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Foundations of mechanoacoustic method of material degradation research

***Abstract**. The paper presents the concept of mechanoacoustic testing of degradation processes of ceramic electrotechnical materials. Samples of small dimensions were subjected to slowly increasing compressive loading with simultaneous recording of acoustic emission descriptors. The process was continued to the destruction or it was stopped at various stages of degradation of the material structure. Microscopic analysis of samples enabled determining the effects of stress action.*

***Streszczenie.** W pracy została przedstawiona koncepcja mechaniczno-akustycznych badań procesów degradacji ceramicznych tworzyw elektrotechnicznych. Małogabarytowe próbki poddawane były wolno narastającemu obciążeniu ściskającemu z jednoczesną rejestracją deskryptorów emisji akustycznej. Proces prowadzono do zniszczenia lub zatrzymania na różnych etapach degradacji struktury. Analiza mikroskopowa próbek pozwoliła określić efekty oddziaływania naprężeń. **(Podstawy mechanoakustycznej metody badań degradacji materiałów)**.*

Keywords: acoustic emission (AE), material degradation, electrotechnical ceramics.
Słowa kluczowe: emisja akustyczna, degradacja materiału, ceramika elektrotechniczna.

Introduction

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. For these products, besides high mechanical strength, a long period of exploitation without breakdown is required. Increasing tendency for production of aluminous porcelain of high mechanical strength (C 130 type) has been observed for several years. This is the 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. The problem considered is closely connected with the resistance to degradation processes - appearance and development of ageing effects in the structure of ceramic body.

The ageing degradation consists in a gradual expansion of already existing microcracks and the formation of new ones under the influence of mechanical, structural and thermal stresses, occurring in the material. They contain internal stresses and the stresses induced by external factors like a working load [1]. The internal stresses are induced during technological production processes, in particular, in the formation and deairing as well as in the last stage of firing, which is cooling. Then significant mechanical stresses appear: in the micro scale – on the grain boundaries of quartz and the glassy matrix; in the semi-macro scale – resulting from textural anisotropy as well as in the macro scale - between the internal and the external areas of the insulator rod - induced by the temperature gradient at cooling. An insulator in operation is subjected to considerable exploitation static stresses, and additionally, which is especially dangerous, to 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 is the temperature changes in the material, attaining within 24 hours even as much as 45 ^{0}C. In case of the insulators of older generation, long lasting periods of severe frost had a particularly destructive influence on the material. They were responsible for sudden increase of failures of domestic power lines during the hard winter of 1986/87.

The local stresses occurring at the grains and the interfacial boundaries as well as the alien inclusions are the most important factors responsible for a gradual degradation of the parameters of electrotechnical ceramic materials. Internal stresses which induce constant increase of microcracks number are present in the material under load. The development of microcracks causes the degradation of the parameters of the material in time. From reports on an older type of 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 new elements [2,3]. In the case of ceramic insulators, the degradation of the mechanical and electric parameters is of great importance, because as a consequence, the reliability of power supply decreases.

The experience gained during the exploitation of older type insulators, produced of aluminous porcelain (C 120 type), has revealed a relatively quick development of ageing processes [1,2,3]. 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 [3,4]. It was found that the high content of quartz, exceeding 20 %, had the essential influence on the degradation of C 120 material. For aluminous material C 130 kind there is not enough experience obtained during longer period of exploitation of the products. This was one of the main reasons to develop a method which in a short-term laboratory test could obtain degradation effects typical for long lasting exploitation. The similar structural effects of operational load acting on the C 120 porcelain material, in the case of long-term exploitation of insulators and compressive stresses in laboratory tests of relatively short duration, have been observed in [5]. This paper presents the concept of mechanoacoustic testing of degradation processes of ceramic electrotechnical materials.

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 obtaining numerous data concerning the dynamic processes, which occur during change of mechanical, thermal or thermo-mechanical stresses in 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 with other methods [6].

