ARCHIVES OF ACOUSTICS
34, 4, 655–676 (2009)

Comparison of Acoustic Emission and Structure Degradation in Compressed Porcelain and Corundum Materials

Przemysław RANACHOWSKI[(1)], Feliks REJMUND[(1)],
Zbigniew RANACHOWSKI[(1)], Andrzej PAWEŁEK[(2)],
Andrzej PIĄTKOWSKI[(2)]

[(1)] *Institute of Fundamental Technological Research*
Polish Academy of Sciences
Pawińskiego 5B, 02-106 Warszawa, Poland
e-mail: pranach@ippt.gov.pl

[(2)] *Institute of Metallurgy and Materials Sciences*
Polish Academy of Sciences
Reymonta 25, 30-059 Kraków, Poland

(*received April 8, 2009; accepted November 16, 2009*)

The paper presents the results of acoustic emission (AE), microscopic and ultrasonic measurements of the samples subjected to slowly increasing compressive stress. On the basis of performed measurements, the successive stages of the materials structural degradation have been recognized. The object of research and comparison were the samples made of the electrotechnical materials – aluminous porcelain and corundum ceramics. Both investigated materials have wide technical application. The analysis of obtained mechanoacoustic characteristics pointed out a complicated mechanism of degradation of the materials. Microscopic investigation of samples, which were stressed to different levels of load, enabled to specify the various course of the gradual growth of microcracks and successive crushing out of elements of the structure. These effects appear to be similar to the aging processes occurring in the materials during long periods of exploitation under working load.

For the two tested materials there were distinguished three stages of the structure degradation. The preliminary and subcritical ones show low or moderate intensity of AE signals and considerable variety for the particular samples. The critical stage directly precedes destruction of the samples. Its range is relatively narrow and contains AE activity of high energy. The presented results enable to draw up conclusions concerning the resistance of investigated materials to the aging degradation processes development during long-term operation.

Comparison of the results of mechanical, acoustic and microscopic studies have revealed that the differences registered for the strength and characteristics are due to inhomogeneities of the materials in the semi-macro as well as in the micro-scale. The effectiveness of dispersive and fibrous reinforcement of aluminous porcelain was described. Strengthening of porcelain by corundum grains and mullite needle-shaped crystals improves mechanical parameters and distinguishes this material from typical aluminosilicate ceramics. The occurrence of groupings of bigger grains in the structure of the corundum material represents most probably the intermediate state, leading to the known effect of the abnormal grain growth (AGG).

Keywords: aluminous porcelain, corundum material, structural degradation, acoustic emission.

1. Introduction

Aluminous porcelain (C 130 type) of high mechanical strength is at present widely applied to produce reliable insulators and small high-strength parts [1–4, 12]. Particularly: line insulators HV (high voltage) and EHV (extra-high voltage), HV post insulators, medium voltage (MV) line and post insulators of increased mechanical requirements, traction insulators and hollow insulators of high parameters. In case of these products, besides high mechanical strength, a long period of exploitation without breakdown is required. For several years there has been observed an increasing tendency for the production of aluminous porcelain and decreasing application of traditional electrotechnical porcelain materials. This is the result not only of the present requirements concerning the short-term mechanical strength of the electroinsulating elements. It is more important 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.

High-alumina ceramics (C 700 group) are characterized by high content of Al_2O_3 (corundum) from 80% to 99.7%. This monophase material has wide and increasing application of general technical purpose. Technical objects made of corundum material take advantages of its high mechanical and thermo-mechanical strength as well as its abrasive resistance. This ceramics is also resistant to oxidation, chemical corrosion and various types of irradiation. Its thermal conductivity is similar to that of stainless steel. Moreover, the corundum material has good electric properties – high dielectric constant and low loss angle. Principal applications are special and low- or ultra-low loss insulators, substrates, metallized parts, sodium vapour lamp envelopes [1–3]. Recently, it is used for the production of the carrying rods of hybrid insulators [4].

The most important factor, responsible for gradual degradation of the parameters of ceramic material, are the local stresses occurring at the grains, the interfacial boundaries and the alien inclusions in the ceramic body. Internal stresses in the micro-areas are located in the brittle medium. The only way of their relaxation is an increase of the already existing (or initiation of the new) microcracks. Thus, relaxation of stresses is connected with decrease of mechanical strength of the material. However, the object in operation is under constant external load. Consequently, the growth of microcracks causes gradual decrease of the working cross-sectional area of the product. So, in the material under load occur the internal stresses, which induce increase of the microcracks. Development of microcracks causes the degradation of the parameters of the material in process of time.

From the reports concerning traditional electrotechnical porcelain (C 120), it is known that about 35 years-long period of exploitation causes over 30% decrease of the mean mechanical strength of the insulating material. Besides that, the mechanical strength dispersion of such objects is about 2.5 times greater than in case of the new ones [5]. In case of ceramic insulators, degradation of mechanical and electric parameters is of great importance, because it decreases reliability of the power supply. Appearance of ageing processes in the ceramic materials has been also confirmed by foreign publications [5, 6]. The factor which has important influence on the degradation of the aluminosilicate materials in the process of time, is high content of quartz. An additional problem is the dispersion of the properties of the material of products, resulting from unsatisfactory repeatability of technological process as well as other effects such as abnormal grain growth (AGG). In case of modern porcelain (C 130), there is not enough experience obtained during a longer period of exploitation of the insulators. Although production of this porcelain in the domestic industry began in the year 1979 (material denoted E-15), it became widely applied not before the nineteen-nineties.

The main aim of present study was description and comparison of the effects of structure degradation in two widely applied ceramic materials. The studied materials are generally different. High-alumina, corundum material belongs to the group of monophase ceramics. Electrotechnical porcelain in turn is an aluminosilicate material, however, containing structural reinforcement by dispersed fine corundum grains and needle-shaped mullite crystals in glassy matrix. The applied methods of testing are hoped to reveal essential differences in mechanical strength and mechanics of degradation process in both materials.

2. Mechanoacoustic method

The method of acoustic emission (AE) is a valuable scientific tool when used for monitoring of the internal structural changes in ceramic materials. This technique allows to obtain numerous data concerning the dynamic processes occurring during changes of mechanical, thermal or thermo-mechanical stresses [7]. The most valuable feature is that AE signals appear already at the threshold stresses, when the generation of microcracks in the material cannot be in practice detected by the other methods.

The samples were subjected to mechanoacoustic measurements using the technique of acoustic emission on a special two-channel measuring system. Pieces of small dimensions were exposed to slowly increasing compressive stress with simultaneous registration of the force in one channel, and AE descriptors in the second one. The investigations enabled the recording and description correlation between the increasing external load and the processes of structure degradation, which are reflected in AE activity. Acoustic method is especially suitable for the investigation of the destruction in ceramic materials, due to the fact that initiation and growth of microcracks belong to the main sources of AE signals. Examination of aluminosilicate and oxide 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. These effects are the cause of the mechanical degradation of brittle materials. There exists a correlation between the rate of increase of cracks and the rate of AE events (number of AE events per unit of time) [8]. Registration of this descriptor allows monitoring of the destruction process of microstructure of the ceramic material under load. This is especially useful for aluminosilicate ceramics, which is the source of numerous acoustic signals of lower energy. In case of corundum material, the authors stated distinct correlation between the structure degradation, mainly connected with microcracks development, and the effective value of AE signal (RMS – root mean square). This is connected with high mechanical strength of the corundum and relatively high energy of the generated AE signals.

