Zbigniew RANACHOWSKI, Feliks REJMUND, Przemysław RANACHOWSKI
INSTYTUT PODSTAWOWYCH PROBLEMÓW TECHNIKI PAN, WARSZAWA

Mechanical-acoustic method used to estimate porcelain materials degradation

Doc. dr hab. inż. Zbigniew RANACHOWSKI

Kierownik Pracowni Analizy Sygnałów Emisji Akustycznej. Autor i współautor ponad 50 publikacji z dziedziny pomiarów i analizy sygnałów emisji akustycznej, konstruktor aparatury pomiarowej. Członek Zarządu Polskiego Towarzystwa Akustycznego

e-mail: zranach@ippt.gov.pl

Dr Przemysław RANACHOWSKI

Adiunkt w Pracowni Analizy Sygnałów Emisji Akustycznej. Autor i współautor ponad 50 publikacji z zakresu akustycznych badań materiałów, zwłaszcza ceramicznych tworzyw elektrotechnicznych. Członek Komisji Rewizyjnej Warszawskiego Oddziału Polskiego Towarzystwa Akustycznego.

e-mail: pranach@ippt.gov.pl

Doc. dr hab. Feliks REJMUND

Zakład Akustyki Fizycznej. Autor i współautor ponad 100 publikacji z zakresu akustyki fizycznej oraz ultradźwiękowych badań materiałów i procesów fizycznych. Przewodniczący Warszawskiego Oddziału Polskiego Towarzystwa Akustycznego.

e-mail: freymund@ippt.gov.pl

Abstract

This paper presents the results of acoustic emission (AE) measurements of samples under slowly increasing compressive stress. Additional research, concerning structure and parameters of the material, was performed using microscopic method. The objects of investigation were samples of porcelain material C 120 and C 130 types. These aluminous porcelains find wide application in modern electrotechnical engineering. The aim of study was recognition of stages of degradation processes of the porcelain structure. The analysis of mechanical-acoustic characteristics pointed out diversified strength and complicated mechanism of material degradation. The effectiveness of structural reinforcement of aluminous porcelains was ascertained. On the basis of AE measurements of compressed samples the successive stages of structure degradation have been distinguished. Detailed microscopic analysis enabled to specify the processes of the gradual growth of microcracks and decohesion of the ceramic body.

Keywords: porcelain materials degradation, acoustic emission measurements, compressive stress.

Mechaniczno-akustyczna metoda badań degradacji tworzyw porcelanowych

Streszczenie

W pracy przedstawiono wyniki badań emisji akustycznej (EA) próbek poddanych wolno narastającemu naprężeniu ściskającemu. Uzupełniające badania strukturalne wykonane zostały metodą mikroskopii optycznej. Przedmiotem badań były próbki tworzyw porcelanowych rodzaju C 120 i C 130. Najważniejszym celem pracy było rozpoznanie etapów degradacji struktury tworzyw. Badania wykazały znaczne zróżnicowanie wytrzymałości próbek oraz złożony, kilkuetapowy mechanizm degradacji struktury porcelany. Opisane etapy niszczenia struktury próbek znajdują odniesienie w wieloletnich procesach starzeniowych tworzywa izolatorów w eksploatacji. Zastosowana mechaniczno-akustyczna metoda pozwoliła na wyróżnienie poszczególnych etapów degradacji badanych struktur. Udokumentowano proces powstawania, rozwoju i łączenia się pęknięć, które stopniowo doprowadzają do zniszczenia badanych próbek.

Słowa kluczowe: degradacja tworzyw porcelanowych, badania emisji akustycznej, naprężenia ściskające.

1. Introduction

The method of acoustic emission (AE) is a valuable tool when used for monitoring internal structural changes in ceramic materials. Acoustic technique is suitable for the investigation of the destruction of ceramic materials, due to the fact that initiation and growth of microcracks belong to the main sources of AE signals. This method allows to obtain numerous data concerning the dynamic processes occurring during change of mechanical, thermal or thermo-mechanical stresses [1, 2]. This is the more essential 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.