The basis of used method is examination of samples which consists in mechanical-acoustic measurements using the technique of acoustic emission by a special two-channel measuring system. Specimens of small dimensions are submitted to slowly increasing compressive stress. The geometry of samples has a significant influence on the obtained results. The surface of specimens should be free from defects, which can initiate the development of cracks. The top and bottom surfaces exposed to compressive forces, ought to be plane and parallel to each other. If this condition is not satisfied properly, a local fracture and splitting up corners or even the walls of the sample may occur. A simultaneous registration of the force and in consequence acting stress in one channel, along with AE descriptors in the second one is performed. Such an 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 the formation and growth of microcracks, which is reflected in the acoustic activity. In consequence, the acoustic method is effective for the investigation of the destruction of brittle materials, in which growth of microcracks belongs to the main sources of AE signals. The examination of different ceramic materials showed that the sum of AE events during the loading period is a good descriptor of the intensity of the cracking processes and in consequence – the degradation of the material. A correlation between the rate of the increase of cracks and the rate of AE events (number of AE events per unit of time) was reported in [7].

The measuring device applied in the examination of ceramic materials was specially designed and constructed. It has been composed of two independent channels, which was the most important feature of the system. The mechanical channel contained testing machine INSTRON 3382, controlled by the computer. The stressed sample was placed on a specially prepared base of elongated shape. This element was produced of hardened steel and functioned additionally as a waveguide of acoustic signals. The range of the velocities of the traverse of the machine was from 10^{-3} to 10^{1} mm/min. Taking into account geometry of the specimens, the acting force was converted into stress. AE descriptors were recorded simultaneously with the measurement of the load acting on the sample. The acoustic registration path contained a broad band transducer of WD PAC type, placed on the steel base of the testing machine. The pass band of the transducer ranged from 80 to 1000 kHz. The transducer was connected to a standardized AE analyser and a computer registering the acoustic data. The amplification in the acoustic channel was equal to 60 dB and one second time interval of summing up the signals was applied. Such a choice was due to the fact that the registration of the force increase was performed every one second. The sampling frequency of AE signal was 44.1 kHz. The final analysis of registered AE signals was carried out using a standard software prepared in Institute of Fundamental Technological Research PAS in Warsaw.

The velocity of stress growth is an important factor affecting the results of the mechanoacoustic tests. The AE descriptors of appearing signals do not form a linear function of changes of the mechanical or thermal stresses. The velocity of the stress changes is one of the factors influencing the acoustic activity. This dependence is additionally difficult to define quantitatively. However, the measurement of the AE descriptors, at very slow increase of mechanical load, of the order of 10^{-2} mm/min allows, that the mechanoacoustic tests become 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 a working load. The velocity of stress changes has essential influence on the structure degradation processes. Higher velocities, of the order of 10^{0} mm/min favour the initiation and growth of transgranular cracks while the lower ones, of the order of 10^{-2} mm/min, favour an increase of intergranular cracks. In the structure of aged ceramic materials, especially insulators after long-term exploitation, mainly intergranular cracks in the matrix are observed [2,3,5]. This fact confirms necessity for the application of lower velocities of stress growth in the investigations of degradation processes. In the case of short-term mechanical strength, high velocity can be used to obtain the proper result. Moreover, the velocity of load increase has important influence on mechanical strength of the tested samples and to smaller extent on dispersion of the results – table 1 [8]. This is the effect of different mechanisms of structure degradation at different experimental conditions. During the investigation of degradation effects in ceramic material the displacement velocity of traverse of INSTRON machine equal to 0.02 mm/min was used. This value enabled the effective performance of the mechanical-acoustic tests of sufficiently numerous groups of samples. Part of them was loaded up to destruction, while in the case of specimens for microscopic study – the 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 [8]
Relative dispersion = 100 %×(value$_{max}$-value$_{min}$)/value$_{average}$

Velocity of stress growth [mm/min]	0.001	0.01	0.1	1
Number of samples	20	20	20	30
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

The descriptors of acoustic emission are registered parallel to mechanical parameters in the second channel of measuring set. In usually brittle ceramic materials, the recorded acoustic emission is mainly associated with the growth of already existing or the initiation of new microcracks. Therefore, a statement, that each microcrack generates a AE pulse defined as the term "event" was the starting point for technical applications of the acoustic emission method [9]. Assuming that the ceramic material is macroscopically homogeneous, it can be supposed that the elongation of each microcrack is roughly equal, and thus the number of AE events is proportional to the elongation of the crack [10].

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. Measurements of acoustic emission have always a comparative nature, because they concern a particular object which is the subject of research. Obtaining the absolute value of energy of AE event is practically impossible [11]. Acoustic emission analyzers, besides the determination of the signal 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:

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

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

The essential factor in AE measurements is the use of such descriptors of signals, which contain the most relevant information for the evaluation of the tested process [12]. 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;
– minimization of influence caused by the signals of the background.

