5 μm. The smallest are fractions of micrometer in size, the largest rarely exceed 20 μm. The average size is a little over 4 μm. The total content of cullet in the material constitutes about 4 %. Figure 7 presents the image of porcelain surface, prepared using the resin. All phases of the porcelain material are well visible.

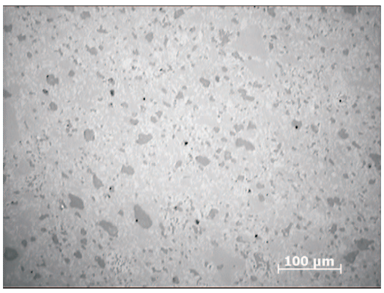

Fig. 6. The image of aluminous porcelain structure, in magnification 200 times, prepared using mechanochemical procedure. Losses after polishing covering crushed out elements of structure are shown as dark fields. They include on average 7.0 % – complete content of cullet – about 4 % and part of quartz phase. Fine black pores constitute negligible part of surface

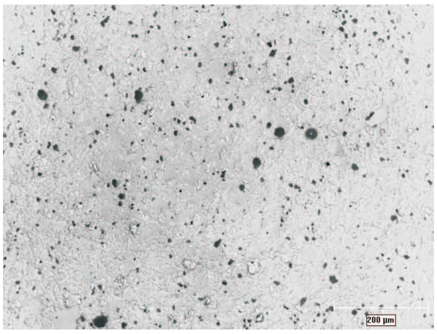

Fig. 7. The image presents the same porcelain material, prepared by the epoxy resin method, in magnification 100 times. Particles of cullet, remaining in the structure constitute 2.6 % of surface. Total content of cullet is about 4 %. Losses after crushed out particles of cullet and quartz grains occupy on average 4.1 %

4. Example of application mechanoacoustic method for plastic alloys

Mechanoacoustic method was used not only for brittle materials like ceramics. Important application is investigation of deformation processes in magnesium-lithium based alloys having plastic properties [18]. The Mg-Li based alloys and composites, reinforced with ceramic fibres, belong to the lightest construction materials and are very attractive from the point of view of practical application. To obtain nanocrystalline structure of Mg-Li based alloys, which have better properties, ECAP (Equal Channel Angular Pressing) technique is used. This method offers the possibility to obtain UFG (ultra fine-grained) materials of relatively big dimensions and free from porosity. Size of grains after application of ECAP method can be diminished to the order from 1000 nm to even 100 nm. Investigation of the mechanism of plastic deformation in UFG alloys was performed using mechanoacoustic method. The compression tests were realized in the channel-die with displacement velocity of the machine equal to 0.05 mm/min. Simultaneously with the registration of the external force, the AE events rate was recorded, within the time interval ranged from 3 s to 6 s. The total amplification of the acoustic signal was 86 dB and the threshold voltage of the discriminator was 1.20 V. As an example there were compared results obtained for the typical alloy sample – not subjected to ECAP operation and after fourfold ECAP process. In figure 8 there is presented behaviour of AE (events rate) versus external force during compression of Mg12Li alloy before (a) and after fourfold (b) ECAP operation.

As the result of application ECAP process the size of alloy grains underwent considerable decreasing. From the order of some hundreds of micrometers in the initial state, to the size of the order of a few micrometers [18]. Diminishing the dimensions of grains in the material influenced mechanoacoustic characteristics of tested sample. Number of AE signals is reduced. Three distinct ranges of acoustic activity, appearing before ECAP (Fig. 8a), are not observed. Only first – initial stage is still present. The other effect concerns plastic properties of Mg8Li alloy. Yield point occurs at considerably lower force and character of slope of the curve of the force increase is different too. That indicates growth of plasticity as a result of lowering of grains dimensions. Similar investigations were performed for MgLi and MgLiAl alloys of various compositions as well as composites based on these alloys [18].