Investigation of ceramic aluminosilicate and of corundum materials allowed to state that the number of AE events is a good measure of the intensity of cracking which constitutes the microscopic process of the destruction of a sample [2, 3]. The correlation between the rate of a fissure growth and the rate of AE events is particularly useful. Recording of this AE descriptor allows monitoring the destruction process of the material under mechanical load. The events rate and the energy of AE signals, however, are not a linear function of changes of the mechanical or thermal stresses. The velocity of these changes is an additional factor influencing the acoustic activity, which is difficult to define quantitatively. The measurement of the AE events rate as a descriptor, at a slow increase of mechanical load (of the order of 0.01 mm/min) allows, however, to a great extent to make the AE investigations independent of the influence of other factors on the degradation process of the material.

Examination of alumino-silicate 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, which are the cause of mechanical degradation of the material. As it was stated, 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) [3]. The authors found as well good correlation between the processes of material structure degradation, mainly connected with microcracks development, and the AE activity represented by the effective value of AE signal (RMS).

There exist serious analogies between the effects of many years long exploitation under working load applied to the object and material degradation during compressive stresses in a relatively short lasting laboratory test. 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 described in papers [4, 5].

The authors are developing method of mechanical-acoustic testing of the ceramic materials. This method, together with comparative microscopic analysis of material structure, was employed for investigation of corundum material [6] and samples of the porcelain. Examinations performed on the electrotechnical porcelain C 120 kind had special importance. Comparing the structural degradation of the material of operated insulators and laboratory compressed samples, significant similarity was established [4]. Structural effects of slowly increasing compressive load applied to the material and aging processes, being result of many years of exploitation on power line, appear similar.

In the paper [5] the authors presented experimental results of study obtained on small, specially prepared samples of C 130 porcelain, containing structural defects of different intensity. That research was intended to determine influence of the structural faults on acoustic and mechanical properties of the samples. Another study was carried out on the specimens cut off from high voltage line insulator, made of the same material.

2. Measuring set-up

Mechanical-acoustic tests were carried out using specially constructed two-channel measuring system - Figure 1. The mechanical channel contained testing machine INSTRON with computer control. The steel base, on which the sample was placed, functioned simultaneously as an acoustic waveguide. Velocity of the traverse of the machine equal to 0.02 mm/min was applied. During registering the load acting on the sample, AE descriptors were recorded. The acoustic measurement path contained a broad band transducer WD PAC type (passband 80÷1000 kHz), preamplifier, AE analyser and a computer. Total amplification equal to 80 dB and the threshold voltage of the AE analyser (discrimination level) 1 V were applied. The rate of counts, the events rate and the energy of AE signals were recorded. As it has been mentioned above, the most valuable information for the evaluation of the examined processes of material degradation is offered by AE events rate and effective value of signal – AE RMS.

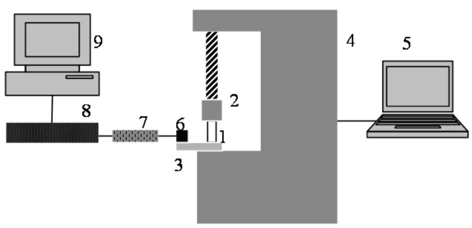

Fig. 1. Scheme of a double-channel measuring system for mechanical-acoustic investigation of the porcelain samples: 1 – specimen, 2 – traverse of the testing machine, 3 – steel base functioning as AE waveguide, 4 – testing machine INSTRON, 5 – computer controlling INSTRON machine, 6 – AE transducer, 7 – preamplifier, 8 – AE analyser, 9 – computer recording AE descriptors

Rys. 1. Schemat dwukanałowego systemu pomiarowego do badań mechano – akustycznych próbek porcelanowych: 1 – próbka, 2 – poprzecznica maszyny wytrzymałościowej, 3 – podstawa stalowa działająca jako falowód emisji akustycznej (EA), 4 – maszyna wytrzymałościowa INSTRON, 5 – komputer sterujący maszyną, 6 – przetwornik EA, 7 – przedwzmacniacz, 8 – analizator EA, 9 – komputer rejestrujący deskryptory EA

3. Research of porcelain C 120 type

Specimens of C 120 type porcelain to be examined were obtained using technology typical for the production of long-rod ceramic insulators. The plastic method of forming and a large chamber furnace for firing the elements, having the shape of rolls with the diameter Φ=8 mm, were used. For the purpose of the investigation a group of samples with the length l=9 mm was cut from the rolls. Both frontal planes were polished to obtain plane and parallel surfaces with the accuracy of 0.1 mm.

Fife specimens were loaded till complete destruction. In the case of three successive ones, the increase of loading was stopped at different stresses from 100 to 300 MPa. The last three samples were loaded to a value preceding the rapid increase of AE activity connected with a process of decohesion, about 400 MPa. Samples, which did not undergo failure, were used for structural research.