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 the AE events rate and RMS as descriptors, at very slow increase of mechanical load (of the order of 10^{-2} mm/min) allows, however, to make the AE investigations almost independently of the influence of experimental factors on the degradation process of the material. This has been confirmed by the authors during research of porcelain (C 120) and cordierite (C 410) materials [9, 10].

The structural effects of slowly increasing compressive load, applied to the material, and aging processes, being result of many years' period of exploitation under working load, appear to be similar. However, it is necessary to apply a quasi-static, very slow increase of stress and a precise registration of the AE descriptors. This observation has been proved by investigations, carried out by the authors [9].

Mechanoacoustic tests were carried out using specially constructed two-channel measuring system. The mechanical channel contained the testing machine INSTRON with computer control. The steel base on which the sample was placed, functioned simultaneously as an acoustic waveguide. During investigation, the velocity of the traverse of the machine equal to 0.02 mm/min was applied. Parallel to the measurement of the load acting on the sample, AE descriptors were recorded. The acoustic registration path contained the broad-band transducer WD PAC type, standardized AE analyser and a computer. Frequency band of measurements was equal to 5–800 kHz. Amplification of AE channel was equal to 60 dB and sampling signal frequency – 44.1 kHz. One-second time interval of summing up the signals was applied. The rate of counts, the events rate and the energy of AE signals were recorded. Valuable information for the evaluation of the examined processes of material degradation is offered by AE events rate, especially for the porcelain, as well as the effective value of AE signal – RMS in case of corundum ceramics. Registration of AE descriptors was performed in accordance with standard [11]. The measuring set is shown in Fig. 1.

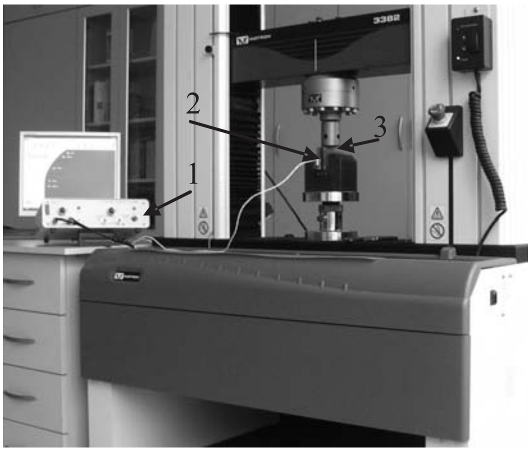

Fig. 1. Two-channel measuring system applied to mechanoacoustic tests of the ceramic samples. There are marked: 1 – AE analyser, 2 – acoustic transducer, 3 – enclosure containing the sample.

Mechanics of degradation of the porcelain material was recognized on the basis of a detailed comparative study of mechanoacoustic characteristics and microscopic analysis of many samples. Compression of particular samples was stopped at different levels of stress – from 50 MPa to the loading just before destruction. Only the destructed samples were not subjected to microscopic study. The structural effects of degradation were compared with acoustic activity of the samples. Detailed study of many samples loaded up to early, intermediate and advanced stages of material degradation, allowed a description of structural changes and mechanics of the process.

3. Preparation of samples

Study of aluminous porcelain was carried out on specimens cut-off from typical high voltage line insulator of foreign production. Traditional and used course of fabrication of long rod insulators consists of many stages [6, 12]: selection of components, control and preparation of raw materials (weighing and milling), plasticization of raw material (mixing with water, filtration, seasoning), formation (deairing, pug pulling and profile turning), drying, glazing (marking), firing (sintering), final treatment (cutting and grinding), montage of fitting devices (assembling), tests of the object. The samples had dimensions $8 \times 8 \times 10$ mm. The composition of the raw mass applied to fabricate the insulator was not known exactly. On the basis of structural analysis were found the contents of ceramic alumina – approximately 24% and cullet – 4%. In comparison to domestic C 130 material, contents of silty minerals (kaolin and refractory plastic clays) was higher – over 50% and of feldspar fluxes was lower – below 20%. Surface of the samples, especially bottom and top, were precisely ground to obtain parallel and smooth planes of the order of 0.05 mm.

There have been also presented the mechanoacoustic and structural investigations of corundum material (C 799) in which Al_2O_3 content was equal to 99.7%. In preparation of the samples, the granulated product NM 9922 of Nabaltec firm was used. The size of grains, collected in the aggregates, was less than 0.5μm. The crystallites were smaller by one order of magnitude. The samples were formed using the method of single axial pressing (10 MPa) and then condensed isostatically (120 MPa). After firing into biscuit at the temperature 1250°C, samples were cut out from a larger element, taking into account the grinding and shrinking effect of the mass. Next, the samples were fired at the maximal temperature 1700°C and stored for 1.5 hr. at the sintering temperature. The samples were ground to obtain the dimensions $5 \times 5 \times 10$ mm, then their density was determined and the absorbability and absence of cracks in the alcohol solution of fuchsine were controlled. The obtained material had the density $\rho = 3.89$ g/cm^3 and did not contain any detectable defects. In order to determine the size of grains in microscopic analysis, some samples were polished, thermally etched at the maximum temperature 1300°C and stored for 1 hr. at this temperature.

4. Ultrasonic study

The acoustic method is based on the dependence between the parameters of waves propagation and the properties of the medium, in which the waves propagate. In case of a solid body, the wave propagation depends on the elastic properties of the material, as well as on its structural composition. The ultrasonic method has been widely applied in the flaw detection. Detecting the discontinuities of the medium is performed by introducing a wave beam into the investigated material and then recording its reflection from the boundary. Among the possible applications of ultrasonic method, very important is the elastometry. On the basis of experimentally determined values of the velocities of the longitudinal – c_L and transverse – c_T ultrasonic waves, as well as the known material density ρ, it is possible to obtain its mechanical parameters, e.g. Young's modulus E [13]:

$$E = \rho c_T^2 (3c_L^2 - 4c_T^2)/(c_L^2 - c_T^2). \tag{1}$$

Attenuation is an additional significant ultrasonic parameter, which above all allows evaluation of the advancement of the degradation processes in the ceramic material. The results of the energy dissipation are the ultrasonic signal amplitude changes and pulse deformation. This effect is due to the existence in the material of the numerous structural inhomogeneities, such as micro-cracks, frequently spaced pores, large crystalline phase precipitations, as well as the areas where mechanical stresses appear. The presence of the network of cracks causes especially strong increase of the attenuation. Ultrasonic measurements can be performed in different directions. So, an evaluation of the material anisotropy is possible.

