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THE ANALYSIS OF THE INFLUENCE OF THE THERMAL SHOCKS ON ACOUSTIC EMISSION SIGNAL GENERATED IN CORDIERITE CERAMICS

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The paper describes a raw materials composition and manufacturing technology of cordierite material, applied to manufacturing of welding backing strips. The resistance of the cordierite specimens to thermal shock within the range of $150-320°C$ was analysed. Shocked samples, submitted to three — point mechanical stress, have been investigated by acoustic emission method. The results let the authors to conclude that there is a correlation between the Acoustic Emission (AE) signals parameter, describing the AE activity during the loading and the stage of material degradation caused by the applied thermal shock.

1. Introduction

The thermomechanical durability of ceramics is highly influenced by the critical microcraks growth due to thermal shock. One of the efficient methods to control that process is the Acoustic Emission measurement [1]. The latter method let us to determine the stress level corresponding with the initiation of the critical destruction processes in the investigated composition. The aim of this paper is to combine the initial stages of crack formation processes and their growth with the descriptors of the AE signal due to the increasing temperature difference of the applied thermal shock. The similar investigations were done on the alumina and the magnezite–zirconic ceramics [2]. The authors of this paper investigated the resistance to thermal shock of the cordierite ceramics and measured the AE activity of this material during three-point bending test after shocking.

Cordierite ceramics was chosen for the investigation due to its wide application in manufacturing of the elements with high thermal shock durability — for example the welding backing stripes.

The welding backing stripes are wide applied in one — operator welding, especially in shipyard and pressurized vessel construction. The stripes should meet several requirements: its shape and dimensions should stimulate the right formation of the welding path, they should not interact with the welding substrates by gas production or intermediate layer formation. Their expansion coefficient should be less than $4\times10^{-6}K^{-1}$, they should also withstand the thermal shock at the temperatures higher than $250°C$ and its bending strength should be higher than 100 MPa. All these requirements are optimally met by the cordierite ceramics.

For the perfect stechiometric composition the melting point is situated relatively high at 1545°C. The real compositions with additions of glass phase or other cristallites melt at lower temperature. The higher contents of the stechiometric cordierite represent the better thermal shock durability but at the real conditions there is mullite as a major addition. The stechiometric contents of the cordierite is following: 13.7% of MgO, 34.9% of Al_2O_3 and 51.4% SiO_2. The latter list indicates the low contents of SiO_2 when comparing to other ceramic compositions. That implies the low liquidity of the cordierite in the sintering process what increases the sensivity of the material on the slight inhomogeneities of the oven temperature distribution.

2. The structure and the thermomechanical durability of cordierite

The contents of the cordierite composition was prepared in accordance to the remarks stated in the previous paragraph. The composition used in following research fulfils the requirements of the Polish Standard No 86/E-06301 for the materials belonging to Group No 410. At the other hand, the mechanical parameters of prepared material are also sufficient for the demands of welding technology. The detailed contents of the composition is as follows: plastic fireproof clay 42%, rough Chinese talc 23%, ceramic alumina 20%, quartz sand 10%, kalium feldspar 5%.

The material contents stated above was designed in the way to obtain reduction of expansion coefficient and immunity for the sintering temperature variations. This was realized by the additional stabilization of the cordierite phase by mullite ($3Al_2O_3{\cdot}2SiO_2$) what caused additional mechanical durability increase. Dimensions of the prepared welding strip bases had the dimensions of $105 \times 25 \times 8$ mm. Their plain shape and relatively small size enabled for using a press in shaping of the details. Two stage grinding process was applied for the substrates — preliminary grinding of hard cristallites and — finally — the whole mass grinding in the presence of plasticizers. The products were sintered in tunnel oven at temperature of 1280 ± 10.

The porosity of the backing strips was in the range of 9.5 — 10%, the average pore size was 5.5 μm and the mulite grain diameter was in order of 4 μm. At the microscopic image of the structure there are oval pores, numerous long mulite crystals placed in glassy cordierite matrix with slight contents of quartz relicts.

To discuss the resistance of the prepared material to thermal shocks according to the Polish Standard, mentioned above it has to be observed that the sample shocked with the temperature 250°C should perform not less than 75% of its normal mechanical strength. The compositions used at high temperature ranges usually can be applied up to threshold shock temperature difference, $\Delta T_{\max}$ and beyond this value the major structural damages arise in the material volume. $\Delta T_{\max}$ depends on mechanical parameters of the compositions according to the following formula:

$$\Delta T_{\max} = \frac{\lambda \cdot R_r}{\alpha \cdot E}(1 - \mu), \tag{1}$$

where: λ — heat conductivity, $-R_r$ tensile strength, α — linear expansion coefficient, E — Young's modulus, μ — Poisson's ratio.