Acoustic-mechanical investigation of a group of porcelain samples enabled to state a general good repeatability of the results. The registered courses of AE descriptors revealed the presence of two stages of acoustic activity. The first comprises the extent of loading from about 30 to over 180 MPa. For most samples this stage can be divided into two intervals. The former one shows maximum at the stress of about 70 MPa, the latter one – at about 140 MPa. The first stage of acoustic activity, defined as a preliminary one, corresponds to the earlier phase of development of defects. They result from the gradual relaxation of stresses formed in the material as a consequence of the technological production processes. As it has been evidenced by the structural investigation, these effects are similar to the development of microcracks which are the consequence of increasing degree of the ageing processes. However, the preliminary stage of acoustic emission shows, for the particular samples, considerable differences concerning, first of all, the quantity of signals of the rate of events.

After a short reduction of acoustic activity, at the stresses of about 260 MPa, there begins the second stage lasting in principle until the sample destruction. This stage may be also divided into two intervals of much greater AE intensity and better repeatability than in the case of the preliminary stage. The first interval, defined as subcritical, corresponds to load within the range of about 260 to 350 MPa. The extreme AE in this interval occurs at the stress of about 280 to 310 MPa. The high level of AE in the subcritical range of loading is connected with the formation of large separations and gradual propagation of cracks towards the sample inside. It has been revealed by microscopic examinations of loaded samples.

The last interval, showing the highest level of acoustic activity, begins at a load by some tens of megapascals smaller than the destructive load and lasts till the sample damages. This interval, defined as the critical one, is characterized by a good repeatability of the level of AE signals. The extent of its occurrence depends on the strength of the particular sample. The breaking stresses for samples loaded till complete destruction were between 379.4 and 428.9 MPa, average value equaled 397,6 MPa. Figure 2 shows the course of the rate of AE events as a function of load for the weakest sample.

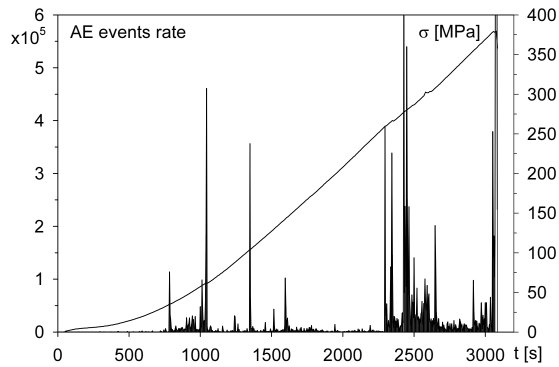

Fig. 2. Example of the course of AE events versus the increase of compressive stress for sample of C 120 porcelain

Rys. 2. Przykład przebiegu zdarzeń EA w funkcji siły ściskającej próbki porcelanowej C 120

Microscopic analysis of the samples, after preliminary stage of AE, revealed the absence of greater delaminations. Occurrence of cracks propagating into glassy matrix was observed only incidentally. There was observed the presence of peripheral cracks around as well as inside quartz grains. Part of them dropped out during polishing. The stress on the grain boundaries and small peripheral cracks were generated already during the process of firing, especially at the stage of cooling. In the case of mullite, precipitates are well connected with glassy matrix and the separations were only incidental [7]. However, large precipitates of mullite contain sometimes fine internal cracks, as a result of the decohesion of needle-shaped crystals.

External loads cause the relaxation of stresses on the grain boundaries – there are formed discontinuities and the existing microcracks become larger. These effects are accompanied by AE signals. The preliminary stage of acoustic emission corresponds to the effects of the propagation of defects of lower threshold energy, introduced at the stage of the technological processes of production. Their increase, however, is rather limited. Figure 3 presents the structural image of the material of sample subjected to stress of 260 MPa.