Besides the above criteria, an additional, very important factor must be considered. The acoustic emission descriptor, selected at the research should be optimal for the tested material. As it was experimentally observed by the authors, the choice of AE descriptor should match the specific material. Aluminosilicate materials such as porcelain, steatite or cordierite, are characterized by numerous signals of lower energy. These signals can be effectively registered using the AE event rate. It is specially important during the preliminary and subcritical stages of structure degradation. During the registration of descriptors based on energy of signals, even at low discrimination level, substantial part of them can be lost. The mechanoacustic characteristics of the samples of aluminous porcelain, cut off from long-rod insulator for 400 kV power lines are the Illustration of this effect. In figures 1 and 2, the recorded courses of the rate of AE events and of the effective value of signal (RMS) for the same porcelain sample are presented.

Another example, in which 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. The 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 the occurrence of numerous, but very weak and short in duration intergranular cracks. The large number of signals of small-amplitude are the acoustic effects of this process, both for recording AE events and RMS descriptors. Therefore, the detailed analysis of effects of the initial stage is difficult. These descriptors illustrate well strong and long-term effects of destruction, particularly the critical stage. For the analysis of subcritical stage and in particular the preliminary one, the AE event energy is the most useful. This parameter represents well the energy of particular events, which correspond to weak intergranular cracking of microseconds duration. Recording AE event energy enables a more detailed analysis of all stages of sample degradation, and the presentation of all three stages in one diagram.

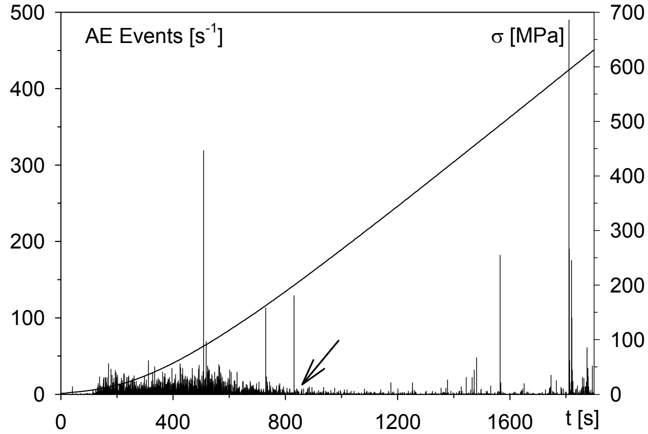

Fig. 1. Typical course of the rate of AE events versus increase of compressive stress for the porcelain sample of the strength 635 MPa. Only preliminary and subcritical stages of degradation in the stress range equal to 0 ÷ 631 MPa are displayed. Arrow points out approximate boundary between stages

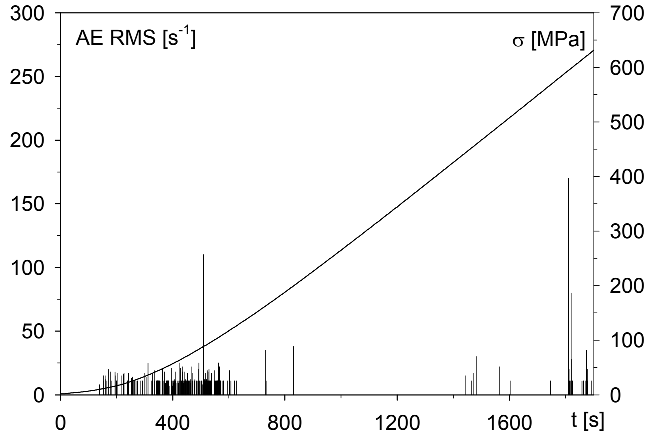

Fig.2. Course of the rate of AE RMS versus compressive stress for the same porcelain sample as in figure 1. Important part of signals is lost because of low energy at preliminary and subcritical stages