The ultrasonic control of the homogeneity of the porcelain revealed the anisotropy typical for ceramic objects, formed by using the screw extrusion method in the vacuum deairing pug mills [4, 12]. The velocities of the longitudinal c_L and the transverse c_T waves, measured along the lengthwise axis of insulator, were equal to 6660 m/s and 3930 m/s, respectively. The calculated value of Young's modulus E equalled 96 GPa (density of the material $\rho = 2.51$ g/cm^3). In the crosswise direction of insulator, c_L had the value 6450 m/s and c_T – 3830 m/s, respectively. The Young's elasticity modulus, determined from these data, was equal to 90 GPa. The uncertainty of measurements for c_L was ± 20 m/s and ± 40 m/s for c_T, whereas for the calculated values of Young's modulus it was about ± 1.5 GPa.

Acoustic control of the homogeneity of the corundum samples revealed a slight anisotropy as well as some differences of the acoustic parameters and the elasticity modulus between the samples. For example, the velocity of the longitudinal waves c_L, measured in the direction perpendicular to the axis for various samples, amounted from 10 480 to 10 600 m/s (the inaccuracy of measurement was equal to ± 20 m/s). Young's elasticity modulus E, determined in the same direction was enclosed, depending on the sample, in the range from 364 to 373 GPa. The average E value was 368 GPa, at the measurement inaccuracy equal to ± 2 GPa. The mean value of Young's modulus in the direction parallel to the sample length was somewhat lower and was equal to 360 GPa. The obtained parameters considerably exceed the required by standard values of $\rho = 3.70$ g/cm^3 and $E = 300$ GPa for the corundum material of C 799 type [3].

5. Mechanoacoustic examination of porcelain

The compressive strength of twelve samples loaded until their complete destruction showed relatively high dispersion. The mean value of strength was equal to 580 MPa, the lowest – 391 MPa and the highest was 735 MPa. The relative dispersion for these results corresponded to value 52.6%. In calculations, the lowest value of strength was neglected and as the weakest result was assumed the value 430 MPa. Besides the broken samples, the group of a few specimens was selected for the microscopic investigation. The compression process of these samples was stopped at different levels of the stresses from 200 to 723 MPa. The registered mechanoacoustic characteristics of the particular samples showed considerable differentiation. Analysis of these results and microscopic study revealed generally the presence of three stages of the structure degradation. Figures 2 and 3 show typical courses of acoustic activity for the samples destroyed at 635 and 642 MPa. In Fig. 2 was presented the range of stresses between 0–631 MPa to show only the preliminary and the subcritical stages of the material degradation in a more distinct way.

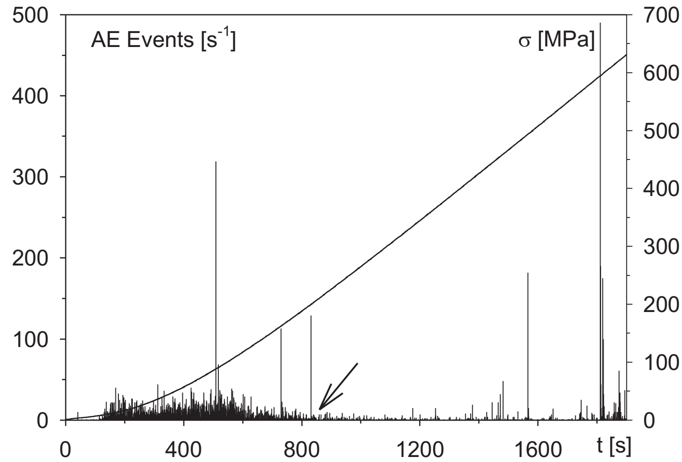

Fig. 2. Typical course of the rate of AE events versus the increase of compressive stress for the sample of the strength 635 MPa. There are displayed only the preliminary and subcritical stages of degradation in the stress range equal to 0–631 MPa. Arrow points at the approximate boundary between stages.

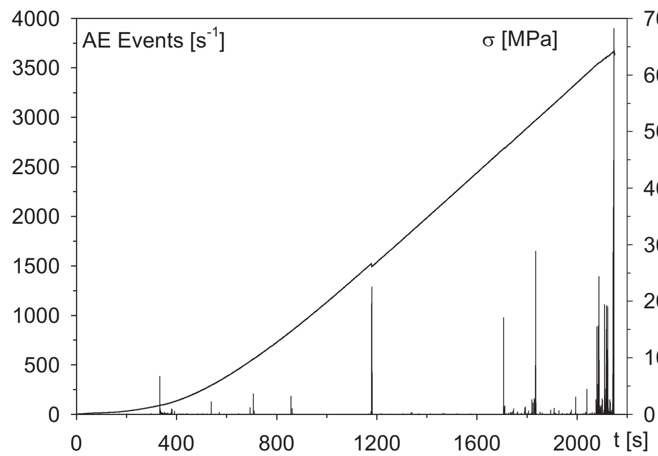

Fig. 3. The course of the rate of AE events versus the increase of compressive stress for the sample of the strength 642 MPa. The AE signals of the preliminary and subcritical stages are almost invisible because of their lower amplitude.

In Fig. 3 is visible a characteristic jump down on the curve of stress increase and strong AE signals, at the load equal to 264 MPa. These effects are the result of local fracture and splitting off a piece of the sample.

The first stage of acoustic activity, described as a preliminary one, reaches approximately from 10 to 200 MPa of compressive stress. This stage is characterized by low or moderate intensity of AE signals and considerable variety for the particular samples. The preliminary stage of the material degradation occurs mainly as a result of internal stresses existing in the ceramic body, primarily in the micro-scale, created during the manufacturing processes. The process of increase of defects has a relatively low threshold energy and can develop already at lower stresses acting on the sample. This period corresponds to a destruction of the particles of cullet, the quartz grains and the earlier stage of the glassy-mullite precipitates damage – Fig. 4.

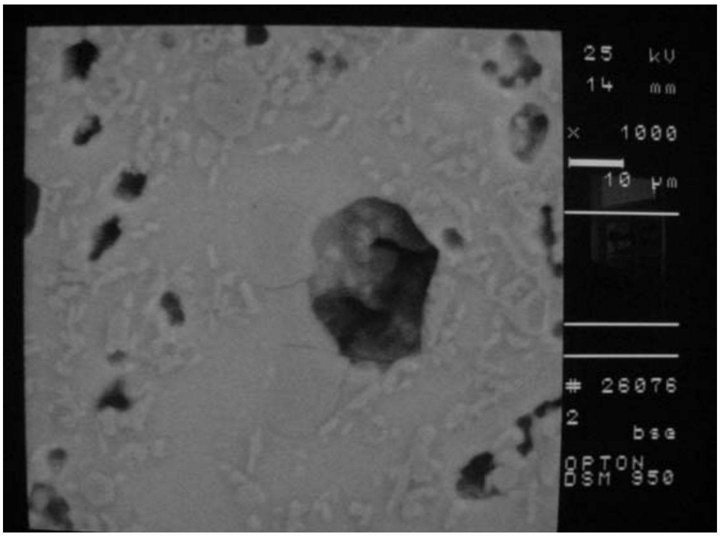

Fig. 4. Structure of the sample loaded up only to the preliminary stage of degradation (200 MPa). The bright damaged glassy-mullite precipitate with crushed out dark part of it is visible. Precipitate contain also internal and peripheral microcracks. Smaller black areas are places after crushed out particles of cullet and quartz grains.

