ARCHIVES OF ELECTRICAL ENGINEERING VOL. 60(2), pp. 109-128 (2011)

DOI 10.2478/v10171-011-0011-x

Aluminous porcelain degradation study using mechanoacoustic and microscopic methods

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(Received: 02.07.2010, revised: 06.12.2010)

Abstract: This experimental paper comprises the results of acoustic emission (AE), microscopic and ultrasonic measurements of samples subjected to slowly increasing compressive stress. On the basis of conducted measurements the successive stages of the material structural degradation have been recognized. The objects of study were samples made of C 120 aluminous porcelain. The investigated material has found at present the application in the fabrication of technical elements like overhead power line insulators. In the case of such objects, not only high mechanical strength, but especially elevated durability as well as operational reliability are required. The expected "life time" of net insulators during exploitation is about 40 years. The analysis of obtained mechano-acoustic dependences pointed out a complex mechanism of degradation of the material. Microscopic investigation of samples, which were stressed to different levels of load, enabled to specify the development of gradual growth of microcracks and successive crushing out of elements of the structure. These processes appear to be similar to the ageing processes occurring in the material during long period of exploitation under a working load. Three stages of the structure degradation were distinguished. 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 the destructtion of samples. Its range is relatively narrow and reveals the AE activity of high energy. The effectiveness of dispersive and fibrous reinforcement of modern aluminous porcelain C 120 type has been described. Structural strengthening by corundum grains and mullite needle shaped crystals improves mechanical parameters and distinguishes this material from typical aluminosilicate ceramics. The presented results enable drawing up the conclusions concerning the resistance of investigated material to the ageing degradation process development during long term operation.

Key words: aluminous porcelain, ceramic insulators, insulator testing, acoustic emission, microscopic analysis, structural degradation

Introduction

The evaluation of operating time of the ceramic materials is based mainly on the analysis of the formation and development of ageing effects in their structure. The essence of ageing degradation is a gradual expansion of the already existing microcracks and the formation of new ones under the influence of mechanical stresses occurring in the material. The total stresses represent the sum of internal stresses and the stresses induced by the external factors [1, 2]. The internal stresses are created during the technological production processes. These stresses are formed on the micro scale e.g. on the boundaries of quartz grains and glassy matrix, on the semi-macro scale – the result textural anisotropy and on the macro scale – between the internal and the external regions of the object. An object in operation is subjected to considerable exploitation static stresses, as well as, additionally, dynamic loads, which are especially dangerous. These stresses, when added to the internal ones, accelerate the ageing processes. An additional factor contributing to the propagation of microcracks are the temperature changes in the body, attaining within 24 hours even as much as 45°C [3].

The most important factor, responsible for the 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. The internal stresses in the micro-areas are located in the brittle medium. The only way of their relaxation is an increase of already existing or the initiation of new microcracks. Thus, relaxation of stresses is connected with a decrease of mechanical strength of the material. However, the object in operation is subjected to constant external load. Consequently, the growth of microcracks causes a slow decrease of the object cross-section area, which effectively withstands the acting stresses. So, in the material under load there are internal stresses, which induce the increase of microcracks. The development of microcracks causes the degradation of parameters of the material – ageing effects.

From the reports concerning an older type of C 120 insulator porcelain, it has been known that about 20 years long period of exploitation causes about 12% decrease of the mean mechanical strength of insulating material. A much worse result is that the dispersion of the strength of the exploited insulators is nearly 2 times greater than that of new elements. After 35 years of operation, the mean mechanical strength is 18% lower and the dispersion of strength is almost 3 times higher [4, 5]. In the case of ceramic insulators, the degradation of mechanical and electric parameters is of great importance, because it decreases the reliability of the power supply. The experience obtained during the exploitation of older type insulators has revealed a relatively quick development of the ageing processes [1, 4-6]. This refers to the objects being in operation for some decades of years on domestic and foreign power lines and stations. The factor which had essential influence on the degradation of the material of older type, in the process of time, was a high content of quartz, exceeding 20%. This component, present often in the form of bigger grains, caused serious internal stresses in the porcelain body. The quartz phase sometimes showed also a weak joint with precipitates of needle-shaped mullite. An additional problem was the dispersion of the properties. It resulted from insufficient repeatability of parameters of technological processes, which was observed still in the nineteen-eighties.

Different investigations showed that the parameters of the insulator ceramic material seriously deteriorate after a long period of service. This applies particularly to the rods of line insulators, but also to the post insulators, whose porcelain demonstrated significantly worse properties. In the case of post insulators, internal stresses had a crucial influence on the degradation processes. The stresses were especially connected with the presence of quartz phase in the porcelain structure. The degradation processes in the rod area of line insulators were mainly the result of the service load, but also the ageing played an important role. It was indicated by the number of breakdowns – similar for strain and suspension insulators. Technological defects, however, proved to be the primary cause of damage. The material of the domestic insulators showed high diversity of its phase composition and parameters. Also the ageing contributed to the variation of the material properties. In comparison with the line insulators, the porcelain of the post objects had generally worse parameters. Surprisingly, serious differences were also found within groups of insulators of the same type [6].

The investigations have widely confirmed the limited resistance of the C 120 material to degradation. On the basis of different research and the operational data, its service life was estimated at maximum 35 years, provided the insulator does not contain any significant inhomogeneities or technological defects [4-7]. This porcelain material has at present wide application in the production of reliable electroinsulating elements of power systems. Line and station medium voltage (MV) insulators, hollow insulators as well as bushings are produced using this kind of ceramic material.

In this work a modern C 120 porcelain material has been examined. Its internal structure, phase composition and operating parameters are different than in the case of traditional – old type aluminous porcelain. The composition of raw materials was changed and technological processes were modified. The aim of study was to describe its structure and recognize successive stages of the material degradation. On the basis of obtained results the authors attempted to draw up conclusions concerning the resistance of investigated material to the ageing degradation processes. The following problem was the comparison of structural composition, mechanical parameters and especially the resistance to degradation of the new material with a typical insulator porcelain of the same kind.

