

INVESTIGATION OF INFLUENCE OF DEFECTIVENESS IN ALUMINOUS PORCELAIN STRUCTURE ON FRACTURE PROCESS UNDER COMPRESSIVE LOADING USING ACOUSTIC EMISSION METHOD

P. RANACHOWSKI, F. REJMUND

Institute of Fundamental Technological Research
Polish Academy of Sciences
Świętokrzyska 21, 00-049 Warszawa, Poland
e-mail: freymund@ippt.gov.pl

A. PAWEŁEK, A. PIĄTKOWSKI

Aleksander Krupkowski Institute of Metallurgy and Materials Sciences
Polish Academy of Sciences
Reymonta 25, 30-059 Kraków, Poland

(received June 15, 2006; accepted September 30, 2006)

This work presents acoustic emission (AE) and microscopic study of ceramic samples subjected to compressive stress. The object of research were specimens made of insulator aluminous porcelain 130 type. Purpose of this work was registration the stages of the process of degradation in ceramic material structure. Microscopic study allowed observing structural parameters of material before loading and on particular stages of the process of defects development. Influence of technological faults existing in material structure on mechanical-acoustic characteristics and mechanical strength of the samples was recognized.

Key words: aluminous porcelain, acoustic emission, degradation process.

1. Introduction

At present aluminous porcelain of high strength C 130 type is widely applied in the production of reliable electroinsulating elements. This material is used to produce line insulators HV and EHV, HV post insulators, medium voltage (MV) line and post insulators of increased mechanical requirements, traction insulators and hollow insulators of high parameters [1]. In the case of these products, besides high mechanical strength, a long period of exploitation without breakdown is required. For the material of 130 kind there is not enough experience obtained during a longer period of exploitation of the products. The investigation results seem to indicate a different character of the development of cracks in the material C 130 in comparison to typical aluminosilicate materials (including porcelain of 120 type). This is the result of effective reinforcement by corundum and mullite phases. The aim of the present study was to investigate the

effects of structural degradation under the influence of slowly increasing compressive stress. There were prepared specimens, which were divided into three groups: first – without any significant defects, second – with defects of smaller or medium intensity, and third – containing numerous defects in structure of samples. Specimens for ultrasonic, mechanical-acoustic and microscopic measurements were prepared according to the technology typical for production of ceramic parts of the insulators of overhead lines. The deaerating extrusion press was used for plastic formation of raw ceramic material. Firing process of specimens was carried out in a large chamber furnace. The applied composition of the mass was typical for electrotechnical aluminous materials of 130 type [1].

2. Mechanical-acoustic and microscopic investigation

The method of acoustic emission is a valuable tool when used for monitoring 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 [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. 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 to make the AE investigation independent of the influence of other factors on the degradation process of the material. This has been confirmed by the authors during the investigation of porcelain and cordierite materials [3, 4]. Mechanical-acoustic tests were carried out using specially constructed two-channel measuring system, described in details in works mentioned above. Mechanical-acoustic measurements confirmed that examined samples can be divided into three groups. The degree of defectiveness of the structure of a porcelain material has a significant influence on the acoustic characteristics of a specimen subjected to increasing mechanical load. Recognition of the stages of material structure degradation was possible on the basis of comparative investigation using microscopic and ultrasonic techniques.

Four samples, denoted as the first group, during the whole process of compression showed low acoustic activity. These specimens did not contain any technological defects of structure. For these samples the occurrence of the stage of continuous AE activity was not observed. Only single signals at different stress values unique for each sample were recorded. These signals had as a rule small amplitude. Strong AE effects appeared only in the short interval of stresses directly preceding the destruction of the specimen. This interval was called the critical one. The destructive stresses for two samples from the first group, which were loaded till complete destruction, were 1225 and 1251 MPa. Figure 1 shows the typical course of the rate of AE events as a function of compressive stress for a sample from the first group.

The samples from second group contained technological defects which resulted in decrease of mechanical strength of aluminous material. In the case of these samples a noticeable initial stage of acoustic activity was observed, which occurred within the

range from about 20 up to over 230 MPa. The continuous AE effect of the initial stage for some samples of second group disappeared already at the stress of about 100 MPa. The amplitude of AE signals for particular samples was quite differentiated. Generally, it can be stated that the amplitude of signals was on a level typical for other materials as porcelain 120 type and cordierite [3, 4]. After the preliminary stage of AE activity there were registered only single signals, mainly of low amplitude. Only at stresses exceeding 600 MPa some of the samples generated the signals of stronger amplitude. A high level of AE activity was registered at stresses preceding the failure of the sample. This relatively short – critical stage of acoustic activity was characterized by less differentiated and a considerably higher intensity in comparison to the preliminary stage. The strength of three destructed samples of the second group was 713, 881 and 918 MPa.