The last effect takes place especially in the central part of samples, where the highest concentration of stresses occurs. The first damages of the glassy-mullite precipitates are registered already at the beginning of the preliminary stage of degradation. It is connected particularly with peripheral cracking at the borders of the glassy-mullite precipitates and the matrix, where mechanical stresses are present. These precipitates come from the particles of silty minerals of raw ceramic composition. Threshold energy of their separation from the matrix is relatively low. In the stress range less than 100 MPa, without any acoustic activity, particles of cullet undergo fracture and separation from the porcelain matrix. Greater part of cullet is separated from the matrix, even without action of external stresses. The majority of the quartz grains go through a similar process in the approximate stress range 50–200 MPa, Though, in case of few greater quartz grains this process requires much higher loading. Degradation of the quartz phase is followed by a weak AE activity. A little stronger acoustic effects follow only the cracks development in the quartz grains of greater size (over 15 μm) and inside the glassy-mullite precipitates. The degradation process of these precipitates, which are numerous in the porcelain structure, takes place up to destruction of the sample. This effect is the most important source of AE signals during preliminary and especially, subcritical stages of the material degradation. The destroyed elements of structure (culet, quartz and precipitates), at the end of preliminary stage, comprise about 10% of the surface of compressed samples, analyzed by means of the microscopic method.

The next stage of structure degradation – the subcritical one - is closely connected with the homogeneity of the sample structure in micro- and semi-macro scales. The subcritical stage begins after the end of preliminary stage and lasts frequently to the beginning of the critical stage. This phase of destruction is considerably varied for particular samples and shows low or moderate intensity of AE activity – Figs. 2 and 3. During this period, further damage of numerous glassy-mullite precipitates takes place. There are created internal and peripheral cracks in the precipitates. Their parts undergo crushing out, especially in the central section of the samples. Moreover, already at the stresses over about 200 MPa occurs degradation of the centers of the agglomerated corundum grains.

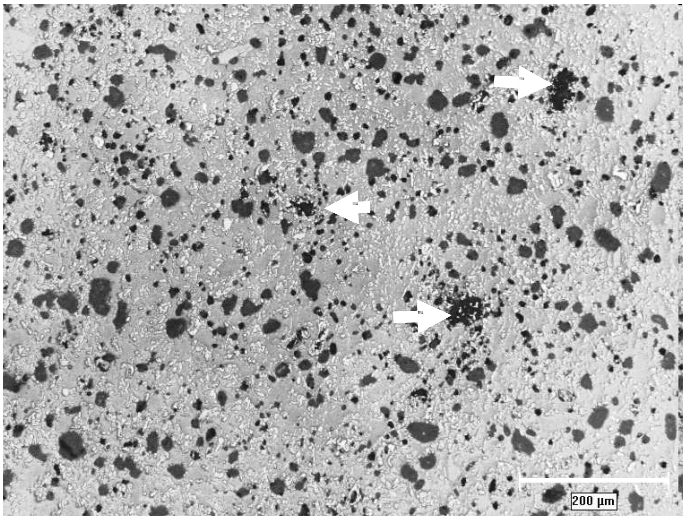

Fig. 5. Typical structure of the sample loaded up to the advanced subcritical level. Black, crushed out parts of glassy-mullite precipitates, quartz grains, cullet fragments and damaged agglomerates of corundum grains (marked by arrows) are visible. Black places comprise together 13 percent of the surface.

As the loading grows, particular corundum grains undergo a separation from the structure of agglomerate – Fig. 5. The agglomerates of collected grains of corundum are randomly distributed in the material structure and the subcritical stage of degradation is connected especially with the destruction of glassy-mullite precipitates. This process is the most intensive in the central part of the samples. At the end of the subcritical phase, area of the damaged, separated and crushed out elements of structure comprises between 10 and 15% – Fig. 5. Important part of them constitute cullet and quartz damaged at the preliminary stage.

The last – critical interval, showing the highest level of the acoustic activity, begins at loading from several to dozen or so of megapascals lower than the destructive stress for the particular sample. It lasts up to the destruction of the specimen. This interval is characterized by generally good repeatability of the level of AE signals, which have the highest intensity. The extent of this process, however, depends on the strength of the particular sample. The range of the critical stresses, observed in the part of destroyed samples, results from a fracture and splitting off the greater pieces of sample – Fig. 6. The most frequent fracture and splitting off the walls and corners from the sample precede its final destruction. This effect is shown on the stress increase curve as characteristic faults (jumps down), preceding decohesion of the sample. However, some specimens underwent destruction unexpectedly – after a very short critical phase. During the critical stage, further glassy-mullite precipitates undergo the internal and peripheral fracture. Degradation of the corundum agglomerates is continued as well. Even single grains of corundum can be separated from the matrix. During critical stage, just before destruction, the area of damaged, separated and

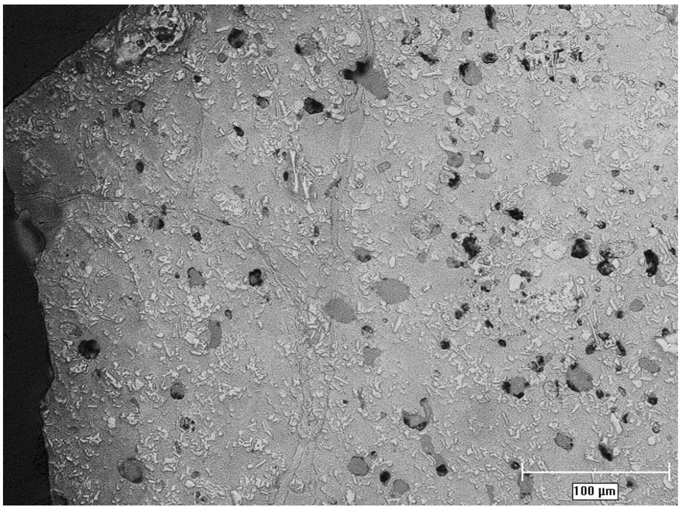

Fig. 6. Side part of the sample loaded to the critical stage, just before destruction. Irregular surface and cracks are the results of splitting off the lateral wall of the sample.

crushed out elements of structure comprises between 14 and 16%. However, the most important and destructive effect, followed by the strong AE signals, is formation and growth of cracks in the porcelain body. These cracks are elongated and in general not branched – Fig. 7. They grow initially between the ruptured glassy-mullite precipitates and other damaged elements of the structure. The strong dispersive and fibrous reinforcement of the porcelain structure hampers their increase. In case of typical aluminosilicate ceramic materials, single cracks join together and form an even network of cracks. These effect was observed in compressed traditional insulator porcelain [9]. At sufficiently high stress the rapid growth of critical cracks in the porcelain body takes place and the sample undergoes destruction.

