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## MECHANOACOUSTIC AND MICROSCOPIC STUDY OF ALUMINOUS PORCELAIN RESISTANCE TO STRUCTURAL DEGRADATION

### MECHANOAKUSTYCZNE I MIKROSKOPOWE BADANIA ODPORNOŚCI PORCELANY WYSOKOGLINOWEJ NA DEGRADACJĘ STRUKTURY

The paper presents mechanoacoustic and microscopic testing of degradation processes of modern C 120 electrotechnical porcelain of domestic medium voltage line insulator. Samples of small dimensions, cut off from the rod of insulator, were subjected to compressive loading, with recording of acoustic emission descriptors. Microscopic analysis enabled determining the advancement of degradation effects. Three stages of the structure degradation were distinguished. 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.

*Keywords:* aluminous porcelain, structural degradation, acoustic emission (AE), microscopic analysis

W pracy przedstawiono mechanoakustyczne i mikroskopowe badania procesów degradacji w nowoczesnym tworzywie porcelanowym rodzaju C 120 liniowego izolatora SN. Małogabarytowe próbki, wycięte z pnia izolatora, były quasi-statycznie ściskane z jednoczesną rejestracją deskryptorów emisji akustycznej. Badania mikroskopowe ściskanych próbek wykorzystano do opisu mechanizmu degradacji tworzywa. Wyróżniono trzy kolejne etapy degradacji struktury tworzywa. Stwierdzono wysoką krótko- i długotrwałą odporność materiału izolatora w porównaniu do typowych tworzyw rodzaju C 120. Jest to wynikiem skutecznego dyspersyjnego i włóknistego wzmocnienia struktury czerepu badanej porcelany.

### 1. Introduction

The aluminous porcelain of C 120 type has at present wide application in the production of reliable electroinsulating objects of power systems. Line and station MV insulators, hollow insulators as well as bushings are produced using this kind of ceramic material. The reliability of power supply is determined primarily by the durability, closely connected with the long-term mechanical strength of insulators. That is why these properties are the most important in case of these objects. The evaluation of operating time of the porcelain material is based mainly on the analysis of the formation and growth of aging degradation 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 of textural anisotropy and on the macro scale – between the internal and the external regions of the object. Insulator 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.

From the reports concerning an older type C 120 insulator porcelain, it is known that about 35 years long

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period of exploitation causes significant decrease of the mean mechanical strength of insulating material. Besides that, the dispersion of the strength of the exploited insulators is about 2.5 times greater than that of the new objects [3]. In instance of ceramic insulators, degradation of the 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 [1,3-6]. This concerns 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 large 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, resulting from insufficient repeatability of parameters of technological processes, which was observed still in the nineteen-eighties.

The results of different tests 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, which porcelain demonstrated significantly worse properties. In the case of post insulators, internal stresses had a crucial influence on the degradation processes. The stresses were specifically connected with the 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, as 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 in its phase composition and parameters. Also the ageing contributed to the variation in the material properties. In comparison with the line insulators, the porcelain of the post insulators had generally worse parameters. Surprisingly, serious differences were found within groups of insulators of the same type [3-5].

The investigations have amply 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 [3-6].

## 2. Examined material and ultrasonic control

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. The specimens were cut out from domestic, unexploited medium voltage (MV) line insulator, produced in 1999. 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 type.

Ultrasonic control of the acoustic properties of the tested material revealed better parameters than in case of the typical ones. Velocities of the longitudinal  $c_L$  and the transverse  $c_T$  waves, measured along the lengthwise axis of insulator, were equal to 6420 m/s and 3780 m/s, respectively. The calculated value of Young's modulus  $E$  was 86 GPa at density of the material  $\rho = 2.44 \text{ g/cm}^3$  [7]. The uncertainty of measurements for  $c_L$  and  $c_T$  was  $\pm 30 \text{ m/s}$  and  $\pm 40 \text{ m/s}$  respectively, whereas for the calculated value of Young's modulus it was about  $\pm 2.0 \text{ GPa}$ . In the case of the typical material C 120 type of line insulators the measured parameters fitted within the ranges:  $5790 \div 6180 \text{ m/s}$  for  $c_L$ ,  $3410 \div 3660 \text{ m/s}$  for  $c_T$  and  $69 \div 79 \text{ GPa}$  for  $E$  value ( $\rho \approx 2.41 \text{ g/cm}^3$ ) [4,5].

