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ACOUSTIC-MECHANICAL AND MICROSCOPIC INVESTIGATION OF CORUNDUM MATERIAL

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This paper presents the results of acoustic emission (AE) measurements of samples under compressive stress. Additional research, concerning structure and parameters of the material, were performed using microscopic and ultrasonic methods. The object of investigation were samples of corundum material C 799 type. The content of alumina in investigated material was equal to 99.7%. The corundum materials demonstrate high mechanical, thermomechanical as well as grindability resistance and find wide application in production of modern technical devices. The aim of study was recognition of stages of degradation processes of the corundum structure. The analysis of AE results pointed out diversified strength and mechanical-acoustic characteristics of particular samples. It was stated during microscopic investigation that the main reason for this effect is due to different grains size and their spatial distribution. The authors consider the abnormal grain growth (AGG) phenomenon as a reason of inhomogeneities of the corundum structures in particular samples. This effect occurs at longer sintering term, especially for fast temperature growth. AGG phenomenon has random character and up to now is not explained in satisfying way.

Keywords: corundum material C 799, acoustic emission, abnormal grain growth.

1. Introduction

Corundum materials with high alumina (Al_2O_3) content, exceeding most often 80% – are widely applied in present-day technology. They are used first of all as elements of technical devices for which high mechanical, thermo-mechanical strength or abrasive

resistance are required. This material is resistant to oxidation, chemical corrosion and various types of irradiation. Its thermal conductivity is similar to that of stainless steel. Moreover, the corundum material has good electric parameters – high dielectric constant and low loss angle. In recent times it is also used for the production of the carrying rods of hybrid insulators [1].

In this study there are presented the mechanical-acoustic and structural investigations of corundum material of C 799 type and Al_2O_3 content equal to 99.7% [2–4]. For the fabrication of the samples the granulated product NM 9922 of Nabaltec firm was used. The size of grains collected in the aggregates was less than 0.5 μm. The crystallites were smaller by one order of magnitude. Samples for the investigations were formed using the method of single axial pressing (10 MPa) and condensed isostatically (120 MPa). After firing into biscuit at the temperature 1250°C, samples intended to be used in the investigations were cut out from a larger element taking into account grinding and shrinking of the mass. Next, the samples were fired at the maximal temperature 1700°C and stored for 1.5 hr at the sintering temperature. The samples were ground to obtain the dimensions $(5 \times 5 \times 10)$ mm^3, then their density was determined and the absorbability and absence of cracks in the alcohol solution of fuchsine were controlled. The obtained material had the density $\rho = 3890$ kg/m^3 and did not contain any detectable defects. In order to determine the size of grains some samples were polished and thermally etched at the maximum temperature 1300°C and stored for 1 hr at this temperature.

Ultrasonic control of the homogeneity of the samples has revealed a slight anisotropy as well as some differences of the acoustic parameters and the elasticity modulus between the samples. For example the velocity of the longitudinal waves c_L, measured in the direction perpendicular to the axis for various samples amounted from 10 480 to 10 600 m/s (the inaccuracy of measurement was equal to ± 20 m/s). Young's elasticity modulus E determined in the same direction was enclosed, depending on the sample, in the range from 364 to 373 GPa. The average E value was 368 GPa, at the measurement inaccuracy equal to ± 2 GPa. The mean value of Young's modulus in the direction parallel to the sample length was somewhat lower and was equal to 360 GPa. The obtained parameters considerably exceed the required by standard values of $\rho = 3700$ kg/m^3 and $E = 300$ GPa for the corundum material of C 799 type [4]. The amplitude damping coefficient for longitudinal waves of the frequency $f = 6$ MHz showed also some differences. In the direction perpendicular to the sample axis it was contained in the interval (0.4÷0.5) dB/cm whereas in the parallel direction it was (0.3÷0.4) dB/cm, at the measurement inaccuracy below 0.1 dB/cm.

2. Acoustic-mechanical and microscopic investigations

The samples were subjected to mechanical-acoustic measurements using the technique of acoustic emission (AE) on a special two-channel measuring system. Pieces of small dimensions were put to slowly increasing compressive stress ($v = 0.02$ mm/min),

with simultaneous registration of the force in one channel, and AE descriptors in the second one. The arrangement of the measurement system and the method of investigations were described in detail in the papers [5, 6]. The investigations enabled the recording and description of the correlation between the increasing external load and the processes of structure degradation, which are reflected in the acoustic activity AE. There exist serious analogies between the effects of many years long exploitation under load applied to the material and the compressive stresses in a relatively short lasting laboratory test. This observation has been proved by investigations, carried out by the authors, on ceramic aluminous materials. However, it is necessary to apply a quasi-static, very slow increase of stress and a precise registration of the AE descriptors [7, 8].