Preparation of samples

The study of aluminous porcelain was carried out on specimens cut out from domestic MV line insulator LP 60/8, produced in 1999 – Fig. 1. The traditional process of production of long rod insulators consists of many stages [2, 8, 11]: selection of components, control and preparation of raw materials (weighing and milling), plasticization of raw material (mixing with water,

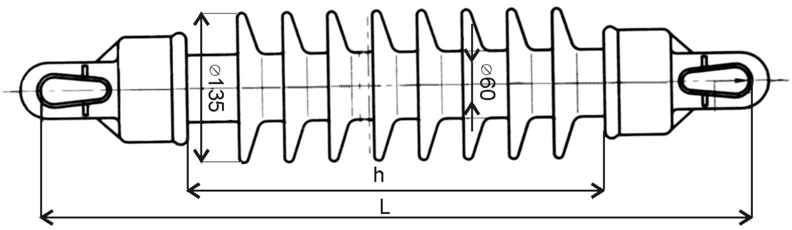

Fig. 1. Diagram of LP 60/8 insulator. Length h = 370 mm, L = 635 mm, diameter of the rod ϕ = 60 mm, diameter of the shed ϕ = 135 mm, leakage path – 800 mm, number of sheds – 8, weight – 9.0 kg, nominal tensile strength – 60 kN

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 dimensions of samples, cut off from the rod of insulator were 8 × 8 × 10 mm. The surface of the samples, especially on bottom and top, were precisely ground to obtain parallel and smooth planes of the order of 0.05 mm.

The microscopic analysis required a special procedure of sample surface preparation. Taking into account acting of compressive forces on the samples, examined after mechanoacoustic tests, a special care in the treatment of samples at each stage of preparation and the study was required. The applied procedures should not change the true image of the effects of degradation. Microscopic analysis of ceramic samples, included the quantity and spatial distribution of particular phases and pores as well as the presence of all kinds of heterogeneities and defects.

To prepare the surface for the microscopic examination a modern method, defined as mechanochemical polishing, is commonly used. The surface microroughness obtained using this method is at the level of nanometers. The colloidal solution of silica (SiO_2), with particle size of not more than 50 nm, plays role of the mechanical polishing medium, while the sodium chlorate (NaClO) is usually used as a chemically active agent. The applied abrasive slurry has a reaction pH equal to 13.5. The process is performed on special polyurethane pads with high porosity. The polishing rate of ceramic materials is about 1 μm/min. Process of mechanochemical polishing depends on large reduction of forces of chemical bonds in the area of several atomic layers, and next a mechanical removal of weakly bounded layer. By using this polishing technique, obtained surfaces, even of highly defected or aged ceramic materials, are very little changed (distorted). The delicate treatment during mechanochemical removal of successive layers of material enables also disclosing even subtle effects of ageing process. For the aluminosilicate materials, traditional grinding and polishing on diamond abrasive powders is not used nowadays. After careful cutting with a saw containing internal diamond edge, a defected layer has only about 100 μm. Then, without grinding, using directly mechanochemical polishing, layer of about 200 μm is removed. Although this method is modern and considerably limits the introduction of additional surface defects, in the case of samples tested using mechanoacoustic method it has turned out to be often insufficient. During mechanochemical polishing, elements of structure weakly integrated with the matrix, undergo crushing out. A good example of such effect are the particles of porcelain cullet, which do not melt on the surface during the process of firing (sintering) and are weakly connected with the glassy matrix.

The proper 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. of Struers company production) is poured over the tested samples to congeal. The two-stage process of polishing is performed next. The top layer of resin is removed in the beginning. The first – coarse stage of polishing is performed on a hard polishing disc, using silica suspension with grain sizes ranging from 10 to 5 μm. The second – final stage is conducted on a soft polishing cloth. Grains in suspension have sizes from 5 to 0.25 μm. Only the application of such procedure enables avoiding the introduction of additional defects and minimizes the effect of crushing out of weakly bounded grains or particles. The microscopic analysis of surfaces of samples, prepared in such a way, showed the presence of cullet in the porcelain structure, which could not been detected after typical mechanochemical polishing. As a result quantity of crushed out elements of the structure has been reduced by over 40%. The phase composition of tested material is presented in section *Material structure*.

Ultrasonic study

The acoustic method is based on the dependences between the parameters of waves propagation and the properties of the medium, in which the waves propagate. In the 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 of inclusions. Among the possible applications of the ultrasonic method, the elastometry is one of the most important methods. 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 such as Young's modulus E and Poisson ratio ν [9]:

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

$$\nu = (c_L^2 - 2c_T^2)/2(c_L^2 - c_T^2). \tag{2}$$

The attenuation is an additional significant ultrasonic parameter, which above all allows evaluating the progress of the degradation processes in the ceramic material. The ultrasonic signal amplitude changes and pulse deformation are the results of the energy dissipation. This effect is due to the existence in the material of the numerous structural inhomogeneities, such as micro-cracks, frequently spaced pores, distorted texture as well as the areas, where mechanical stresses appear. The presence of the network of cracks causes especially strong increase of the attenuation. Amplitude attenuation coefficient α determines the relative amplitude loss per unit path travelled by the wave. In the case of a plane wave, propagating in the direction x, the following equation takes place [10]:

$$\frac{dA}{A} = -\alpha\, d\,x, \tag{3}$$

where: A – amplitude of elastic wave, dA – change of amplitude for the distance dx, symbol minus denotes that amplitude value goes down.

After integration of the equation (3) there is obtained formula:

$$A = A_0 \exp(-\alpha x), \tag{4}$$

where A_0 is initial value of amplitude for $x = x_0 = 0$.

Taking into account, that wave intensity I is proportional to the square of amplitude, the following dependence can be obtained:

$$I \approx A^2 = A_0^2 \exp(-2\alpha x). \tag{5}$$

Wave attenuation amplitude coefficient is a characteristic parameter of medium, like the velocity of propagation of elastic waves and can be directly measured. The value of coefficient α (in dB/cm units) can be calculated from the formula:

$$-\alpha = \frac{8.686}{l} \log \frac{A_2}{A_1}, \tag{6}$$

where: A_1 denotes the amplitude of first reflected signal, A_2 – the amplitude of second reflected signal, l – the distance passed by the signal between the first and second reflection. Considering inequality $A_2 < A_1$ symbol minus is used.