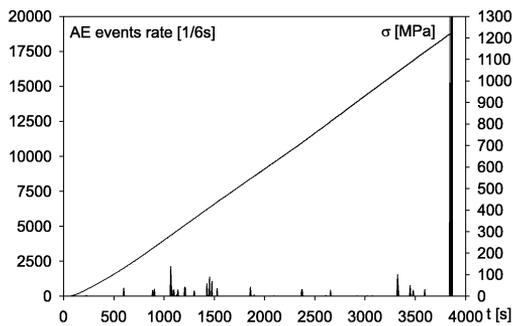


Fig. 1. Course of the rate of AE events as a function of compressive stress for a sample from the first group, which became destroyed at the stress 1225 MPa.

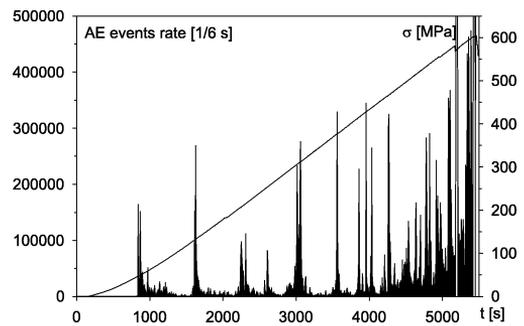


Fig. 2. Course of AE events rate for a sample from the third group, which was destroyed at the stress 604 MPa. The continuous acoustic activity and the high level of AE effect can be observed.

The last three samples, denoted as the third group, were characterized by relatively high AE activity in a wide range of stresses. In the case of these samples there were observed both the initial stage – up to about 200 MPa, the interval, defined as subcritical – differing in the range and intensity for the particular samples, and the final – critical stage, directly preceding the destruction of the specimens. The strength of the samples compressed up to failure was 604 and 647 MPa. Figure 2 shows the course of acoustic activity for a specimen which was destroyed at a lower stress. Serious structural defects were responsible for a substantial reduction of mechanical strength of the samples – about 50%. Although the defects were serious in the scale of the relatively small dimensions of the specimens, their strength was anyway high, exceeding the values obtained for samples of porcelain 120 type [3] and the cordierite material [4].

Samples from each of the three groups were selected for ultrasonic and structural investigation. Two samples from the first group, compressed up to 880 and 1144 MPa were taken for further investigation. In the case of specimens qualified to the second group – one sample was loaded up to 758 MPa – to the occurrence of a strong AE activity, which was the beginning of the critical stage. The loading of the second one

was stopped at 250 MPa – after the preliminary stage of acoustic activity. From among the samples of the third group, for further investigation there was selected a specimen loaded up to stress equal to 747 MPa. The stress was stopped already during the critical stage, close before reaching the destructive value.

Carrying out of microscopic investigation of the samples of the material required careful preparation of the areas of observation. A correct analysis of the structure required the use of a special procedure of preparing the polished sections. The specimens subjected to high compression stresses had to be especially carefully cut and polished. Thanks to use of a special saw, with finer graining of disc, the standard grinding of the examined surfaces was not necessary. Preparation of polished sections technique was described in article [3].

Phase analysis of the examined samples enabled to qualify the material as typical aluminous porcelain of high strength C 130 type [1]. The structure of the material is very similar to that observed in electrotechnical engineering products, e.g. insulators of overhead power lines. The dominating crystal phase constitutes fine-grained corundum, in amount of about 19%. It occurs in the form of elongated grains, of a few micrometers in size. Quartz grains with the diameter of $15 \pm 6 \mu\text{m}$ occupy above 8% of the surface of the polished sections. Next about 4% of surface area contained grains, which underwent crushing out during polishing process. The grey background constitutes the glassy-mullite matrix of the content more than 65%. Greater, needle-like crystals of mullite form big precipitates, usually of regular shape. Their size is of the order of $50 \div 100 \mu\text{m}$ and they represent about 25% of the surface of the polished sections. The spatial distribution of mullite precipitates is often inhomogeneous. The porosity of the samples varies in the range from 0.9 to 2.2%. The size of pores does not exceed $10 \mu\text{m}$, most often they are in the range $2 \div 5 \mu\text{m}$. They have a regular, rounded shape. In the samples of the second, and especially of the third group, the presence of large pores

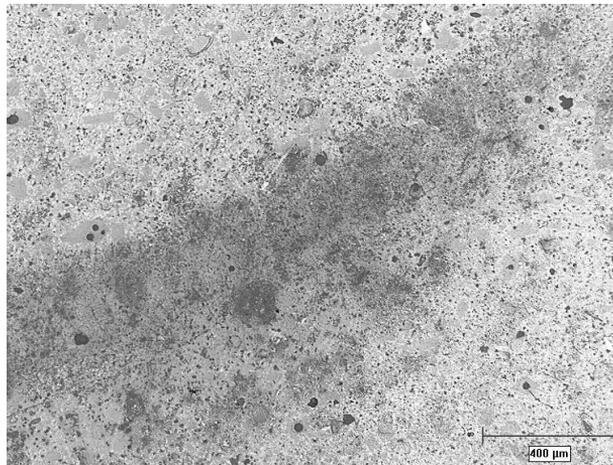


Fig. 3. Image of the structure of the material of a sample from the third group magnified $50\times$. The textural defect is clearly visible as a dark band. Large pores and cracks can be also observed.