## 3. Mechanoacoustic method and material structure

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 applied 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 acoustic emission (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. Research of the material degradation on the basis of mechanoacoustic and microscopic methods was the subject of the work presented by the authors in 2009 [8].

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 [5,8].

The specimens, cut off from the central part of the insulator rod, were subjected to mechanoacoustic measurements. Pieces of small dimensions (8x8x10 mm) were put to slowly increasing compressive stress with the velocity  $v = 0.02$  mm/min, with a simultaneous registration of the force in one channel, and AE descriptors in the other one. The investigations enabled the recording and description of correlation between the increasing external load and processes of structure degradation, which are reflected in the AE activity. It was necessary to apply a quasi-static, very slow increase of stress, a precise registration of the AE descriptors and accurate preparation the geometry of the samples [8].

The microscopic phase analysis of examined samples of the insulator material revealed generally satisfying homogeneity along the length of the rod (macro-scale) as well as in 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 was presented in Figure 1.

The important crystal phase constitutes fine-grained corundum, in amount of 7.5% by volume. Needle-like crystals of mullite form elongated precipitates 20–30  $\mu\text{m}$  in size. The content of mullite phase was about 26%. In the glassy matrix, in amount of 52–55%, several percent of dispersed crystals of mullite were either present. The quartz grains, with the diameter from a few to almost 50  $\mu\text{m}$ , occupied 8 – 9% of the surface. The majority of grains were sufficiently melted at the boundaries and adhered to the glassy matrix. The initial content of cullet was 5%. Approximately half of it fell out during preparation of the polished sections. The particles of cullet had

different size, they were about 6  $\mu\text{m}$  on average. Small pores (the most frequently below 3  $\mu\text{m}$ ) occupied 0.7% of the surface.

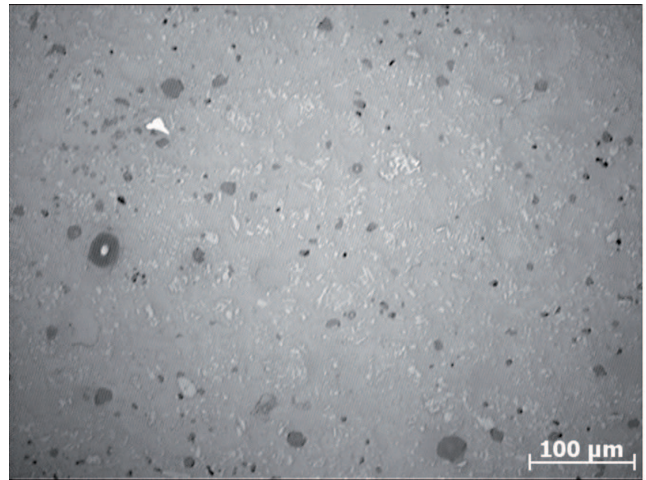


Fig. 1. Image of the structure of examined insulator material, magnification 200x. Fine, bright grains of corundum, a little greater quartz grains and white particles of cullet are visible. Darker precipitates of mullite are almost indistinguishable from glassy matrix. Dark areas of crushed out cullet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

The typical C 120 material, especially of the older line insulators, was characterized by a moderate homogeneity [4,5]. The quartz content ranged usually between 20 and 30%. It often occurred in the form of larger grains, on average about 30  $\mu\text{m}$ . 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  $\mu\text{m}$ , 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 did not exceed 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.

Tested material clearly differed from the typical C 120 type porcelain. Comparison of phase composition of the typical aluminous porcelain and tested material

is presented in Table 1. Examined porcelain contained much less quartz, 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 regarded to be stronger and more resistant to ageing processes. It was stated 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.

Differences in the structure result mainly from application of a larger share of alumina in composition of raw material C 120 modern type porcelain. It is particularly important that in addition to metallurgical alumina, there was used a dozen or so percent of ceramic alumina. Apart from the significant changes in the phase composition, it also resulted in an increase of  $Al_2O_3:SiO_2$  ratio. In a typical material kind C 120, it equals approximately 1:1 (both components about 44%). In the material that has been studied dominates  $Al_2O_3$ . One of the consequences of that is a higher content of alumina in chemical composition of the glassy matrix. This increases its mechanical and thermomechanical properties.

TABLE 1

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

#### 4. Mechanoacoustic measurements and discussion

The compressive strength of the samples, loaded until a complete destruction was equal to: 421, 443, 469, 491, 512, 557, 563, 608 MPa. 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 obtained mechanoacoustic characteristics of the particular samples showed considerable differentiation. Figure 2 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 3 presents the energy of events in the interval of one second versus stress for the strongest specimen damaged at 608 MPa.