The 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 the use of the screw extrusion method in the vacuum deairing pug mills [8, 11]. The ultrasonic examination of acoustic properties of the tested material revealed better parameters than in the case of typical ones – Table 1. Velocities of the longitudinal − c_L and the transverse − c_T waves, measured along the lengthwise axis of the insulator, were equal to 6420 m/s and 3780 m/s, respectively. The calculated values of Young's modulus E and Poisson ratio ν using formulas (1 and 2), were 86 GPa and 0.23 respectively, at the density of the material $\rho = 2.44 \pm 0.02$ g/cm^3. The uncertainty of measurements for c_L and c_T was ±30 m/s and ±40 m/s respectively, whereas for the calculated value of Young's modulus it was about ±2.0 GPa. The attenuation coefficient, calculated from the equation (6), had typical values in the range 0.6 ÷ 1.0 dB/cm. Uncertainty was equal to ±0.1 dB/cm. In the case of typical material of C 120 kind of domestic HV line insulators, the measured parameters fitted within the ranges: 5790 ÷ 6180 m/s for c_L, 3410 ÷ 3660 m/s for c_T and 69 ÷ 79 GPa for E value ($\rho \approx 2.41$ g/cm^3) [6, 17].

Table 1. Selected parameters of C 120 type porcelain of typical domestic HV line insulators and examined samples from MV insulator

Parameter – symbol and unit	Typical insulator material	Tested material
Density ρ [g/cm^3]	2.41	2.44
Young modulus E [GPa]	69 ÷ 79	86
Poisson ratio ν	0.23	0.23
Wave velocity c_L [m/s]	5790 ÷ 6180	6420
Wave velocity c_T [m/s]	3410 ÷ 3660	3780
Attenuation coefficient α [dB/cm]	0.4 ÷ 1.0	0.6 ÷ 1.0

Mechanoacoustic method and measuring set

The method of acoustic emission (AE) is a valuable scientific tool when used for monitorring internal structural changes in ceramic materials. This technique allows obtaining numerous data concerning the dynamic processes occurring during change of mechanical, thermal or thermo-mechanical stresses in the materials. The initiation and growth of microcracks is one of the main sources of the acoustic events in brittle bodies, which include ceramic materials. It should be emphasized that AE signals appear already at the threshold stresses, when the generation of microcracks in the material cannot be in practice detected by other methods [12].

The examination of samples, which are subjected to mechanoacoustic measurements, using the technique of acoustic emission on a special two-channel measuring system, is a basis of the used method. Specimens of small dimensions are submitted to slowly increasing compressive stress. The geometry of samples has significant influence on obtained results. Surface of specimens should be free from defects, which can initiate cracks development. Top and bottom surfaces, being affected by compressive force, ought to be plane and parallel to each other. If this condition is not satisfied enough, a local fracture and splitting off corners or even the whole wall of sample can occur. There is performed simultaneous registration of the force, and in consequence acting stress in one channel, along with AE descriptors in the second channel. This investigation enables recording and the description correlation between the increasing external load and the processes of structure degradation. Changes of the material structure are mainly connected with formation and growth of microcracks, which is reflected in the acoustic activity. In the consequence, the acoustic method is effective for the investigation of destruction of brittle materials, where the growth of microcracks belongs to the main sources of AE signals. The examination of different ceramic materials enabled to state that the sum of AE events during the loading period is a good descriptor of the intensity of cracking processes, and in the consequence – the degradation of material. A correlation between the rate of the increase of cracks *da/dt* and the rate of AE events *dN/dt* (number of AE events per unit of time) was reported by [13, 14]:

$$\frac{da}{dt} = B\frac{dN}{dt}. \tag{7}$$

The measuring set applied in the examination of ceramic materials was specially designed and constructed. The most important feature of this system was that it has been composed of two independent channels. The mechanical channel contained testing machine INSTRON 3382, controlled by the computer. The stressed sample was placed on a specially prepared base of elongated shape. This element was made of hardened steel and functioned additionally as a waveguide of acoustic signals. The range of the velocities of the traverse of the machine, possible to apply, was from 10^{-3} to 10^{1} mm/min. Taking into account the geometry of specimens, the acting force was converted into stress. Parallel to the measurement of the load acting on the sample, AE descriptors were recorded. The acoustic registration path contained a broad band piezoelectric transducer WD PAC type, placed on the steel base of the testing machine. The passband of transducer ranged from 80 to 1000 kHz. The transducer was connected to a standardized AE analyser and a computer registering acoustic data. The amplification in acoustic channel was equal to 60 dB, one second time interval of summing up the signals was applied. This choice was due to the fact that the force increase registration was performed every one second. The sampling frequency of AE signal was 44.1 kHz. The final analysis of registered AE signals was carried out using standard software made in Institute of Fundamental Technological Research PAS in Warsaw. The measuring set is presented in Figure 2.

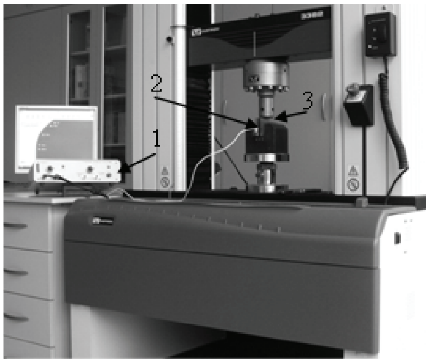

Fig. 2. Two-channel measuring system applied to mechanoacoustic tests of the ceramic samples. There are marked: 1 – AE analyser, 2 – acoustic transducer, 3 – enclosure containing the sample

The velocity of stress growth is an important factor affecting results of mechanoacoustic tests. The AE descriptors of appearing signals are not a linear function of changes of the mechanical or thermal stresses. The velocity of these changes is one of the factors influencing the acoustic activity. This dependence is additionally difficult to define quantitatively. The measurement of AE descriptors, at very slow increase of mechanical load, of the order of 10^{-2} mm/min allows, however, making the mechanoacoustic tests almost independent of the influence of the experimental factors. At a very low velocity of stress increase the process of structure degradation has quasi-static character, which better reflects operational conditions, when a ceramic element is under 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, support the initiation and growth of transglanular cracks, while lower velocities, of the order of 10^{-2} mm/min, favour interglanular cracks increase [15]. In the structure of the aged ceramic material of the objects, especially insulators after a long period of exploitation, interglanular cracks in the matrix are mainly observed [5, 6]. This fact confirms the necessity of application of lower velocities of stress growth in the investigation of degradation processes.