(above 20 μm) and usually of non-uniform distribution, is observed. They occur most often inside and on the peripheries of big, macroscopic bands of characteristic dark colour – Fig. 3. The dark bands contain first of all the precipitates of mullite. The presence of the bands, with non-uniform distribution of the phases inside them, is regarded as a textural defect. In the neighbourhood of the bands there were also observed characteristic fissures. It should be noted that the revealed defects were of big dimensions, especially in comparison to small size of the samples.

Investigation of sample from the first group, loaded to the value of 1144 MPa, revealed the presence of more substantial cracks only in its central part. Length of cracks was about 250 μm and they occasionally branched out. However, they were rather few and they could not be regarded as critical defects. There was also observed the presence of peripheral cracks around quartz grains as well as inside and around of some precipitates of mullite – Fig. 4. High, exceeding 1200 MPa, strength of specimens of the first group was due to their homogeneous structure. The content of big pores was not higher than 0.3%, there were no textural defects or fissures.

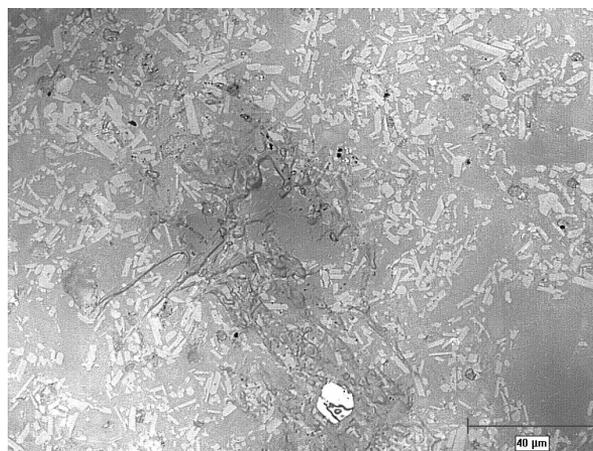


Fig. 4. Structural image of the peripheral part of a sample from the first group, loaded up to 1144 MPa, magnified 500 \times . Cracks of a big precipitate of mullite are visible.

In the material of a sample from the second group, loaded up to 758 MPa, the content of big pores was greater than 0.5% and their distribution was not uniform. The same concerned also the precipitates of mullite and quartz grains. A small number of elongated fissures were also present. These defects were the source of intensive generation and growth of microcracks during the preliminary stage of AE. Nevertheless, strong structural reinforcement hampered their further growth. At sufficiently high stresses (of about 700–900 MPa) rapid increase of the arrested cracks took place. It resulted in AE activity of the critical stage. Figure 5 shows the structure of the central part of the described sample from the second group. Big precipitates of mullite and long cracks are well visible. It should be noted that the structural defects of samples from the second group, considerably favored the formation and growth of cracks.

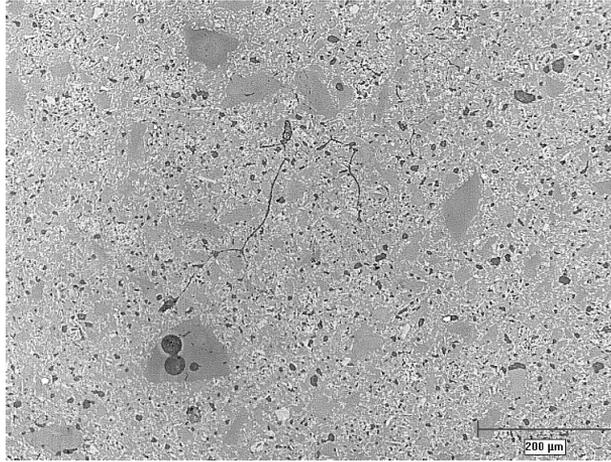


Fig. 5. Image of the structure of the central part of a sample from the second group, loaded up to the beginning of the critical stage of AE (758 MPa), magnified $100\times$. Cracks of various length are visible. Big precipitates of mullite and spherical pores inside the biggest one are observed.