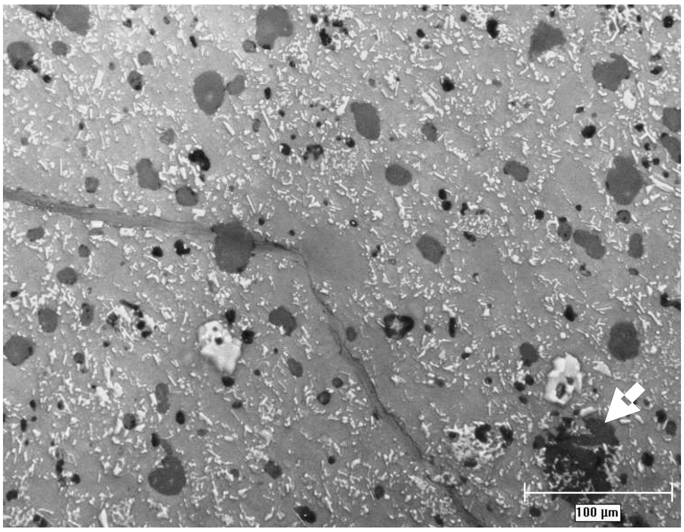

Fig. 7. Effects of critical stage of the porcelain structure degradation at loading 723 MPa. Long bent crack and damaged glassy-mullite precipitates as well as agglomerate of corundum (marked by an arrow) are visible. All quartz grains and particles of cullet are crushed out.

In Fig. 8 is presented the dependence of a number of AE events versus the energy for four samples denoted 2, 13, 6 and 7 – damaged at compressive stresses 391, 451, 632 and 641 MPa respectively. Weaker samples are characterized by a greater number of AE signals of low energy, which corresponds to the damage of glassy-mullite precipitates and quartz grains weakly connected with the matrix. Stronger samples – containing more resistant glassy-mullite precipitates, smaller number of the centers of aggregated corundum grains and more homogeneously distributed crystalline phases in the matrix, show a greater number of signals of higher energy. The number of recorded AE signals is generally inversely proportional to the compressive strength – Table 1. The same dependence was found by the authors for insulator porcelain of domestic production [14].

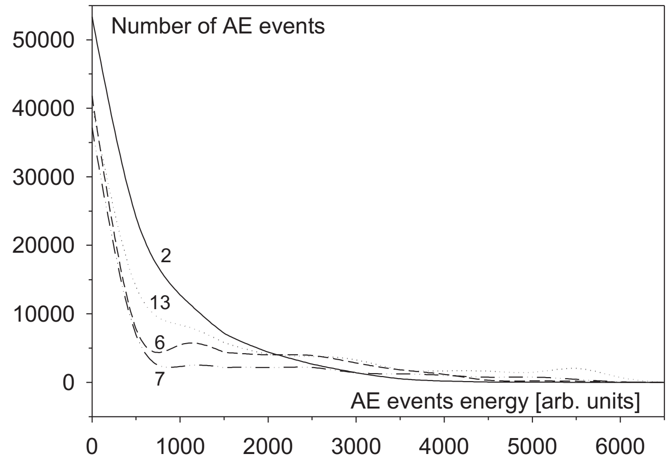

Fig. 8. Plots of the total number of AE events as a function of AE event energy for the porcelain samples of different compressive strengths: 2 – 391 MPa, 13 – 451 MPa, 6 – 632 MPa and 7 – 641 MPa.

Table 1. Dependence between the compressive strength and number of AE events for selected porcelain samples.

Mark of sample	Compressive strength [MPa]	Number of events
2	391	39 183
13	451	37 108
6	632	22 477
10	635	22 027
7	641	14 205
12	642	17 589

6. Mechanoacoustic research of corundum material

The compressive strength of the corundum samples has shown an unexpectedly wide distribution. The mean strength of nine destroyed specimens was 3290 MPa. The least resistant of the samples underwent decohesion already at the stress equal to 2040 MPa. This result was neglected in statistical calculations. The next weakest sample had a strength of 2660 MPa. It was found impossible to measure the strength of the most resistant sample. For technical reasons it was necessary to stop the process of loading at the stress of 3800 MPa. Relative dispersion of strength equaled 34.6%.

The mechanoacoustic characteristics of the particular samples show high differentiation – Figs. 9 and 10. At the stresses below 2500 MPa the samples, especially of more inhomogeneous structure, demonstrate AE signals of various intensities. If there appear the intervals of continuous acoustic activity, the AE signals

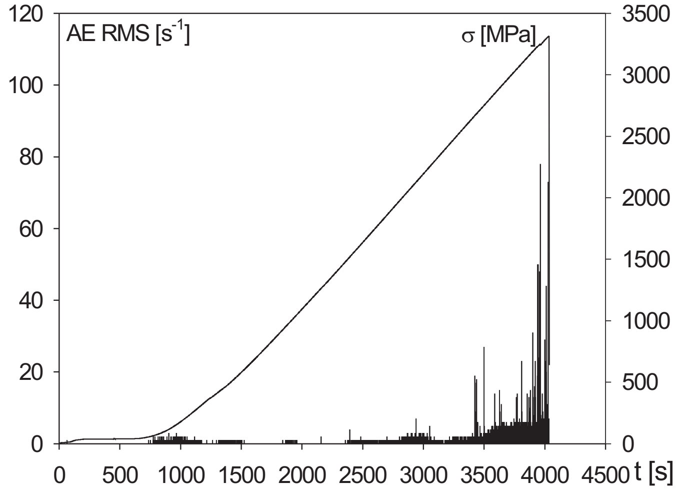

Fig. 9. Typical course of RMS AE rate versus the increase of compressive stress for the corundum sample of the strength 3 320 MPa.

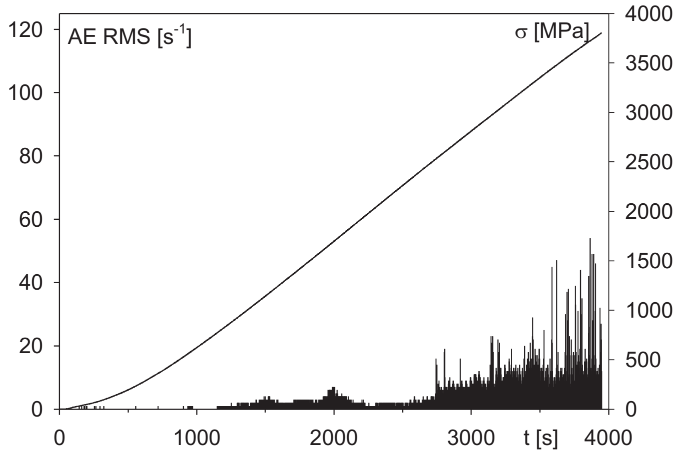

Fig. 10. The course of RMS AE rate versus the increase of compression stress for the most resistant sample. Compression was stopped at 3 800 MPa, before the critical stage of degradation.

are the most frequently characterized by low energy. This interval of stresses corresponds to the first – the preliminary stage of the corundum structure degradation. Above the stress 2500 MPa the loaded samples show AE effects in the form of single signals, sometimes forming intervals of continuous acoustic activity of moderate energy. The interval of subcritical stresses is characterized by diversified width, depending on the sample strength. The range of stresses and intensity of AE signals are characteristic for the particular samples. The interval of the subcritical effects spreads throughout several hundreds of megapascals. The wide subcritical stage precede a short critical interval, containing a group of signals of high energy. The critical interval occurs in the range of stress of some tens of megapascals and it directly precedes the decohesion of the sample. Thus, the stress at which the critical stage takes place is closely connected with the sample strength – Fig. 9.