The basic mechanical parameters of the investigated materials were following: specific density — 2540 kg/m^3, Young's modulus, measured with ultrasound method — 91 GPa, critical stress intensity ratio — 2.9 MPa·m$^{1/2}$, average bending strength — 132 MPa, Weibull's coefficient of the bending strength distribution — 21, linear expansion coefficient at 200 – 700°C — 2.9 ÷ 3.9×10^{-6} K^{-1}. Calculating the formula (1), using the typical values for the cordierite — $R_r = 1000$ GPa, $\lambda = 10^{-4}$ J/m s°C, $\mu = 0.1$ one can obtain the result that the ideal shaped cordierite sample should withstand the shock $\Delta T_{\max} \sim 1000$°C. In real conditions, the measured value of $\Delta T_{\max}$ reaches 25% of its theoretical value. The thermal shocks were applying to the samples, preheated to the temperature T_1 by placing them in water bath in temperature T_0. The determination of $\Delta T_{\max}$ was undertaken measuring the three-point bending strength R_b of the samples as a function of shocking temperature difference. The measurements of R_b were made on the loading machine type Zwick 1446. The traverse velocity during the measurements was 1 mm/min and the sample supporting prisms were placed at the distance of 60 mm from each other. The results of R_b measurements are shown in Table 1 and in Fig. 1. The accuracy of the R_b measurements was ca. 2%.

Table 1. Results of the measurements of three-point bending strength R_b for the cordierite samples, as a function of shocking temperature difference ΔT.

ΔT [°C]	0	100	150	200	220	250	280	300	320	400
R_b [MPa]	102.9	102.3	103.2	107.5	105.9	33.5	29.6	27.7	35.4	24.5

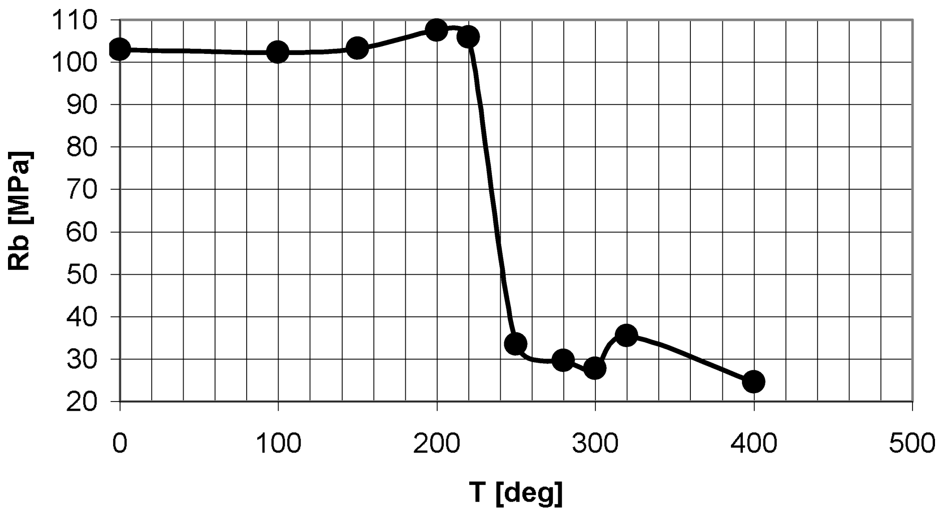

Fig. 1. Results of the measurements of three-point bending strength R_b for the cordierite samples, as a function of shocking temperature difference ΔT, visible value of threshold shock temperature difference $\Delta T_{\max}$.

3. Acoustic Emission measurements

In the course of bending test a wideband AE sensor (Physical Acoustic Corp., WD Type) was attached to the loaded cordierite samples. Acoustic Emission signal processor consisted of the band-pass amplifier, working in the frequency band 500 – 2000 kHz and AE counts processor, connected with personal computer via fast parallel interface. Because of rapid character of microcrack propagation in cordierite the EA counts registration was made at a rate of 100 measurements per second. For the purpose of further analysis of AE activity in different regions of bending stress, current level of loading force was also registered in the computer. Eighteen samples were tested for each shocking temperature difference ΔT indicated in Table 1. For all ΔT levels the large variations in measured counts sum per entire loading process were observed. Therefore no correlation between AE counts sum and shocking temperature difference ΔT could be found. The most probable reason of this effect could be the fact that the sensivity of EA sensor remarkably depended on the direction of crack propagation and the influence of termal shock structure degradation was weaker than the factor mentioned above. However AE activity was indicated both at the low levels of bending stress and at the critical levels of that stress (comparable to rupture level). Figure 2 illustrates typical time dependence of AE counts rate at three-point bending test. Current AE count recordings are marked as triangles, the black line indicates the stress applied to the sample.