Measurements of acoustic emission have always a comparative nature, because they concern a particular object which is the subject of research. Obtaining of the absolute value of energy of AE event is practically impossible. Acoustic emission analyzers, besides determining of the signals amplitude, are equipped with functional blocks calculating the energy of measured signals or the energy-related functions. The most commonly used solution is electronic conversion of a group of measured signals amplitudes $V(t)$ into the effective value V_{RMS} (RMS − root mean square) over the time period T, according to the formula:

$$V_{RMS} = \sqrt{\frac{1}{T}\int_0^T V^2(t)\,d\,t} \tag{8}$$

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

Descriptors of acoustic emission are registered parallel to mechanical parameters in the second channel of measuring set. In ceramic materials, generally brittle, the recorded acoustic emission is mainly associated with the growth of already existing or initiation of new microcracks. The essential factor in AE measurements is to use such descriptors of signals that contain the most relevant information for the evaluation of tested process. The choice of the optimal descriptor for the definite measurement is determined by the following criteria:

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

Besides the above criteria, an additional, very important factor must be taken into consideration. An acoustic emission descriptor, selected at the research should be optimal for the tested material. As it was established by the authors, the choice of AE descriptor should be different in the case of aluminosilicate materials and oxide ceramics. The aluminosilicate materials such as porcelain, steatite or cordierite, are characterized by numerous signals of lower energy. These signals can be effectively recorded using AE events rate. It is especially important during preliminary and subcritical stages of structure degradation. During the registration, substantial part of the descriptors based on energy of signals can be lost even at low discrimination level. However, during the analysis of mechanoacoustic characteristics of porcelain samples it was stated good usefulness of descriptor connected with the energy of AE events. Recording the energy of events in the interval of one second allows a better interpretation of the stages of structure degradation. In addition, the graphs of this descriptor in logarithmic scale allow obtaining a clear presentation of the whole process – Figures 9 and 10. In the case of corundum material, the authors observed an appropriate correlation between the structure degradation, mainly connected with the development of intergranular cracks, and the effective value of AE signal V_{RMS} – equation (8). This is connected with high mechanical strength of the corundum and a relatively high energy of generated AE signals. The use of RMS rate is more effective than the rate of AE events in the case of the oxide material.

Material structure

Samples, which preparation was described above, were analyzed by using optical microscopy method. The microscopic phase analysis of examined samples of the insulator material revealed generally satisfying homogeneity along the length of the rod (in macro-scale) as well as in the semi-macro scale. Grains of corundum, pores and particles of cullet were uniformly distributed in a glassy matrix in the micro-scale. The typical image of the material structure is presented in Figure 3.

A fine-grained corundum constituted the important crystal phase, in the amount of 7.5% by volume.

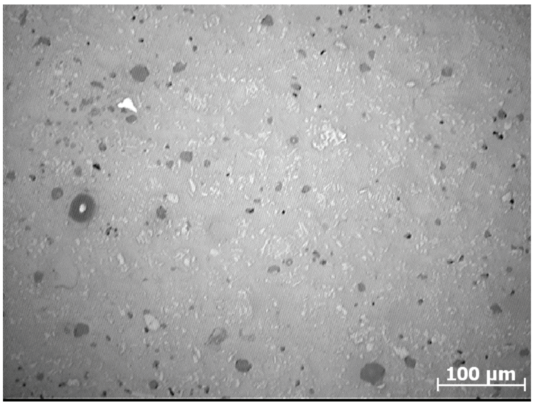

Fig. 3. Image of the structure of examined insulator material, magnification 200×. Fine, bright grains of corundum (about 7%), a little greater quartz grains (about 8%) and white particles of cullet are visible. Darker precipitates of mullite are almost indistinguishable from glassy matrix. Dark areas of crushed out cullet and quartz as well as fine black pores (below 1%) can be observed

Their size – most frequently below 7 µm – was typical for aluminous porcelain materials, such as their characteristic lengthened shape. Needle-like crystals of mullite formed elongated precipitates, usually 20-30 µm in size. The content of mullite precipitates was about 26%. In the glassy matrix, in amount of 52-55%, several percent of dispersed crystals of mullite were present. The quartz grains, with the diameter from a few to almost 50 µm, occupied 8-9% of the material surface. Their distribution in micro- and semi-macro scales was inhomogeneous. The majority of grains were sufficiently melted at the boundaries and adhered to the glassy matrix. The initial content of cullet was 5%. Approximately a half of it fell out during the preparation of polished sections. The particles of cullet were of diverse size, on average about 6 µm. Their distribution in the material structure was homogeneous. The surface of the cullet particles did not undergo melting during the technological process of firing (sintering) and they were weakly connected with the glassy matrix. Small pores (most frequently below 3 µm) occupied 0.7% of the surface.

The typical C 120 material, especially of the older line insulators, was characterized by a moderate homogeneity – Figure 4. The quartz content ranged usually between 20 and 30%. It often occurred in the form of larger grains, on average about 30 µm. This phase was mainly responsible for internal stresses and the initiation and development of cracks, as a consequence of ageing processes. A significant amount of the quartz grains fell out during the preparation of surface of the samples. The mullite precipitates occupied about 33-35%. Relatively large precipitates, mostly 25÷40 µm, were usually uniformly distributed in the material. They were well bounded with matrix and did not contain internal cracks. The corundum phase was present only incidentally as single small grains and they had not any influence on the material strengthening. The pore content was in the range of 2÷5%. The glassy matrix content in the material amounted to 40÷60% (usually over 50%). The matrix was strongly bounded with the mullite precipitates and contained dispersed crystals of mullite, that could constitute several percent of the total material. The dispersed mullite needle-shaped crystals played an important role as the fibrous reinforcement of the material structure. However, their content was low. Small and very small cracks appeared in the neighbourhood of quartz grains. The material did not contain any cullet.