Preparation of ceramic samples for microscopic study

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. The role of mechanical polishing factor plays colloidal solution of silica (SiO_2). As a chemically active agent sodium chlorate (NaClO) is usually used. The process of mechanochemical polishing depends on large reduction of forces of chemical bonds in the area of several atomic layers, and next, the mechanical removal of weak bounded layer. The delicate treatment during the mechanochemical removal of successive layers of material enables disclosing even subtle effects of ageing process. Although this method is modern and effective, in the case of mechanoacoustic technique of material degradation research, it has turned out insufficient. During the mechanochemical polishing, the elements of structure weakly integrated with the matrix, undergo crushing out – figure 3. The exact analysis of images of porcelain, especially after the application of compressive stress, requires a different method of preparing the sample surface. A special epoxy resin (e.g. Struers' company production) is poured over the tested samples. Next, the multi-stages process of polishing is performed. The top layer of resin is removed at the beginning. The first - coarse stage of polishing is performed using gradually a diamond suspensions with grain sizes ranging from 9 to 0.1 μm. The final stage is conducted using the colloidal silica suspension with grains having sizes equal to 50 nm. Only such procedure enables avoiding the introduction of additional defects and minimizes the effect of crushing out elements of structure – figure 4.

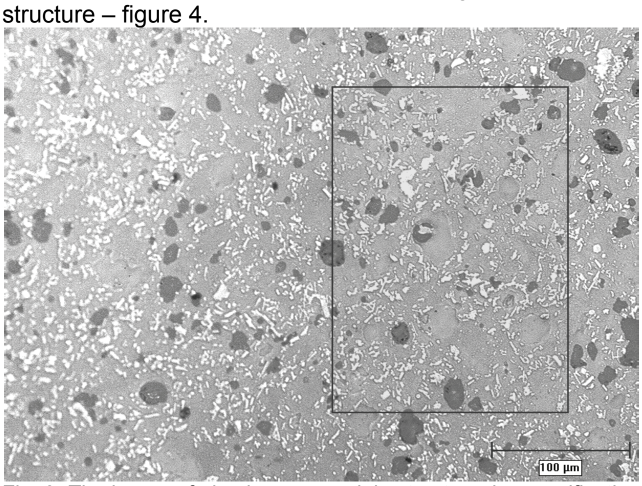

Fig. 3. The image of aluminous porcelain structure, in magnification 200 times, prepared using mechanochemical procedure. Losses after polishing covering crushed out elements of structure are shown as dark fields. They include on average 7.0 % - complete content of cullet – about 4 % and part of quartz phase

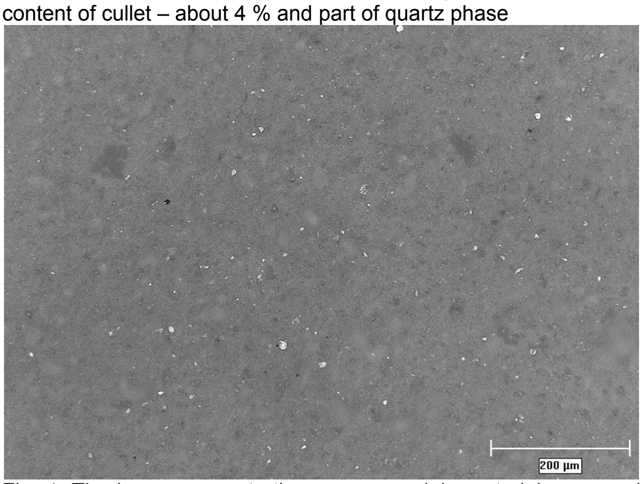

Fig. 4. The image presents the same porcelain material, prepared by the epoxy resin method, in magnification 100 times. White particles of cullet are visible due to special Nomarski's interference phase contrast and polarization. Particles of cullet constitute 2.6 % of surface (total content - about 4 %). Losses after crushed out particles of cullet and quartz grains occupy on average 4.1 %

Concluding remarks

The paper presents mechanoacoustic method of examination of the degradation effects in ceramic materials as well as composites based on them. The application of AE method during slowly increasing compressive stress and comparative microscopic analysis enable the recognition of successive stages of the structure degradation. The sequence of degradation effects, concerning mechanics and components of structure, specified during mechanoacoustic and microscopic examinations, may be compared with real ageing effects of long lasting operation. The required conditions to obtain the above similarity are – very slow, quasistatic growth of loading and sensitive monitoring changes of structure by a properly chosen AE descriptor. Such measurements should be carried out using specially constructed two-channel experimental set-up. The microscopic analysis of sample structure must be performed using special procedures, which do not change the image of the structure degradation. The results of mechanoacoustic measurements of different ceramics materials, obtained so far, enabled distinguishing three stages of structure degradation. The experimentally registered mechanism show similarity to ageing processes observed in the material of exploited insulators.

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