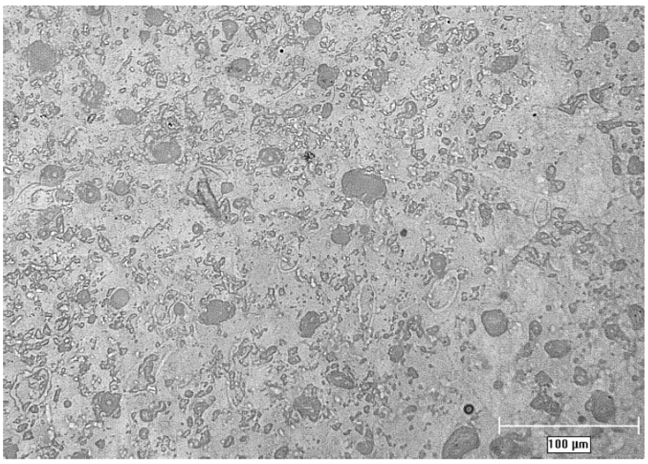

Fig. 3. Structure of C 120 material after preliminary stage of loading, magnified 200x. Majority of quartz grains was crushed out during polishing of the sample surface, remaining are separated from matrix

Rys. 3. Struktura materiału C 120 po naprężeniu wstępnym (wzmocnienie 200×). Większość ziaren kwarcu została wykruszona w czasie polerowania powierzchni próbki, a pozostałe są oddzielone od podłoża

Investigation of samples subjected to stresses in the subcritical range – up to about 400 MPa indicated high degree of the material defectiveness. Independently of the quartz phase, which underwent almost complete destruction, part of mullite precipitates showed internal and less frequently peripheral cracks. In many places the smaller cracks combined, forming the longer ones, particularly in the central parts of samples. Cascade splitting of greater cracks can be observed there – Figure 4.

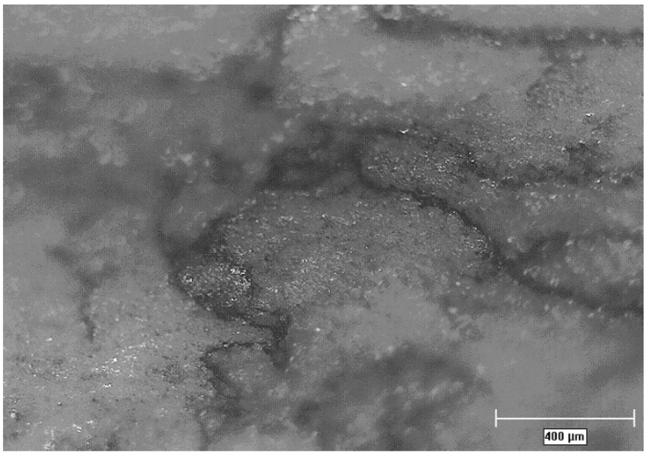

Fig. 4. Cross-section of a sample of C 120 porcelain loaded up to the beginning of critical stage of structure degradation (about 400 MPa), magnification 50x. Branched cracks in the central part of the sample are visible

Rys. 4. Przekrój poprzeczny próbki porcelanowej C 120 obciążanej do początku stanu krytycznego degradacji struktury (około 400 MPa), wzmocnienie 50×. Widoczne są rozgałęzione pęknięcia w centralnej części próbki

However, it was found that even samples with many defects do not undergo with ease disintegration. The cracks are blocked, which points out effective strengthening of the structure by the mullite phase. The analysis of mechanical-acoustic characteristics pointed out not only diversified strength but either complicated mechanism of multiphase porcelain structure. Parameters of the material are the result of its homogeneity, determined by number, size and spatial distribution of mullite precipitates, quartz grains and pores. Propagating cracks had, as a rule, intercrystalline character, which was the consequence of the slow increase of the load (0.01 mm/min) and of phase structure of the electrotechnical porcelain material. Generally degradation of quartz phase is followed by weak AE activity. Stronger acoustic effects are generated by big grains and especially during initiation and growth of cracks in glassy-mullite matrix.

4. Investigation of porcelain C 130 type

Similar investigations were carried out also on specially prepared samples of porcelain C 130 kind. These tests were performed on small specimens containing fine, medium or numerous structural defects [5]. Examination was aimed to recognize influence of technological faults in the material structure on mechanical-acoustic characteristics and mechanical strength of the porcelain samples. It was found that the presence of areas of high internal stresses favors the generation and propagation of cracks, which causes the decrease of the strength of the samples by some tens of percent. This refers to areas with disturbed texture as well as fissures and densely distributed large pores. The non-homogeneities of the distribution of mullite precipitates and particularly of quartz grains are definitely less important. The mechanical strength of the material is determined primarily by the properties of the glassy matrix, containing a lattice of tiny, needle-shaped crystals of mullite and densely distributed fine grains of corundum. Even samples containing significantly defected structure, such as macroscopic textural defects, demonstrated relatively high compressive strength, exceeding 50 % of strength of specimens without defects. Mechanical-acoustic characteristics of the sample without as well as containing serious structural faults are presented in Figures 5 and 6.