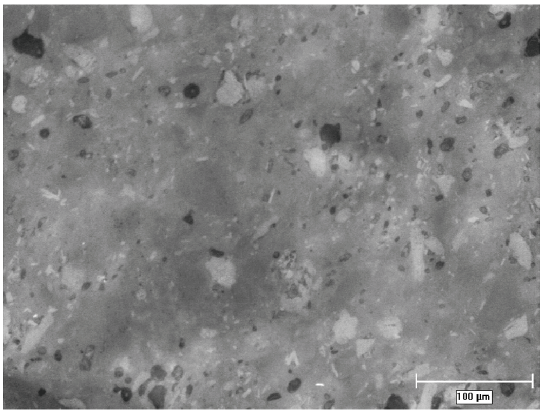

Fig. 4. Structure of typical C 120 kind insulator porcelain, magnification 200×. Bright grains of quartz (about 20%), darker precipitates of mullite (above 30%), black areas of crushed out quartz grains (about 4%) as well as fine round-shaped pores in glassy matrix are visible

Tested material clearly differed from the typical C 120 kind porcelain. Comparison of phase composition of the typical aluminous porcelain and tested material is presented in Table 2. Examined porcelain contained much less quartz, a little less mullite and pores. The material structure included 7.5% of corundum and 5% of cullet, which were absent in a typical porcelain. The amount of glassy matrix was considerably higher. The main difference consisted in the presence of dispersive structure reinforcement. The structural strengthening was represented by fine grains of corundum and more numerous needle-like crystals of mullite dispersed in the matrix, apart from the precipitates. Such phase composition is considered to be stronger and more resistant to ageing processes. It was stated that that structure was intermediate between the typical C 120 material and C 130 kind corundum-mullite porcelain. The considered aluminous ceramic materials are rated among the grain type composites.

Table 2. Phase composition of C 120 type porcelain of typical domestic HV line insulators and examined samples from MV insulator. Data presented in volume percents

Phase component	Typical insulator material	Tested material
Corundum	below 1	7.5
Quartz	20-30	8.5
Cullet	–	5
Pores	2-5	0.7
Precipitates of mullite	30-35	about 26
Matrix	over 40	52.5

Mechanoacoustic measurements of typical C 120 material

The mechanoacoustic method, together with a comparative microscopic analysis of the ceramic structure, were earlier employed to investigate porcelain and corundum materials [16]. The examinations of electrotechnical porcelain C 120 had significant practical importance. By comparing the structural degradation of the material of the insulators removed from service and that of laboratory compressed samples, a close similarity was established. The structural effects of slowly increasing compressive load applied to the material, and the aging processes being the result of many years long service on a power line appear to be similar. The comparative microscopic investigation of compressed samples of aluminous material and the analysis of known mechanism of structural degradation of electrotechnical porcelain [6, 17], enabled the interpretation of obtained mechanoacoustic patterns. On the basis of these results three successive stages of degradation of C 120 type material could be recognized.

The first stage of the material degradation occurs mainly as a result of internal stresses existing in the ceramic body, especially in the micro scale. They were created during the manufacturing processes. The growth of preliminary defects has a relatively low threshold energy and can develop already at lower stresses of the sample. The process of their propagation under exploitation conditions, however, is not rapid and takes years. This stage corresponds to the destruction of quartz grains and the beginning of mullite phase damage. The second effect takes place only in the central part of compressed samples, where the highest concentration of stress is present. The development of such effects in the material of working insulator, under operational stresses – static and especially dynamic – takes place in period between 10 and 20 years. In the case of mechanoacoustic test, the preliminary stage occurs in a wide range of stresses – from a few tens of megapascals to about 200 MPa.

The second stage of the structural degradation corresponds to long lasting development of subcritical defects. The microcracks, initiated at the boundaries of quartz grains or incidentally besides mullite precipitates, propagate in glassy matrix of the porcelain. This is simplified by densely distributed, cracked quartz grains. The subcritical stage of degradation is closely connected with homogeneity of the sample structure in micro- and semi-macro scales. Both stages are strongly influenced by the contents, size and spatial distribution of quartz grains as well as mullite precipitates. The process of decohesion is inhibited. The growth of microcracks is hindered and the latter branch out on the intergranular and phase boundaries. An appropriate amount of energy is needed in order to cross each boundary. Subcritical stage corresponds to a stress range of 250-350 MPa.

When a certain level of load is reached, cracks start propagating rapidly. This final (and the shortest) stage of the degradation process is referred to as the critical one. During this stage

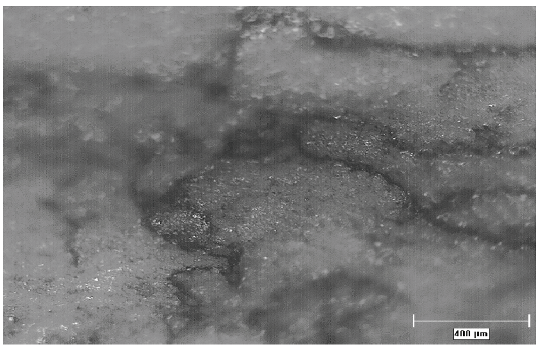

Fig. 5. Image of typical C 120 porcelain structure of the sample loaded up to critical stage of degradation, magnification 50 ×. Branched cracks in the central part of the specimen

single cracks join together gradually and after branching out, they lead to the formation of network cracks – Figure 5. These effects find a strong reflection in the registered AE activity. The critical stage begins at a stress a few tens of megapascals lower than the ultimate load and ends with the failure of the sample. In the case of operating insulator, the final of the aging process is its breakage. A typical mechanoacoustic characteristic of the C 120 material sample is shown in Figure 6.