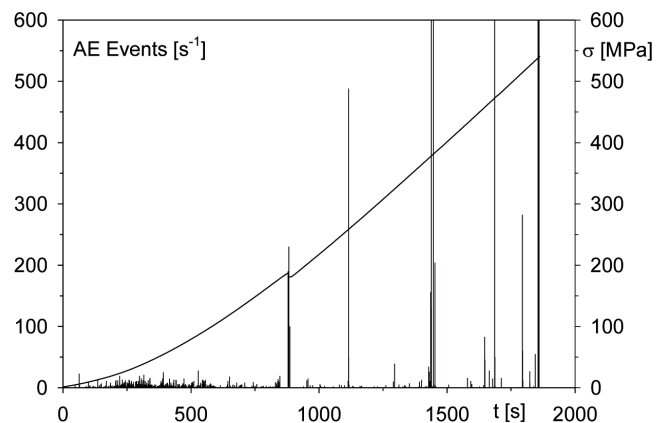


Fig. 2. Course of the rate of AE events versus increase of compressive stress for the tested sample, whose loading was stopped at 541 MPa, just before destruction. Only the preliminary and subcritical stages of degradation in stress range 0 ÷ 538 MPa are displayed. Strong signals of the last – critical stage are not included

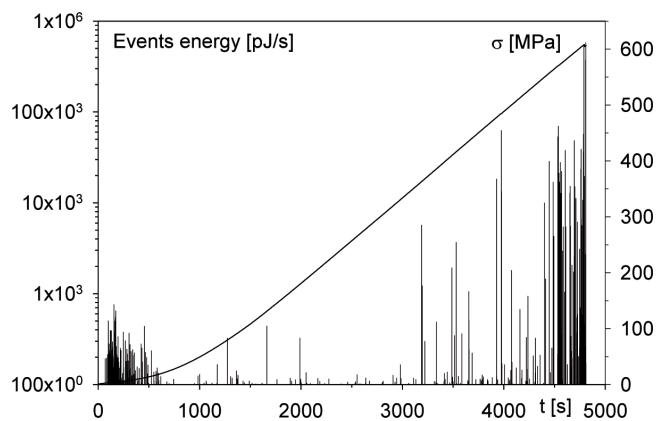


Fig. 3. Course of the energy of AE events in the interval of one second (pJ/s) versus stress for the strongest sample damaged at 608 MPa. There was applied logarithmic scale for AE descriptor



The comparative microscopic investigation of compressed samples and analysis of known mechanics of structural degradation of electrotechnical porcelain enable the interpretation of obtained mechanoacoustic patterns [4,5,8]. On the basis of these results three successive stages of degradation of the studied material could be recognized.

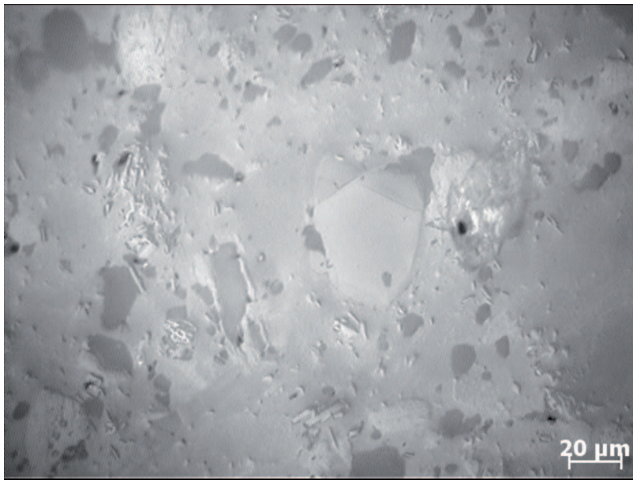


Fig. 4. Representative structure of the sample loaded up to the early subcritical stage of degradation, magnification 500x. Dark areas remaining after crushed out particles of cullet and quartz grains constitute about 4% of the surface. Fine bright grains of corundum and grey mullite precipitates are not affected by degradation

The first – preliminary stage of material degradation occurs as a result of the internal stresses, 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 low intensity of AE signals and a considerable differentiation amount individual samples. The microscopic analysis of prepared surface of specimen loaded to 100 MPa confirmed that preliminary stage of degradation corresponds to the crushing out greater part of cullet. The particles underwent fracture and separation from the porcelain matrix without any recordable acoustic activity. However, the degradation of significant part of the quartz phase was the source of AE signals. There took place initiation and development of cracks in the perimeter and to a lesser extent – inside of grains. Destruction and crushing out underwent no more than half of the quartz phase and especially small grains of size below 10 μm. The degradation did not affect corundum and mullite phases. The destroyed components of structure comprise 3–4% of the surface of compressed samples.