In order to explain the effects of structural degradation of the corundum material, there were carried out extended microscopic investigations of the samples. Structural research concerned the size and the uniformity of distribution of the grains and the effects of the compressive stress at various areas of the specimens. A special attention was paid to the presence of defects in the structure before and after compression of the samples.

Research revealed that considerable differences registered for the strength and the mechanoacoustic characteristics of the samples are the result of inhomogeneities and faults in the structure in the semi-macro as well as in the microscale. The samples contain fine technological defects such as gaseous and solid inclusions, fissures and partly broken grain boundaries – Fig. 11. Almost all of the samples demonstrate the bimodal size distribution of the grains. The sizes of bigger grains are most often in the range from less than 10 to over 30 μm, whereas the diameter of smaller grains is from a fraction to a several micrometers – Fig. 12. Only few samples have a uniform size of grains and proper internal texture. The grains of the most resistant sample show one-modal size distribution. Their diameters are enclosed in a narrow range from above 1 to 14 μm, with the mean value equal to 7 μm.

There are observed textural inhomogeneities in the structure of the samples. The bigger grains often form centres of the size of the order of tens micrometers

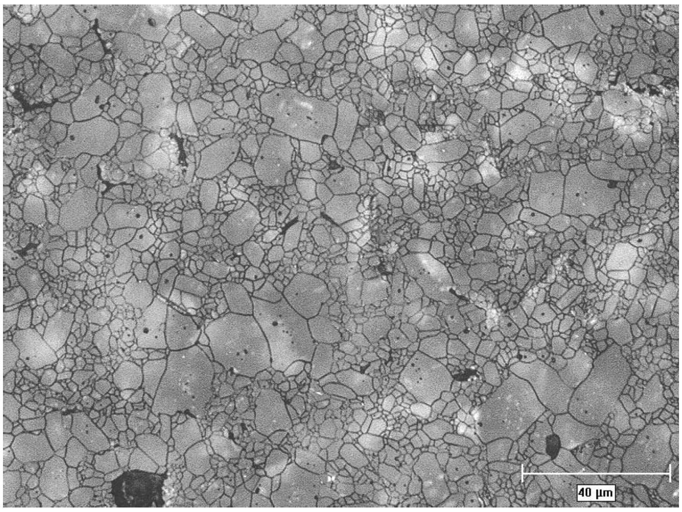

Fig. 11. Textural inhomogeneity (centres of bigger grains surrounded by finer grains), weakened grain boundaries and a big pore are visible. Structure of the corundum material before the beginning of compressive loading.

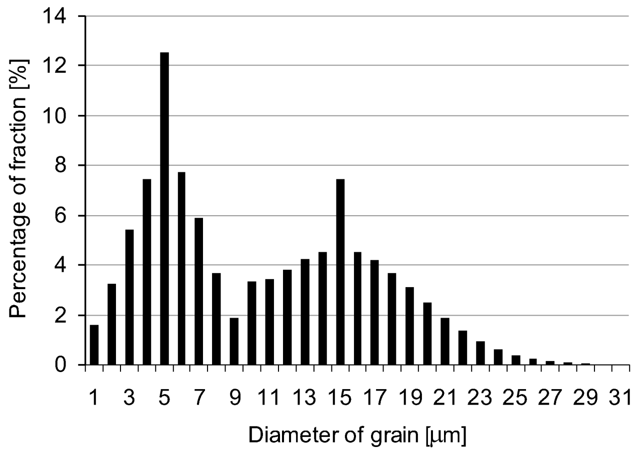

Fig. 12. Typical bimodal distribution of the grains diameters, obtained for the sample of the corundum material.

(up to 100 μm), surrounded by smaller areas containing finer grains – Fig. 11. Another discovered textural inhomogeneity is arrangement of the structure in the form of bands. The greater and/or the smaller grains are structured as separate bands, having width of the order of some tens of micrometers. The most regular structure is observed in case of the sample of the highest strength.

After the compression tests, several samples of various mechanoacoustic characteristics were selected for microscopic investigations. Loading of these samples was stopped at different stresses. A few were stopped closely before the critical stage of structure destruction. The compressed samples were cut and polished properly for the microscopic study.

In all the specimens there were observed the effects of structure loosening. This concerns especially the central part of the samples, where the stresses are cumulated. Propagation of microcracks occurs almost only along the grain boundaries. There are observed black areas displaying crushed out grains or parts of grains. This effect is most distinctive in the images of analysed structures. For samples compressed up to the advanced subcritical effects, the areas of damaged corundum grains cover 0.2–0.3% of the surface of polished sections at the boundaries and up to 1.0% in the middle of the samples. While, the initial porosity of the material in any of the specimens does not exceed 0.1%. The size of crushed out grains or their fragments is generally below twenty micrometers. The structure of the compressed samples undergoes evident but varied loosening. The length of intergranular cracks is at peripheral parts of the samples in the range of 10–30 μm, and 40–60 μm – in the middle. Many of them have a circular character (around single or groups of grains). There occur also bigger cracks of the nature of fissures. Various areas of the samples demonstrate different degrees of structural degradation. The most resistant of the investigated samples (compression stopped at 3800 MPa), shows a moderate degree of structure degradation. Even in its central part, the surface area of the crushed out grains is lower than 0.3%. The mean length of cracks is about 20 μm and they are shorter than 50 μm – Fig. 13. However, in the central part of a typical sample compressed up to the beginning of critical stage – 3180 MPa, there are observed even long cracks of the length of some hundreds of micrometers – Fig. 14.

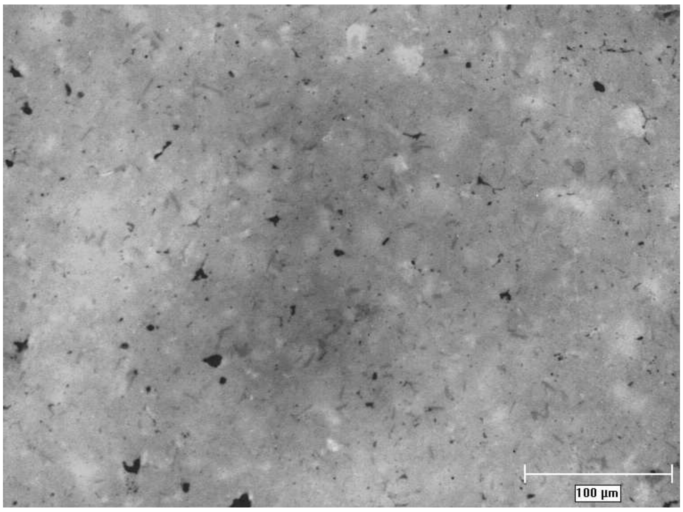

Fig. 13. Structure of the central part of the most resistant sample, compressed to 3800 MPa.