Microscopic investigation carried out for many fragments of the samples from the third group revealed the presence of serious structural defects. Besides defects of textural nature (elongated dark bands), the effects of agglomeration, presence of fissures and big pores were observed. They were present most often at the boundaries of dark bands and were the source of significant internal stresses. The concentration of the defects was considerably higher than in the case of samples from the second group and their distribution was more non-homogeneous. Investigation of a sample from the third group, loaded up to 747 MPa, revealed a considerable number of cracks, especially in-

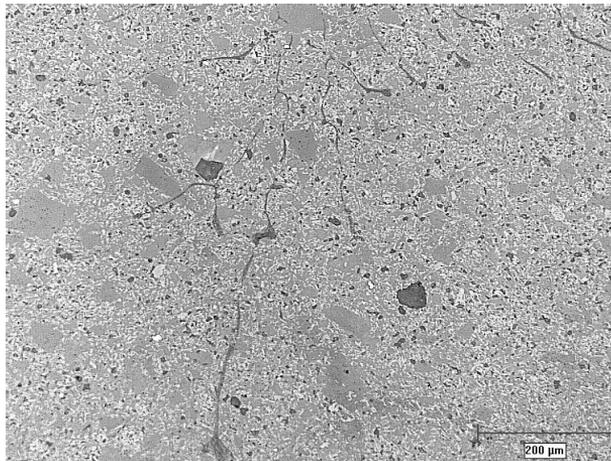


Fig. 6. Image of the material of the central part of a specimen from the third group, loaded up to 747 MPa, magnified $100\times$. Numerous large cracks, non-homogeneous distribution of darker mullite precipitates, bright quartz grains and dark areas after crushed out grains are visible.

side and at the boundary of the dark bands. The generation and the development of cracks occurred particularly in the areas of defected structure, characterized by strong internal stresses. The cracks often came out from the fissures and big pores. Numerous cracks grew up already at moderate external loads. The threshold energy of this process was low. Hence, they were the source of acoustic signals in wide range of stresses, starting from the preliminary stage of AE activity – Fig. 2. Higher external load generated series of large cracks in the central part of the sample, where the stresses became cumulated. These cracks had already a subcritical character and they propagated through all the phases of the porcelain material – Fig. 6. Considerably reduced strength – even by 50% – and high acoustic activity of samples of the third group were the result of improper homogenization of the raw ceramic body. Particularly important are properly performed firing process, a correct plasticization as well as homogenization of raw ceramic body, especially during deaeration in an extrusion press. Nevertheless all the stages of the complicated production technology have influence on operational parameters of the aluminous porcelain.

3. Final remarks

The object of complex mechanical-acoustic, microscopic and ultrasonic investigation were samples made of aluminous porcelain C 130 type. At present it is one of the most important materials used in electrical engineering. It is widely applied for the production of elements with required high durability and operational reliability – first of all power lines insulators and hollow insulators. The relatively short period of exploitation of products made of the material C 130 type has not yet allowed obtaining sufficient information about the processes of aging degradation in the porcelain of this type. The presented method and results of investigation have an innovative character. They demonstrate significant analogy between the structural effects during long period of exploitation and compressive stresses on the material in a laboratory test of relatively short duration, carried out on small specimens. It was found that the presence of areas of high internal stresses favours 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, which relatively easily undergo separation from the matrix and show internal cracking, are definitely less important. The mechanical strength of the material is determined primarily by the properties of the glassy matrix, containing a lattice of submicron, needle-shaped crystals of mullite and densely distributed fine grains of corundum.

The effectiveness of the dispersive and fibrous reinforcement of the structure of aluminous porcelain C 130 type was confirmed. Even samples containing significantly defected structure, such as macroscopic defects, demonstrated high compressive strength, exceeding 600 MPa. This means decrease of about 50% of strength in comparison to specimens without defects.

References

- [1] IEC Publication 672-1:1995 *Ceramic and glass-insulating materials, Part 1: Definitions and classification*.
- [2] RANACHOWSKI J., REJMUND F., *Acoustic emission in technical ceramics* [in Polish], [in:] *Acoustic Emission – Sources, Methods and Applications*, 55–107, MALECKI I., RANACHOWSKI J. [Eds.], Biuro PASCAL, Warszawa 1994.
- [3] RANACHOWSKI P., REJMUND F., PAWEŁEK A., PIĄTKOWSKI A., *Structural and acoustic measurements of aluminous porcelain under compression load*, 14, *Molecular and Quantum Acoustics*, **23**, 363–376, Gliwice 2002.
- [4] RANACHOWSKI P., REJMUND F., PAWEŁEK A., PIĄTKOWSKI A., *Acoustic and structural measurements of cordierite material under compression load* [in Polish], *Mat. LI Open Seminar on Acoustics OSA'2004*, pp. 195–200, Gdańsk – Sobieszewo 2004.