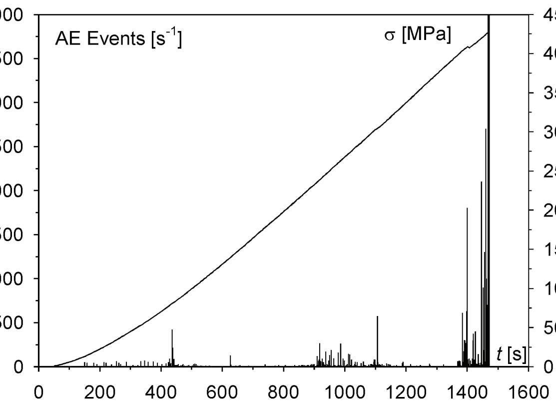

Fig. 6. Representative course of the rate of AE events versus the increase of compressive stress for typical C 120 porcelain sample, which was loaded up to destruction at 429 MPa. The AE signals of the preliminary and subcritical stages are weakly visible because of their lower amplitude

Mechanoacoustic measurements of tested material

The compressive strength of samples, loaded until a complete destruction is presented in Table 3.

Table 3. Compressive strength of the examined samples loaded until destruction

Number of sample	9	8	3	5	1	7	4	10
Compressive strength [MPa]	421	443	469	491	512	557	563	608

The lowest value of strength was unreliable because of surface defects of the sample and was neglected. The mean strength was equal to 520 MPa. This value is relatively very high, compared with the typical strength of C 120 kind material − usually about 400 MPa. The obtained resistance was slightly lower than that of the weaker C 130 type porcelain (about 580 MPa). The relative dispersion of compressive strength was low and equalled 31.7%. Besides the damaged samples, a group of specimens was selected for the microscopic investigation. The compression process of these samples was stopped at different levels of stresses: 100, 250, 460, 521 and 541 MPa. Greater pieces of destroyed samples were also subjected to microscopic study. The applied procedure enabled a detailed study of degradation progress in the porcelain material, subjected to increasing load. The analysis of the results revealed the presence of three stages of the process. The obtained mechanoacoustic characteristics of the particular samples showed considerable differentiation. Figure 7 presents course of the rate of

AE events versus the increase of compressive stress for the sample, which loading was stopped at 541 MPa, just before the destruction. Figure 8 shows the course of the same descriptor for the sample destroyed at stress 512 MPa. However, Figures 9 and 10 present the energy of events in the interval of one second versus stress for the weaker sample destroyed at 469 MPa and for the strongest specimen damaged at 608 MPa.

The first − preliminary stage of material degradation occurs as a result of the internal stressses, created during the manufacturing processes and existing mainly on the micro-scale in the ceramic body. Defects may start to develop at a relatively low energy threshold and under small stresses acting on the sample. The propagation of microcracks in service conditions is slow and takes many years. The preliminary stage of material degradation usually takes place up to about 100 MPa and for some samples only to 60 MPa. This stage is characterized by a low intensity of AE signals and a considerable differentiation among individual samples. The microscopic analysis of surface of the specimen loaded to 100 MPa confirmed that preliminary stage of degradation corresponded to the crushing out a greater part of cullet. The particles underwent fracture and separation from the porcelain matrix, without any recordable acoustic activity.

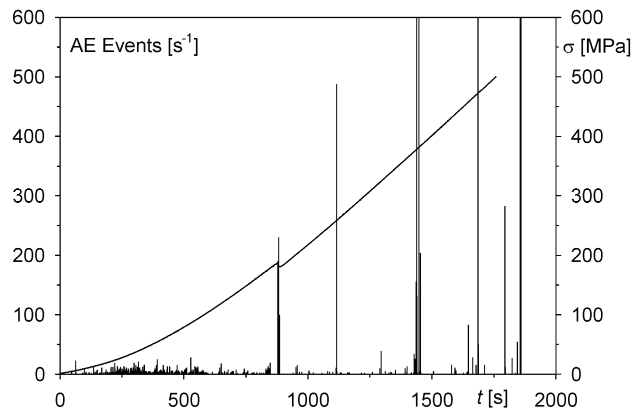

Fig. 7. Course of the rate of AE events versus increase of compressive stress for the tested sample, whose loading was stopped at 541 MPa, just before destruction. Only the preliminary and subcritical stages of degradation in stress range 0 ÷ 538 MPa are displayed. Strong signals of the last – critical stage are not included

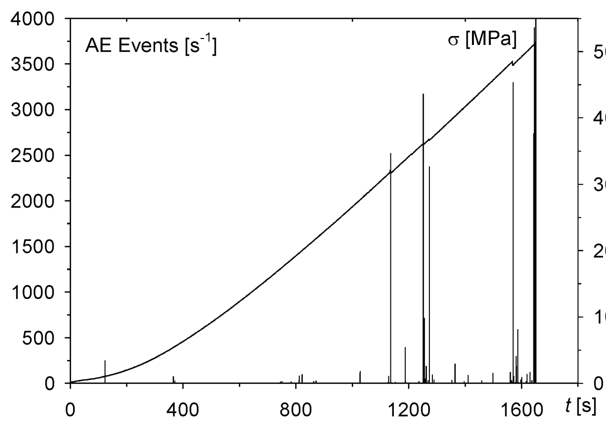

Fig. 8. Course of AE event rate versus increase of compressive stress for investigated sample loaded up to destruction at 512 MPa. The AE signals of the preliminary and subcritical stages are almost invisible because of their lower amplitude

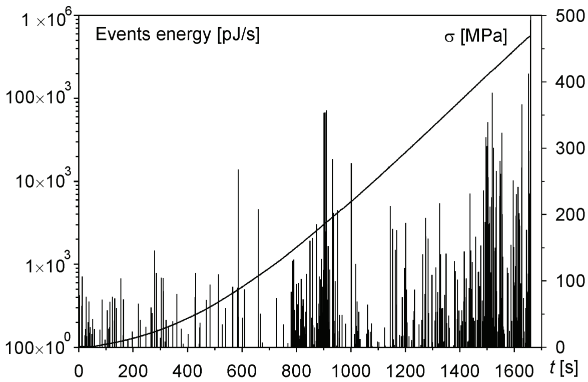

Fig. 9. Course of the energy of AE events in the interval of one second (pJ/s) versus stress for the weaker sample destroyed at 469 MPa. There was applied logarithmic scale for AE descriptor