The compressive strength of the samples has shown an unexpected high dispersion. The mean strength of four destroyed specimens was 3180 MPa. The weakest of the samples underwent decohesion already at the stress equal to 2660 MPa; however, it was found impossible to measure the strength of the strongest. For technical reasons it was necessary to stop the process of loading at the stress of 3800 MPa. The mechanical-acoustic characteristics of the particular samples showed high differentiation – Figs. 1 and 2. At stresses below 2500 MPa only some samples, of more inhomogeneous structure, showed only single AE signals of various intensities. Among them was the weakest of the specimens (Fig. 1). If there appear the intervals of continuous acoustic activity – the AE signals are characterized by very low energy (Fig. 2). Above the stress 2500 MPa the loaded samples showed AE effects in the form of single signals, occasionally forming intervals of continuous acoustic activity. The interval of subcritical stresses was characterized by diversified width – depending on the sample strength. The subcritical stage preceded a short critical interval, containing a group of signals of high energy. The critical interval occurs in the range of more than a dozen – some tens of megapascals and it directly precedes the decohesion of the sample. Thus, the stress at which the critical stage occurs is closely connected with the sample strength.

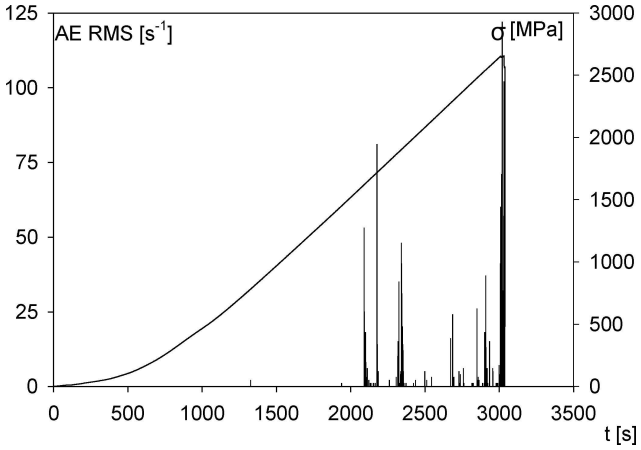

Fig. 1. The course of RMS AE rate versus the increase of compression stress for the weakest sample, denoted 5, of the strength 2660 MPa.

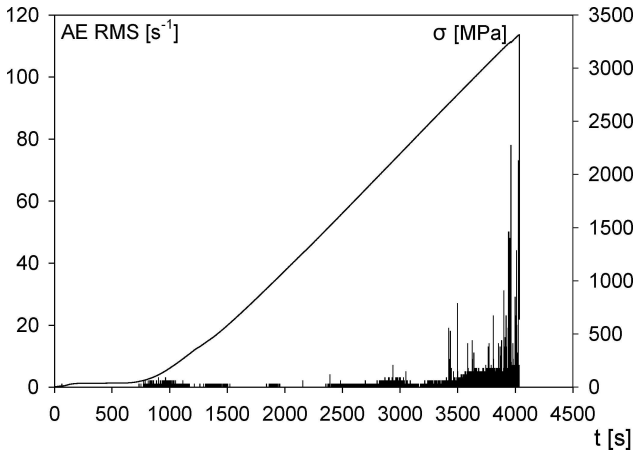

Fig. 2. The course of RMS AE rate versus the increase of compression stress for the sample, denoted 6, of the strength 3320 MPa.

Considering the great differentiation in the values of the destructive stresses for the particular samples and their various mechanical-acoustic characteristics, it was necessary to carry out accurate microscopic investigations of the corundum material structure. The investigations concerned the size and the uniformity of distribution of the grains and the effects of the compressive stress on various parts of the samples.

Five samples of various mechanical-acoustic characteristics were selected for microscopic investigations. Their loading was stopped shortly before the critical stage of structure destruction. In all the samples there were observed the effects of structure loosening. This concerned especially the central part of the samples where the stresses undergo cumulation. Propagation of microcracks occurs only along the grain boundaries. Nevertheless, there have been observed places of crushed out grains or parts of grains, comprising 0.2÷0.3% at the boundaries and up 1.0% of the surface of a polished section in the middle of the samples. Porosity of the material in any of the samples did not exceed 0.11%. Figure 3 shows the central area of a sample compressed up to 3200 MPa, on which the crushed out fragments and even the whole grains are visible. Their size is below twenty micrometers. The structure underwent evident loosening. The length of cracks between the grains is found in the interval (15÷50) μm, and many of them have a closed character. There occur also wider cracks of the character of fissures. In the central part of a sample stressed up to 3180 MPa there were observed even long cracks of the length of some hundreds of micrometers – Fig. 4. The strongest of the investigated samples (compressed up to 3800 MPa), showed a relatively moderate degree of structure degradation – Fig. 5. Even in its central part, the surface area of the crushed out grains did not exceed 0.3%. The mean length of cracks was about 20 μm and they were not longer than 50 μm. The strength of the sample was the result of its homogeneous structure in the micro- and semi-macro scale. The grains showed single-lobe size distribution. Their diameters were contained in a nar-