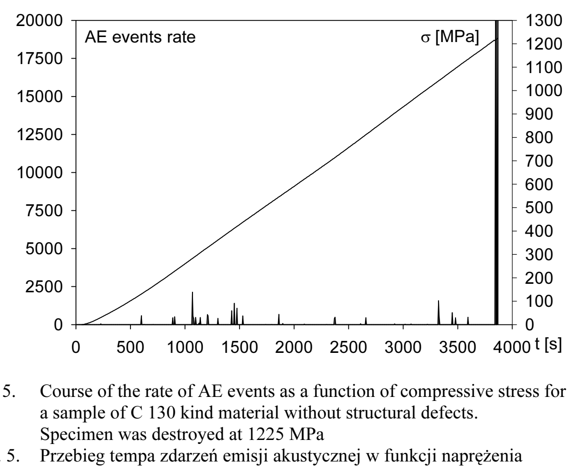

Fig. 5. Course of the rate of AE events as a function of compressive stress for a sample of C 130 kind material without structural defects. Specimen was destroyed at 1225 MPa

Rys. 5. Przebieg tempa zdarzeń emisji akustycznej w funkcji naprężenia ściskającego dla próbki z materiału typu C 130 bez defektów struktury. Próbka została zniszczona przy 1225 MPa

The most recent investigations of the porcelain material C 130 kind were performed on the samples cut off from the rod of typical HV line insulator. Main purpose of performed research was registration of the stages of structural degradation of the insulator's material. There were registered only very weak signals corresponding to the preliminary stage of the material degradation.

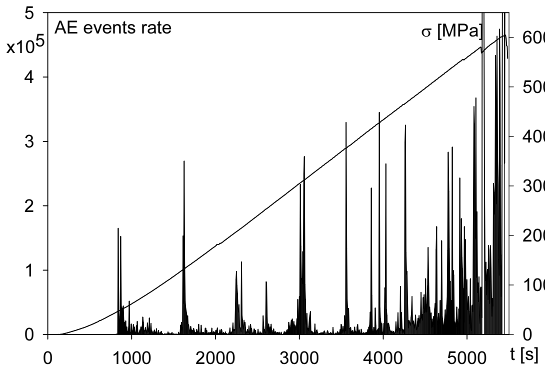

Fig. 6. Course of AE events for a sample C 130 porcelain containing serious textural faults, sample was destroyed at the stress 604 MPa
The continuous acoustic activity and high level of signals was observed
Rys. 6. Przebieg zdarzeń EA dla próbki porcelanowej z materiału C 130 z poważnymi defektami tekstury. Próbka została zniszczona przy naprężeniu 604 MPa

These effects were recognized mainly as the result of separation from matrix grains of quartz. Threshold energy of these AE sources is low and weak signals could be hardly recorded. Next stage takes place at the stress of several hundreds of megapascals. This phase, named as subcritical, corresponds to long lasting effects of cracks development in the agglomerations of corundum grains. Particular grains are separated from matrix due to peripheral cracks growth. While stress increases, more number of grains inside the agglomeration becomes separated and cracks are getting longer. Simultaneously microcracks are initiated and grow inside bigger precipitates of mullite - compare Figure 7.

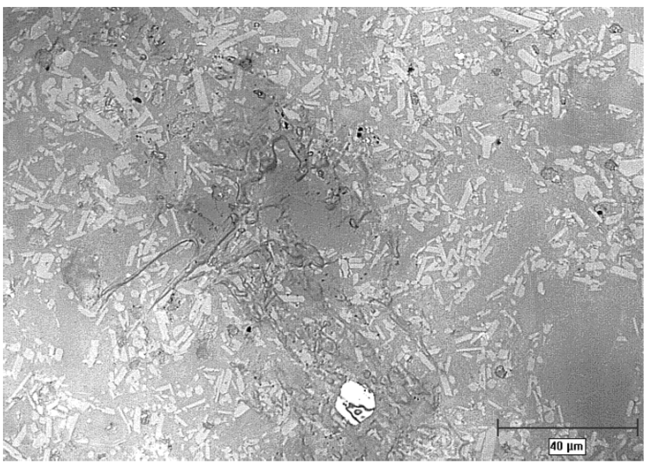

Fig. 7. Cracks of big precipitate of mullite in C 130 type material at the end of subcritical stage of degradation, magnification 500x
Rys. 7. Pęknięcia dużych wtrąceń mullitu w materiale typu C 130 przy końcu stanu podkrytycznego degradacji. Wzmocnienie 500×