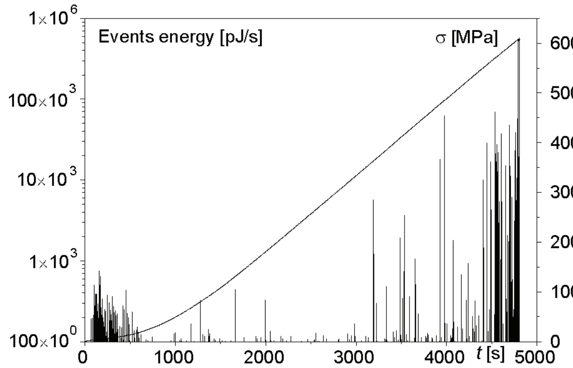

Fig. 10. Course of the energy of AE events in the interval of one second (pJ/s) versus stress for the strongest sample damaged at 608 MPa. There was applied logarithmic scale for AE descriptor

Whereas the degradation of significant part of the quartz phase was the source of AE signals. Cracks initiated and developed mainly in the perimeter of the quartz grains. Internal cracks were observed less frequently. No more than half of the quartz phase and especially small grains of size below 10 μm underwent destruction and crushing out. The degradation did not concern the corundum and mullite phases. The destroyed elements of ceramic structure comprised 3-4% of the surface of compressed samples. Figure 11 presents the material of the sample loaded up to 250 MPa, in which almost only effects of the preliminary stage are visible.

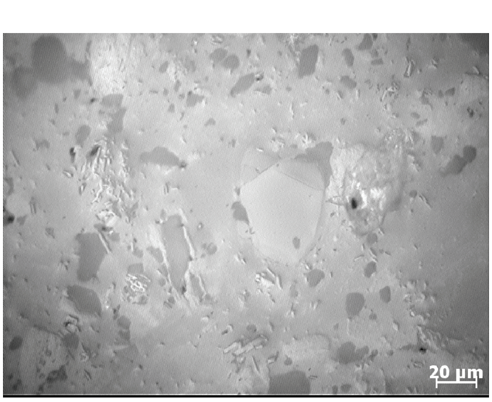

Fig. 11. Representative structure of the sample loaded up to the early sub-critical stage of degradation, magnify-cation 500×. Dark areas remaining after crushed out particles of cullet and quartz grains constitute about 4% of the surface. Fine bright grains of corundum and grey mullite precipi-tates are not affected by degradation

The second – subcritical stage of degradation is closely connected with the homogeneity of the samples structure in the micro and semi-macro scales. The subcritical stage follows the preliminary stage and lasts to the beginning of the critical stage. This phase of destruction varies considerably for particular samples and shows single or groups of acoustic signals of low or moderate level of AE effects. Longer intervals of AE activity occur rarely. The strongest signals follow the fracture and splitting off the walls as well as corners from the sample. During subcritical period further damage of cullet particles (without acoustic effects) and quartz grains (weak AE signals) takes place. Peripheral and internal cracks in grains are created. A slow degradation of mullite phase was registered too. The stresses of advanced subcritical stage, especially in the central section of the samples, caused the initiation of internal cracks and sometimes crushing out parts of precipitates. They were strongly bonded with the glassy matrix and peripheral cracks occurred very rarely. In the case of the samples, stressed up to the end of subcritical stage (460 and 521 MPa), the area of damaged, separated and crushed out elements of structure comprised about 8% of the surface. This value included almost the whole cullet (below 5%), a part of quartz grains (especially of small size) and less than 1% of the mullite phase. Figure 12 presents the structure of the sample containing moderate effects of subcritical degradation. In Figure 13 strongly advanced subcritical effects, in the central part of the sample compressed up to 521 MPa, are visible.

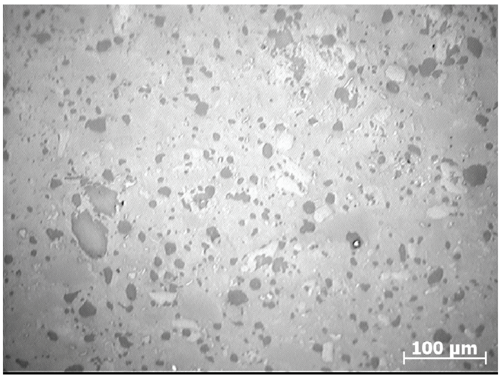

Fig. 12. Image of the structure at the boundary part of the sample stressed up to 460 MPa, magnification 200 ×. Dark areas remaining after crushed out particles of cullet and quartz grains of different size constitute about 7.5% of surface. Almost all bigger quartz grains contain internal cracks. Damage of mullite and corundum phases are only incidental

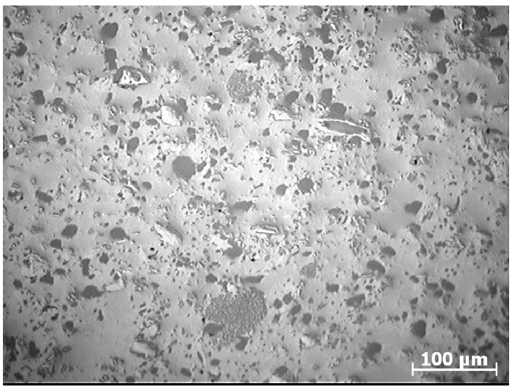

Fig. 13. Image of the structure at central part of the sample stressed up to 521 MPa, magnification 200×. Black areas remaining after crushed out particles of cullet and quartz grains constitute above 8% of surface. Strongly cracked, great and dark precipitates of mullite are visible. Quartz grains contain peripheral and internal cracks. Damages of corundum phase are rare

The last one – critical interval, showing the highest level of the acoustic activity, began at loading from several to dozen or so megapascals lower than the destructive stress for the particular sample. It lasted up to the destruction of the specimen. This interval was characterized by generally good repeatability of the level of AE signals. They had the highest intensity in the whole degradation process. Some samples generated signals from the fracture and splitting off greater pieces of the specimen. A local fracture and splitting off corners and even a wall of the sample could occur – Figure 14.