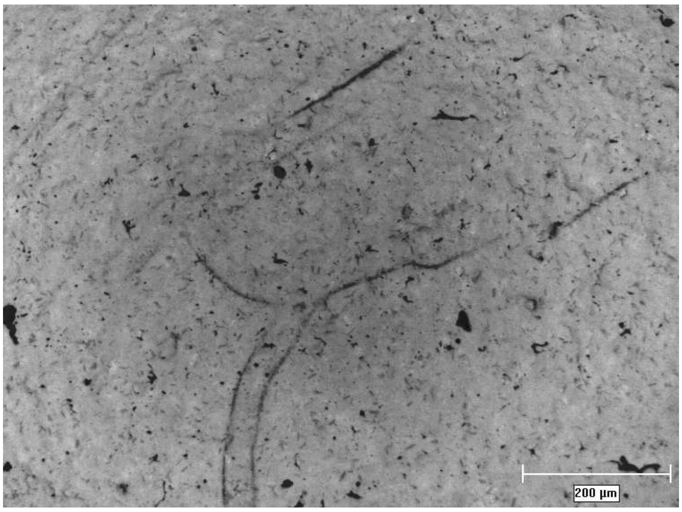

Fig. 14. Cracks in the central area of the sample loaded up to 3180 MPa.

Corundum samples compressed to advanced subcritical stage reveal the presence of micro- and macrocracks. Over 90% of cracks undergo propagation along the grain boundaries and only under 10% of them – within the grains. Points of initiation of the cracks are technological defects (gaseous and solid inclusions, fissures and partly broken grain boundaries), shown in Fig. 11. The earlier, preliminary stage of AE activity is connected with these defects. Threshold energy of relaxation of the stresses, connected with occurring defects, is low and such is the effective value of AE signals. This phase of degradation is varied for particular samples. It concerns the range of stresses, where signals and their intensity are registered. The preliminary stage depends closely on the number, size and spatial distribution of technological faults. This stage of degradation can take place in a very wide range of loading, even up to about 2500 MPa – Figs. 9 and 10.

During the subcritical stage of the material degradation, further growth of intergranular cracks takes place. The most easily – fracture bounds between bigger grains, especially these elongated, and mainly in the direction perpendicular to the direction of compression. Centers of joined bigger grains are particularly susceptible to the destruction process as well. As a result, the strength of the samples, containing greater number of centers and bands of the bigger grains (10–40 μm), are considerably decreased. Cracking within the bigger grains as well as at the boundaries of small grains is considerably less frequent because of the much higher threshold energy. AE signals appearing during the subcritical stage are strongly diversified. Their energy is moderate but growing and generally higher than in the previous stage. The range of stresses, for which subcritical stage takes place, is varied and most frequently extends to a few hundreds of megapascals.

During the critical stage of destruction, propagation of cracks occurs with high velocity and throughout intergranular boundaries and across the corundum grains. Figure 15 presents strong damages, which take place at the beginning of the critical period of the structure degradation. Growth of visible cracks results in continuous and high-energy AE activity. Sometimes, before destruction of the whole sample, its macroscopic parts (e.g. a piece of wall or corner) undergoes separation. The range of the critical stage of the corundum material includes the range of stresses of several tens of megapascals and is generally wider when compared to that of the aluminosilicate ceramics.

Textural defects occurring in the examined ceramics are connected with incidence of grains of different size, which are not uniformly distributed in the space of the samples. Sometimes they group into centres or bands. The observed diversification of grains' size in the structure of the corundum material (Fig. 11 and 12) represents most probably the intermediate state, leading to the known effect of abnormal grain growth (AGG) [15]. This phenomenon occurs most frequently in the oxide ceramic materials. It has a probabilistic character and its origin, despite many years of investigations, has not been sufficiently explained. The AGG effect occurs after longer time of firing than in case of the applied technology of fabrication of the samples. The quick increase of temperature in the course of thermal treatment favours its occurrence and such a temperature growth – of

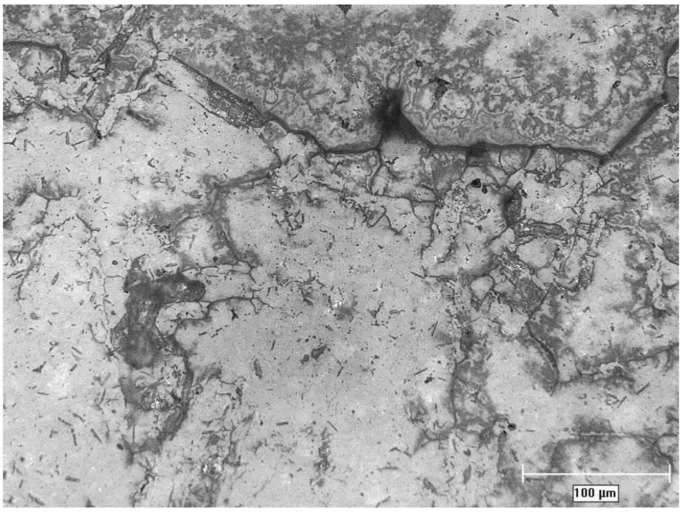

Fig. 15. Critical degradation effects visible on the surface of the sample, its compression being stopped at 3190 MPa.

the order of 200°C by one hour – was realized. In case of obtaining bigger and longer time sintered elements, the AGG effect would cause considerably greater differences in sizes of the grains.

7. Concluding remarks

In the paper were described mechanoacoustic, ultrasonic and microscopic investigations of two ceramic materials, widely applied in contemporary technology. The corundum samples of low dimensions were fabricated specially for the study, while the porcelain specimens were cut out from the rod of high-voltage line insulator of foreign production. Technology of fabrication for both ceramic materials was quite dissimilar. Aluminous porcelain is an aluminosilicate material, characterized by complex multiphase composition, with dispersive and fibrous structural reinforcement, while the corundum material belongs to monophase oxide ceramics. Different was the scale of production and size of the objects. The results enabled a comparison of mechanical and acoustic properties, microstructure, texture and homogeneity of the investigated ceramics. Additionally, registering of the mechanoacoustic characteristics made it possible to distinguish subsequent stages of the structure degradation process. The authors suppose that there exists a similarity between the effects of long-term exploitation under working load and the material degradation during relatively short compressive test [9, 10, 16]. There was used a very slow, quasistatic growth of loading and sensitive monitoring changes of structure by AE descriptors. It was the necessary condition to obtain similar effects as in the case of long-term degradation.