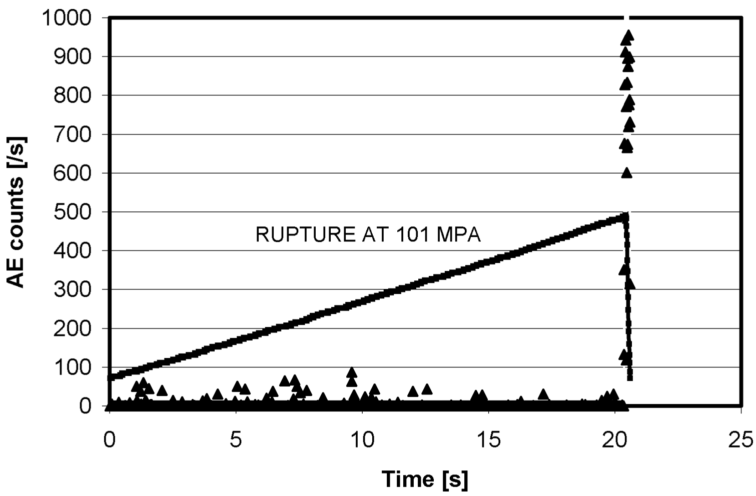

Fig. 2. Typical time dependence of AE counts (triangles) and applied bending stress (solid line) registered in investigated cordierite sample.

The authors of this paper processed the AE data putting into account the feature shown in Fig. 2 — recordable AE activity at the entire process of mechanical loading of the samples. For each sample AE counts were totalized in two separate regions. The first region, denoted as N_I included AE counts totalized from the load start up to 95% of the bending stress. The second region, denoted as N_R included AE counts from 95% of the bending stress to the rupture. The calculations indicated that N_I and N_R are proportional to each other within the group of the samples treated with the same thermal shock temperature difference. In each group only 25% of the samples failed to present the trend described above (probably they were defected at the manufacturing process). Therefore the ratio N_I/N_R could be treated as acoustic measure of material degradation due to thermal shock. The average N_I/N_R ratios for each sample group (at least 18 members) together with averaged bending strength and shocking temperature difference ΔT are shown in Table 2. The dependence N_I/N_R ratio on shocking temperature difference ΔT are shown in Table 2.

Table 2. The average N_I/N_R ratios for each sample group shown together with averaged bending strength and shocking temperature difference ΔT measured for the group.

ΔT [°C]	0	150	200	220	250	280	320
R_b [MPa] (averaged)	106.2	102.4	102.7	104.3	34.2	35.1	30.7
$N_I/N_R \times$ 100%	40	36	83	66	5	0.6	0.4

The dependence N_I/N_R ratio on shocking temperature difference ΔT is also presented in Fig. 3.

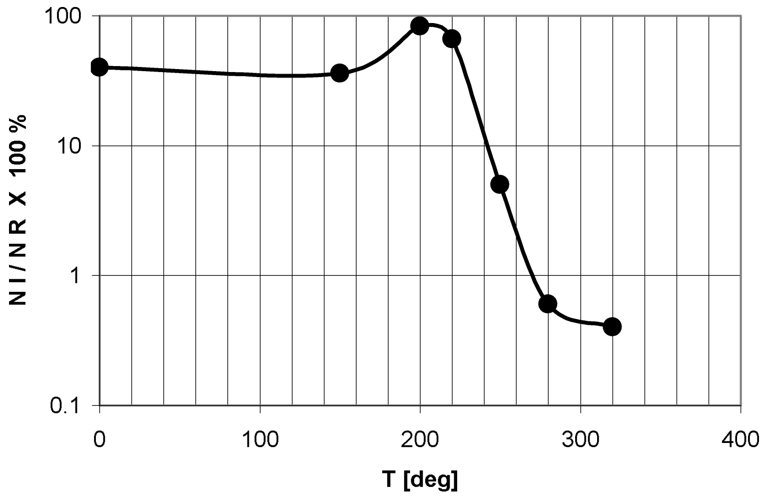

Fig. 3. The dependence N_I/N_R ratio on shocking temperature difference ΔT for the investigated cordierite samples.

There are remarkable similarities in the shape of the curve shown in Fig. 1 and in the shape of the curve shown in Fig. 3. The first curve presents the results of the mechanical strength test. The latter presents the data derived from the acoustical measurements. To fulfil the requirements of the Polish Standard No 86/E-06301, mentioned in second paragraph — according to the results presented in Fig. 1 — the shocking temperature difference should not exceed 230 degrees. The same result with accuracy of ±5% is possible to obtain using the data submitted in Fig. 3 if we modify the definition of significant structure degradation what was assumpted in the Polish Standard described above. For the results of AE measurements it should be specified that the significant structure degradation appears if the N_I/N_R ratio measured in logarithmic scale as a function of shocking temperature difference ΔT falls down to 75% of its initial value.

The investigation of material resistance to thermal shock with application of mechanical loading is expensive kind of testing. The additional problems with application of the described method are caused by the dispersion of the mechanical strength of the investigated specimens. Therefore, application of acoustic method of shocking stress monitoring during mechanical test might provide the additional verification of the obtained experimental results.

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