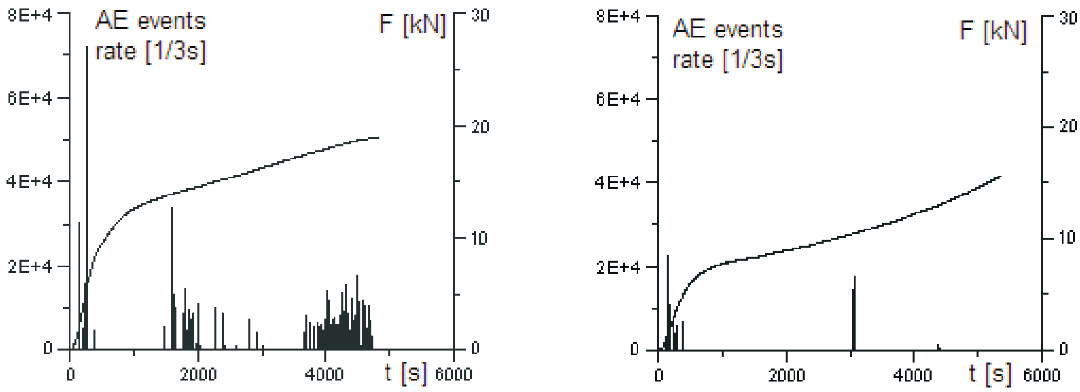

Fig. 8. Course of AE events versus force during channel-die compression of Mg12Li alloy sample before (a-left) and after fourfold (b-right) ECAP operation [18]

5. Concluding remarks

The paper presents mechanoacoustic method of examination of the degradation effects in ceramic materials as well as alloys and composites based on them. Application AE method during slowly increasing compressive stress and comparative microscopic analysis enable recognition of successive stages of the structure degradation. Worked out method makes possible comparison of mechanical and acoustic properties, microstructure, texture, homogeneity and resistance to degradation of different, especially ceramics, materials. On the basis of performed investigation on electrotechnical materials, it can be supposed that there exists similarity between effects of long-term exploitation under working load and material degradation during relatively short laboratory tests. Sequence of degradation effects, concerning mechanics and components of structure, specified during mechanoacoustic examination, occurs for the period of long lasting operation. The required conditions to obtain above similarity are – very slow, quasistatic growth of loading and sensitive monitoring changes of structure by properly chosen AE descriptor. Such measurements should be carried out using specially constructed two channel experimental set-up.

Description of degradation stages and its mechanics requires using the most effective AE descriptor. During compression tests, aluminosilicate ceramics e.g. porcelain, generate numerous signals of relatively low energy. They can be effectively registered using events rate. Application of RMS , based on energy of AE signals can result in lost a lot of them. In case of oxide materials, e.g. corundum, which are the source of AE signals of comparatively high energy, recording of RMS is more effective and better reflects mechanics of degradation process.

Microscopic analysis of samples structure must be performed using special procedures which do not change image of the structure degradation. It is particularly important in case of materials, which were exposed to long lasting working load and compressive stresses. Even modern and mild mechanochemical method of surface preparation turned out insufficient for samples after mechanoacoustic tests. Only application epoxy resin technique enables to avoid introduction of new defects of structure. Then it is possible microscopic analysis of the material containing strongly damaged elements of structure.

Results of mechanoacoustic measurements of different ceramics materials, obtained up to now, enable distinguishing three stages of structure degradation. The preliminary and subcritical ones show low or moderate intensity of AE signals and considerable variety for the particular samples. The first stage is connected with development of defects of low threshold energy, initiated during any technological operations. The subcritical stage is closely connected with the inhomogeneity of the samples structure in micro and semi-macro scales. The last – critical stage, directly precedes decohesion of the samples. It is narrow and is followed by very strong continuous activity.

Besides ceramics, mechanoacoustic method is applied to investigate plastic materials, such as light metals alloys and composites based on these alloys. On the basis of obtained courses of AE activity and force growth, there can be compared plastic properties of the material structure before and after intensive deformation (ECAP operation).

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