Their parts are being surrounded by increasing microcracks. Cracks development in the matrix is however effectively hampered by strong structural reinforcement. This role play densely distributed fine grains of corundum and needle-shaped small crystals of mullite, acting as armament of the structure. AE effects of subcritical phase form single signals and occasionally intervals of continuous acoustic activity at differentiated values of stress.

The AE activity corresponding to the critical stage of material degradation is continuous and has much higher energy than in case of the earlier ones. Large cracks are growing especially in the middle part of samples, where the stresses become cumulated. The critical interval is comparatively short and characterized by a good repeatability of energy level of AE signals.

5. Summerazing remarks

Mechanical-acoustic method, used together with comparative microscopic technique, enables description of the ageing processes in exploited insulator porcelain material. Parameters of the porcelain are the consequence of its homogeneity, determined by texture - number, size and spatial distribution of mullite precipitates, corundum grains – in C 130 porcelain, quartz grains and pores. Very important role play mechanical parameters of glassy-mullite matrix. They are relatively weak in C 120 kind material but high in C 130 porcelain. Microscopic research and nondestructive ultrasonic control revealed acceptable homogeneity of the insulator material and simultaneously occurrence of anisotropy typical for ceramic products formed using screw extrusion method in a vacuum deairing pug mills.

Performed research confirmed the effectiveness of internal reinforcement of the structure of aluminous porcelains. The weakest mechanically element of the structure are quartz grains. Subcritical stage of destruction is connected with long lasting effects of mullite phase damage and in C 130 type porcelain - separation of particular corundum grains from structure of agglomerates. These effects appear especially in the central part of the samples, where the highest concentration of mechanical stress occures. The last, critical stage of structure degradation is followed by strong AE activity. It is mainly connected with generation and growth of elongated cracks in reinforced glassy-mullite matrix of the porcelain material. This effect is strongly hampered in the structure of C 130 porcelain. In the opinion of authors degradation processes, being the result of long-term exploitation of insulator on power line, have similar sequence and character as in case of applied short lasting measurement. Researches of insulators and samples made of C 120 material appear to point out such similarity [4].

6. References

[1] Ranachowski J., Rejmund F.: Emisja akustyczna w ceramice technicznej, in: Emisja Akustyczna - Źródła Metody Zastosowania, red. Malecki I., Ranachowski J., wyd. Biuro PASCAL, Warszawa, 1994, 55-107.

[2] Ranachowski J., Malecki I.: Możliwości oceny pseudoplastyczności materiałów ceramicznych metodą emisji akustycznej, in: Nowe Kierunki Technologii i Badań Materiałowych, Praca zbiorowa pod red. Ranachowski J., Raabe J., Petrovski V., Wydawnictwo IPPT PAN, Warszawa, 1999, 35-46.

[3] Evans A. S., Langdon T. G.: Structural Ceramics, in: Progress in Materials Science, red. Chalmers S., Christian J. W., Massalski T. S., vol. 21, Pergamon Press, 1976, 171-441.

[4] 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, Conference Proceedings 4 AMAS Workshop on Nondestructive Testing of Materials NTM'03, Warsaw, IFTR PAS, 2003, 179-196.

[5] Ranachowski P., Rejmund F., Pawełek A., Piątkowski A.: Mechanical-Acoustic and Structural Investigations of Degradation Processes of Aluminous Insulator Porcelain C 130 Type, Archives of Metallurgy and Materials, No. 52, 2007, 641-654.

[6] Ranachowski P., Rejmund F., Pawełek A., Piątkowski A.: Mechanical-acoustic study of degradation processes of ceramic structure ilustrated by corundum material, Przegląd Elektrotechniczny, Materiały IX Ogólnopolskiego Sympozjum Inżynieria Wysokich Napięć IW-2008, in print.

[7] Carty W., Senapati U.: Porcelain – Raw Materials, Processing, Phase Evolution and Mechanical Behavior, J. Am. Ceram. Soc., 81 [1], 1998, 3-20.

Artykuł recenzowany