In Figure 4 there is presented material of the sample loaded up to 250 MPa. There are visible almost only effects of the preliminary stage.

The second – subcritical stage of structure degradation is closely connected with the homogeneity of the sample structure in 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 is considerably varied for particular samples and shows single or series of acoustic signals of low or moderate intensity of AE activity. Intervals of longer AE activity occurred rarely. The strongest signals follow the fracture and splitting off the walls and corners from the sample. During this period further damage of particles of cullet (without acoustic effects) and quartz grains (weak AE signals) takes place. There are created peripheral and internal cracks in grains. A slow degradation of mullite phase was registered too. The stresses of advanced subcritical stage, especially in the central section of the samples, caused initiation of internal cracks and sometimes crushing out parts of precipitates. They were strongly bounded with the glassy matrix and peripheral cracks occurred very rarely. In the case of the samples, which were 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 surface. This value included almost the whole cullet (below 5%), a part of quartz grains (especially of small size) and less than 1% of mullite phase. Figure 5 presents the structure of the sample containing moderate effects of subcritical degradation. In Figure 6 strongly advanced subcritical effects, in the central part of the sample compressed up to 521 MPa, are visible.

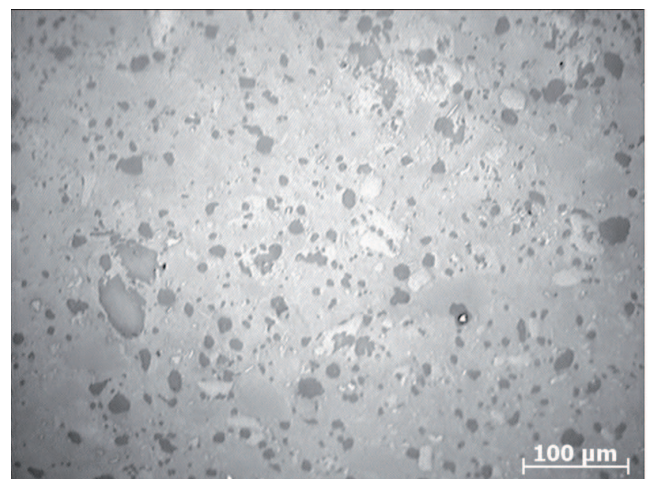


Fig. 5. Image of the structure at the boundary part of the sample stressed up to 460 MPa, magnification 200x. Dark areas remaining after crushed out particles of cullet and quartz grains of different size constitute about 7.5% of surface. Almost all bigger quartz grains contain internal cracks. Damage of mullite and corundum phases are only incidental

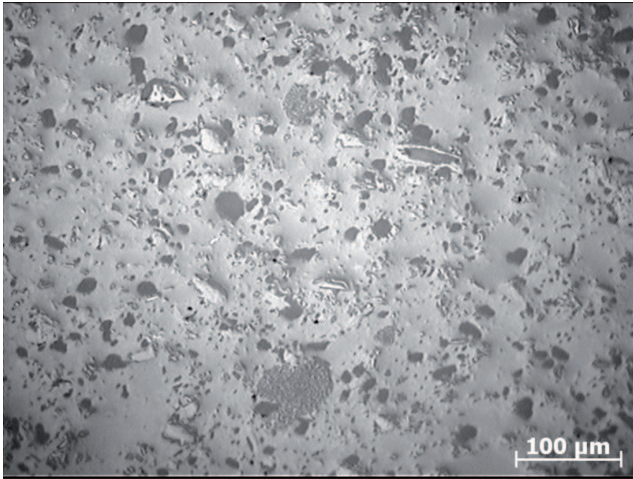


Fig. 6. Image of the structure at central part of the sample stressed up to 521 MPa, magnification 200x. Black areas remaining after crushed out particles of cullet and quartz grains constitute above 8% of surface. Strongly cracked, great and dark precipitates of mullite are visible. Quartz grain contains peripheral and internal cracks. Damages of corundum phase are rare