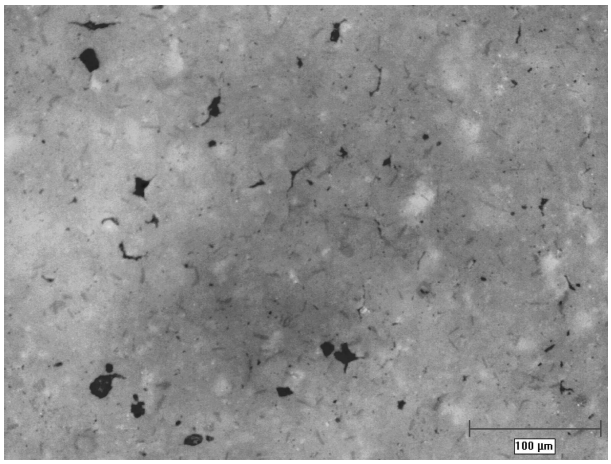

Fig. 3. Image of the middle part of the sample, denoted 2, loaded up to 3200 MPa.

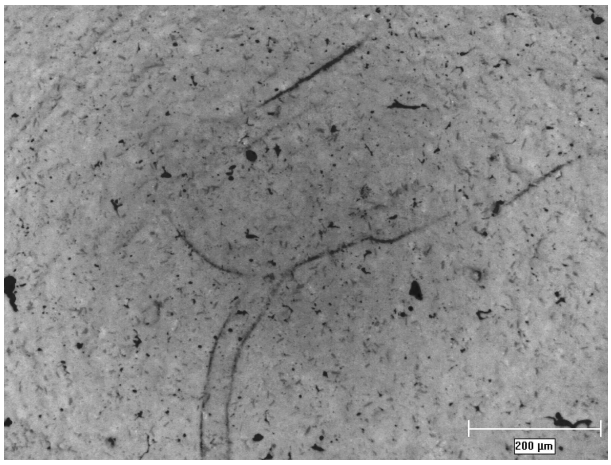

Fig. 4. Cracks in the central area of the sample, denoted 3, loaded up to 3180 MPa.

row range from above 1 to 14 µm, with the mean value equal to 7 µm. In the case of the other samples a two-lobe size distribution of the grains was as a rule registered. The sizes of bigger grains were most often in the range from less than 10 to over 30 µm, whereas the diameter of fine grains was from a fraction to a few micrometers. The majority of samples showed distinct inhomogeneities of the structure in the semi-macro scale as well as insufficient homogeneity in the micro scale. The bigger grains often formed centres of the size of the order of 100 µm, surrounded by smaller areas containing finer grains. Another discovered inhomogeneity was arrangement of the structure in form of bands. The greater and the smaller grains become arranged as separate bands having width of the order of some tens of micrometers. A com-

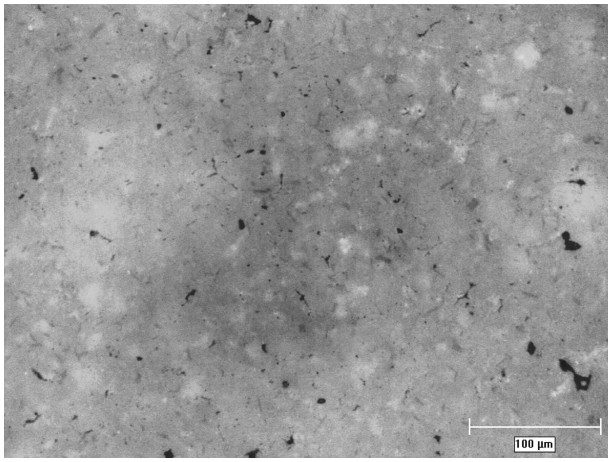

Fig. 5. Structure of the material in the boundary part of the strongest sample, denoted 4, loaded up to 3800 MPa.

pletely regular structure was observed only in the mentioned sample of the highest strength.

3. Conclusions

Microscopic and ultrasonic investigations have revealed that the great differences registered for the strength and the mechanical-acoustic characteristics of the samples are due to the inhomogeneity of the material in the semi-macro as well as the micro scale. The distribution of the grains sizes demonstrates most often a two-lobe form.

They are not uniformly distributed in the space, grouping into centres or bands. The occurrence of groupings of grains in the structure of the corundum material represents most probably the intermediate state, leading to the known effect of the abnormal grain growth (AGG) [9]. This phenomenon occurs in oxide materials, it has a probabilistic character and its origin in spite of many years of investigations, has not been sufficiently explained [10, 11]. AGG effect occurs after a longer time of firing than in the case of the applied technology of the production of the samples. The quick increase of temperature in the course of thermal treatment favours its occurrence and such temperature raise – of the order of 200°C within one hour – was realized. In the case of obtaining greater and longer sintered elements, the AGG effect would cause considerably greater differences in the size of the grains. The bigger grains grouped in centres or bands would join, attaining the size exceeding 100 µm.

Acknowledgment

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