In the both materials were discovered inhomogeneities. In case of porcelain, they are of small importance and restricted to spatial distribution of glassy-mullite precipitates and to the presence of corundum agglomerates in the material. Whereas, the best part of corundum samples contain distinct inhomogeneities: inclusions, fissures and partly broken grain boundaries. There are present also bimodal grain-size distributions. The bigger grains often form centers or bands. This textural defect probably results from an earlier stage of abnormal grain growth (AGG) effect [15]. In spite of these disadvantageous factors, the compressive strength of the corundum samples is high, in average 3290 MPa, relative dispersion of this parameter does not exceed 35%. In case of the porcelain, mean strength equals 580 MPa and relative dispersion – about 53%.

For the two tested materials, there were distinguished three stages of the structure degradation. The preliminary and subcritical ones show low or moderate intensity of AE signals and considerable variety of the particular samples. The critical stage directly precedes decohesion of the samples. Its width is relatively narrow and contains AE activity of high energy.

The primary stage is connected with development of defects, initiated during any technological operations. These defects introduce internal stresses. Their relaxation has low threshold energy and generates AE signals of low intensity. For the porcelain, there occurs primarily an earlier stage of glassy-mullite precipitates damage and degradation of the quartz phase. The range of stress comprises up to 200 MPa. In case of corundum it is related to restricted growth of intergranular cracks. The stress range of this stage is very wide and can approach even 2500 MPa.

The subcritical stage is strongly varied for the particular samples and shows low or moderate intensity of AE activity. Signals occur separately or in groups. The range of stresses and descriptors of AE signals are characteristic for particular samples and usually differ considerably between themselves. In the structure of porcelain occur the effects of degradation of glassy-mullite precipitates and agglomerates of collected grains of corundum. Damage of numerous glassy-mullite precipitates, where internal and peripheral cracks raise, is characteristic for the porcelain material. The subcritical stage results in crushing out of many pieces of precipitates and agglomerates. The joining element of porcelain structure, reinforced by mullite and corundum matrix during subcritical stage, undergoes damage only to limited extend. The interval of stresses for corundum material is much wider because of its high strength, when compared to porcelain. The subcritical stage is closely connected with the inhomogeneity of the samples structure in micro and semi-macro scales. In the corundum material it results from textural defects – bigger grains grouped into centers or bands. Groupings of bigger grains are more susceptible to intergranular cracks growth. It is observed lengthening of cracks, especially in the central part of samples as well as longwise of elongated grains, and in the perpendicular direction to the compressive load. As a consequence, degree of structure loosening increases as the stress grows.

The critical stage is the shortest one and directly depends on the sample strength. Propagation of long critical cracks is followed by very strong continuous AE activity. Fracture and splitting off greater pieces (walls and corners) from the sample precede destruction of the specimen. This effect is more frequent in porcelain. Degradation of glassy-mullite precipitates and corundum aggregates, even of singular grains of corundum, is being continued. However, the most characteristic phenomenon during the critical stage, is propagation of cracks in porcelain matrix, reinforced by dispersed mullite and corundum. At the beginning, long cracks develop between glassy-mullite precipitates, especially those damaged. At suitable high stress, the cracks and seldom its cascades grow through all phases of the porcelain body. In case of the corundum material, long cracks appear mostly in the areas of bigger grains groupings. At sufficiently high-stress very fast propagation of these cracks takes place. This rapid process occurs not only along the boundaries but also through the grains of different size.

In opinion of the authors, the presented sequence of degradation effects, concerning mechanics and components of structure, is similar to the material degradation occurring during long-term exploitation of the insulator rod under working load. The results of the research concerning the porcelain taken from the operated insulators also appear to confirm the described results [5, 9, 16].

Acknowledgments

This work was financially supported by the Ministry of Science and Higher Education – Research Project No. N507 056 31/1289.

This article is an extended version of the paper presented at the 56th Open Seminar on Acoustics – OSA2009, September 15–18 in Goniądz.

References

[1] IEC Publication 672-1:1995 Ceramic and glass-insulating materials, Part 1: Definitions and classification.

[2] IEC Publication 672-2:1999 Ceramic and glass-insulating materials, Part 2: Methods of test.

[3] IEC Publication 672-3:1997 Ceramic and glass-insulating materials, Part 3: Specifications for individual materials.

[4] Pohl Z., *Outdoor high voltage insulation in electrical engineering* [in Polish], published by Wrocław Technical University, 2003.

[5] Liebermann J., *Avoiding Quartz in Alumina Porcelain for High-Voltage Insulators*, American Ceramic Society Bulletin, **80**, 6–7, 37–48 (2001).

[6] Carty W., Senapati U., *Porcelain – Raw Materials, Processing, Phase Evolution and Mechanical Behavior*, J. Am. Ceram. Soc., **81**, 1, 3–20 (1998).

[7] Ranachowski J., Rejmund F., *Acoustic Emission in Technical Ceramics* [in Polish], [in:] *Acoustic Emission – Sources, Methods, Applications*, Malecki I., Ranachowski J. [Eds.], pp. 55–107, Biuro PASCAL, Warszawa 1994.

[8] Evans A.S., Langdon T.G., *Structural Ceramics*, [in:] *Progress in Materials Science*, Chalmers S., Christian J.W., Massalski T.S. [Eds.], 21, Pergamon Press, 1976, 171–441.

[9] Ranachowski P., Rejmund F., Pawełek A., Piątkowski A., *Structural and acoustic investigation of the quality and degradation processes of electrotechnical insulator porcelain under compressive stress*, Proc. of AMAS Workshop on Nondestructive Testing of Materials and Structures II NTM' 03, pp. 179–196, IPPT PAN, Warszawa 2003.

[10] Ranachowski P., Rejmund F., Pawełek A., Piątkowski A., *Studies of cordierite material under compressive load at different temperatures and after thermal shock* [in Polish], Ceramics, **89**, 101–115 (2005).

[11] PN-EN 1330-9, October 2002, Nondestructive Testing – Terminology – Part 9: Terms used in acoustic emission testing.

[12] Richerson D.W., *Modern Ceramic Engineering, Properties, Processing and Use in Design*, CRC Taylor & Francis, Boca Raton London, New York 2006.

[13] Malecki I., *Physical Foundations of Technical Acoustics*, Pergamon Press, Oxford – Braunschweig, Chapter 3, 1969.

[14] Ranachowski P., Rejmund F., Ranachowski Z., Pawełek A., Piątkowski A., *Research of degradation processes of insulator porcelan C 130 type*, Archives of Acoustics, **33**, 4, 117–122 (2008).

[15] Rios P.R., *Abnormal grain growth development from uniform grain size distributions due to a mobility advantage*, Scripta Materialia, **38**, 9, 1359–1364 (1998).

[16] Ranachowski P., Rejmund F., Jaroszewski M., Wieczorek K., *Study of Structural Degradation of Ceramic Material of Insulators in Long Term Operation*, Archives of Metallurgy and Materials, **54**, 1, 205–216 (2009).