The last one – 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. In the case of some samples, there were generated signals following fracture and splitting off the greater pieces of specimen. This effect is visible in the stress curve as a characteristic faults (abrupt decreases). During the critical stage the remaining quartz grains undergo the peripheral and internal fracture. The degradation of the mullite precipitates is being continued as well. Grains of corundum are separated from alumina agglomerates. However, such agglomerates are observed very rarely. During the critical stage, just before the destruction, area of damaged, separated and crushed out elements of structure comprises about 13% of analyzed surface. It includes nearly whole cullet, about 3/4 of quartz, a small part of mullite as well as corundum. However, the most important and destructive effect, followed by the strong AE signals, is formation and growth of cracks in the porcelain body. The propagation of cracks is facilitated by previously destroyed elements of structure. These cracks are elongated and in general not branched – Figure 7. They grow initially amount the damaged particles of cullet, quartz grains and other crushed out components of the structure. Similarly, as in the case of C 130 type porcelain, the dispersive and fibrous reinforcement of the material structure hampers their increase. For this reason cracks are usually unbranched. In the case of typical aluminosilicate ceramic materials, including C 120 type porcelain,

single cracks join together and form even network of cracks. These effect was observed in traditional insulator porcelain [4,5]. At sufficiently high stress the rapid growth of critical cracks in the porcelain body takes place and the sample undergoes irreversible destruction.

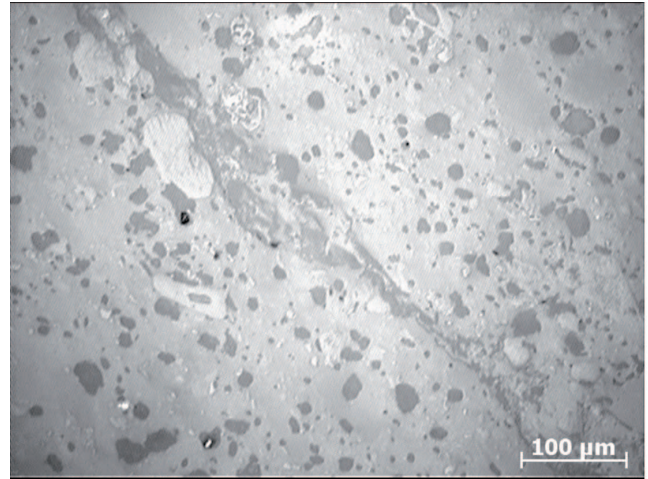


Fig. 7. Image of the structure of the piece of destroyed sample, magnification 200x. Large crack and bright fractured grains of quartz are visible. Damages of mullite phase are small and negligible in case of corundum. Dark areas remaining after crushed out elements of structure exceed 13% of the surface

## 5. Conclusions

The mechanoacoustic study showed distinct 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. There was stated a greater mechanical resistance of mullite precipitates and glassy matrix, which contained scattered grains of corundum and single mullite crystals. 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 are 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 contains dispersed reinforcement of the material structure. Scattered grains of corundum and single crystals of mullite were not as numerous as in the material C 130 type. However, they constituted the factor which effectively hinders the creation and growth of cracks. In addition, the glassy matrix of the tested material contains more alumina than the typical C 120 kind porcelain.



Consequently, the glassy phase has a higher mechanical strength. It should be noted that the porosity of the tested porcelain is relatively very low. Therefore, the mechanism of the degradation process is similar to that of C 130 type material. It concerns especially the last – critical stage of degradation. Then, at sufficiently high stress, elongated cracks, usually not branched, undergo fast growth and lead to the damage of the compressed sample.

The numerous investigations, including those performed by the authors, proved serious weakening of parameters of C 120 kind material after a long period of exploitation [1,3-6]. It concerned especially rods of line insulators, but as well post insulators, which porcelain had worse properties. The technological defects were as a role direct cause of breakdowns. The material of older insulators demonstrated high diversity of phase constitution and technical parameters. Ageing processes had further influence on properties variety magnification [3]. The different studies 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 [8]. Therefore, these tests can be used to evaluate the operational durability of insulators. On the ground of earlier researches and data from the exploitation, operational durability of porcelain C 120 type, can be assumed as limited to no more than 35 years [3-6]. 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 [6]. 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 significant inhomogeneities or technological defects in the structure of insulator.

A considerable 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. Moreover, there was per-

formed a modification of the complex multistage technological process of insulators production.

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