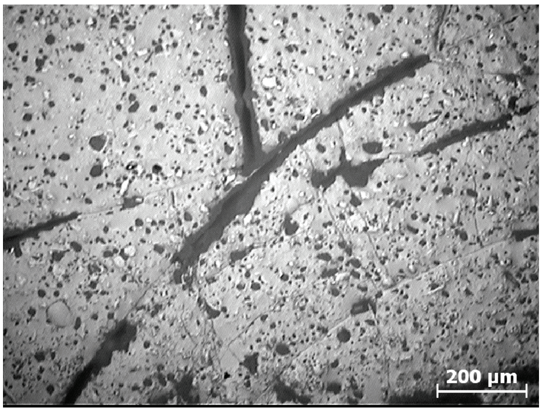

Fig. 14. Image of the structure at the boundary part of the sample stressed up to beginning of the critical stage – 541 MPa, magnification 100×. Lateral wall of sample underwent splitting off. Great cracks in its neighbourhood are visible. Crushed out parts of structure, apart from cracks, comprise above 10% of surface

This effect was visible in the stress curve as characteristic step down (abrupt decrease). During the critical stage, still remaining quartz grains underwent the peripheral and internal fracture. The degradation of the mullite precipitates was continued as well. Grains of corundum were separated from alumina agglomerates. However, such agglomerates were very rarely observed. During the critical stage, just before the destruction, the area of damaged, separated and crushed out elements of structure comprised about 13% of the analyzed surface − Figure 15.

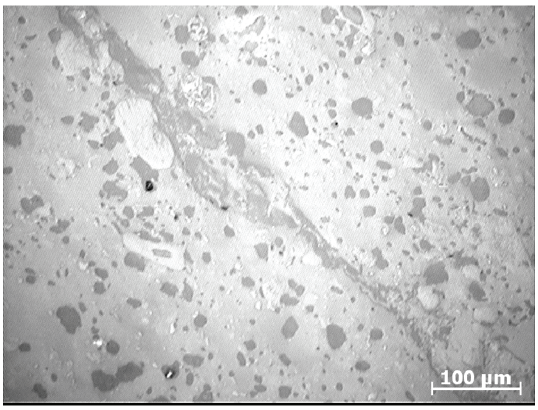

Fig. 15. Image of the structure of the piece of destroyed sample, magnify-cation 200×. Large crack and bright fractured grains of quartz are visible. Damages of mullite phase are moderate and in the case of corundum negligible. Dark areas remaining after crushed out elements of structure exceed 13% of the surface

The sum of crushed out components included almost the whole cullet, about 3/4 fraction of quartz, a small part of mullite as well as corundum. However, the formation and growth of cracks in the porcelain body was the most important and destructive effect, accompanied by strong AE signals. The propagation of cracks was facilitated by previously destroyed elements of structure. These cracks were elongated and in general not branched − Figure 15. They grew initially among the damaged particles of cullet, quartz grains and other crushed out elements of the structure. Similarly, as in the case of C 130 type porcelain, the dispersive and fibrous reinforcement of the material structure hamper their increase.

For that reason cracks were usually unbranched. In the case of typical aluminosilicate ceramic materials, including the C 120 type porcelain, single cracks joined together and even formed a network of cracks. Such effect was observed in the traditional insulator porcelain [6]. At sufficiently high stress, the rapid growth of critical cracks in the porcelain body took place and the sample underwent irreversible destruction.

Final remarks

The results of the mechanoacoustic study showed clear differences in the degradation process of the tested material, when compared with the typical porcelain C 120 kind. This was the consequence of different phase composition, and especially the presence of dispersed strengthening of the material structure. A greater mechanical resistance of mullite precipitates and glassy matrix, which contained scattered grains of corundum and single mullite crystals, was observed. As a result, the resistance to the processes of formation and growth of cracks of the tested material was considerably higher, when compared with the typical porcelain of the same kind.

The microscopic, ultrasonic and mechanoacoustic examination of the tested insulator material showed, that its properties were intermediate between those of typical kind of material C 120 and a much stronger type of porcelain C 130. Like the latter, the tested material contained a dispersed reinforcement of the material structure. Scattered grains of corundum and single crystals of mullite were not as numerous as in the material of C 130 type. However, they constituted the factor which effectively hindered the creation and growth of cracks. In addition, the glassy matrix of the tested material contained more alumina than the typical 120 type porcelain. Consequently, the glassy phase had a higher mechanical strength. It should be noted that the porosity of the tested porcelain was relatively very low. Therefore, the mechanism of the degradation process was similar to that of C 130 type material. It concerned especially the last – critical stage of degradation. Then, at sufficiently high stress, elongated cracks, usually not branched, underwent fast growth and led to the damage of the compressed sample.

The results of numerous tests, including those performed by the authors, proved serious weakening of parameters of C 120 kind material after a long period of work [1, 4-7, 11, 17]. It concerned especially rods of line insulators, but as well post insulators, which porcelain had worse properties. In the case of post insulators, the internal stresses, quartz-related particularly, had a decisive influence on the degradation processes. The ageing effects in the region of rods of line insulators mainly resulted from the operational load. However, quartz-related stresses played an important role as well. It was confirmed by the number of breakdowns – similar of strain and suspension insulators. Nevertheless, technological defects were as a role direct cause of breakdowns. The material of tested insulators demonstrated high diversity of phase constitution and parameters. Ageing processes had further influence on properties variety magnification [4, 5]. Although the variation of properties made the unequivocal assessment of the material of tested insulators difficult, it was possible to draw general conclusions. The investigations fully confirmed the limited resistance of C 120 material to degradation processes.

The structural effects of slowly increasing compressive loading applied to the insulator material, and the ageing processes being the result of many years service on a power line are regarded to be similar [6, 16, 17]. Therefore, these tests can be used to evaluate the operational durability of insulators. On the ground of earlier researches, data from the exploitation and presented examination, operational durability of porcelain C 120 type, can be assumed as limited to no more than 35 years. This period of work can be believable provided that insulator does not contain significant inhomogeneities or technological defects. In the case of C 130 type porcelain "life time" was assessed to be approximately 50 years [7, 11]. On the basis of described examinations, it can be assumed that the insulator made of modern, modified C 120 type material, may operate for about 40 years. The crucial condition for such estimation is the lack of meaningful inhomogeneities or technological flaws in the structure of insulator.

A significant improvement in the properties of C 120 type porcelain was obtained mainly through the use of ceramic alumina instead of metallurgical Al_2O_3 in the raw material composition. A modification of the technological process of insulator porcelain production was also necessary.

Acknowledgment

This work has been financed by the Research Project N N507 598038

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