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# Fracture Toughness of Ceramics Fired at Different Temperatures

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The fracture toughness test was performed at room temperature on sets of 5 ceramic samples made from material for high voltage insulators (kaolin 36 wt. %, $Al_2O_3$ 30 wt. %, clay 12 wt. % and feldspar 22 wt. %) fired at temperatures 400, 500, 600, 700, 800, 900, 1000, 1100, 1200, 1250, 1300, 1400, 1500 °C at heating and cooling rate of 5 °C/min. The precrack was made to each sample by indentation under the loads 10 N – 200 N, the dwell time was 45 s and the loading rate was 10 N/s. Results of the fracture toughness tests were in accordance with changes of structure of the samples after the partial firings. Fracture toughness from 20 °C to 500 °C is almost constant and it varies between 0.1 MPa·m$^{0.5}$and 0.2 MPa·m$^{0.5}$. Dehydroxylation (420 °C – 600 °C) does not influence the value of fracture toughness. At temperature interval where we assume sintering (700 °C – 1250 °C) we observe exponential dependence of fracture toughness up to 1.5 MPa·m$^{0.5}$. From comparison of the fracture toughness, Young's modulus and flexural strength follows a correlation and proporcionality of these mechanical properties.

*Keywords*: ceramics, fracture toughness, mechanical strength, solid-state sintering, liquid-state sintering, dehydroxylation.

## INTRODUCTION

Mechanical parameters are important characteristics of ceramic materials. Each ceramic product is mechanically stressed in technological processes during drying and firing as well as in actual service. For example it is known that increasing of the temperature during firing is possible only until limit, when thermomechanical stress is lower than mechanical strength [1, 2].

The dependence of the mechanical strength during firing was investigated in [3, 4]. Classical ceramics based on kaolinite and illite is presented in many fields of industry. In the last decades, a significant progress in increasing of the mechanical strength was made for alumina porcelain.

Mechanical strength after firing at different temperatures was measured in [5]. However, in literature we did not find results for the fracture toughness ($K_{Ic}$) for porcelain, apparently because this value is low, so no attention was given to this problem.

Ceramic materials are brittle. A lot of research is conducted to decrease the brittleness of advanced ceramics [6, 7]. This brittleness may be judged indirectly by $K_{Ic}$ which denotes material's resistance to brittle fracture when a crack of critical size is present. The subscript *Ic* denotes the mode *I* crack opening under the normal tensile stress perpendicular to the crack path. Brittle fracture is very characteristic for materials with low fracture toughness [8]. A related concept is the work of fracture ($\gamma_{wof}$), which is directly proportional to $K_{Ic}^2/E$, where $E$ is the Young's modulus of material [9].

The aim of this article is to find out how $K_{Ic}$ of the ceramic material depends on the firing temperature.

## THEORETICAL PART

According to standard ASTM C1421-10 [10], the dimensions of the sample subjected to fracture toughness test as well as the direction and position of force application and precrack orientation, indentation location and orientation, and indentation and (Palmqist) crack dimensions are depicted at Fig. 1 where d*W* and d*B* means the maximum deviation of sample dimensions acording to standard [10].

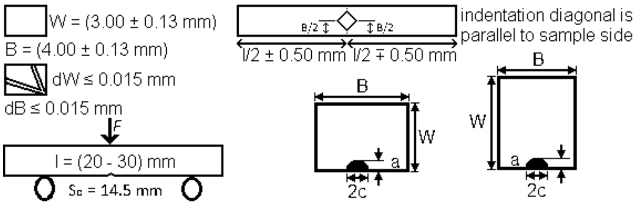

**Fig. 1.** Dimension of the sample, indentation and location orientation and determinimg the crack dimensions

For the sc (surface crack) method and the three-point-bending in flexure we calculate the fracture toughness $K_{Isc}$ from the following equation:

$$K_{Isc} = Y\left(\frac{3P_{max}S_0}{2BW^2}\right)\sqrt{a}\,10^{-6}, \tag{1}$$

where $K_{Isc}$ is the fracture toughness [MPa·m$^{0.5}$], $Y$ is the stress intensity factor (Eq. 2 or Eq. 6, whichever is greater), $P_{max}$ is the maximum force (the breaking force) [N], $S_o$ is the outer span (0.0145 m in this case), $B$ is the side to side dimension of the test specimen perpendicular to the crack length (depth) [m], $W$ is the top to bottom dimension of the tested specimen parallel to crack length (depth) [m], $a$ is the crack depth [m], $c$ is the crack half width [m].

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We calculate the stress intensity shape factor coefficients for both the deepest point of precrack periphery $Y_d$ and for the point at the surface $Y_s$, which give a maximum error of 3 % for an ideal precrack and an estimated maximum error of 5 % for a realistic precrack.

For the deepest point of precrack the stress intensity factor is

$$Y_d = \frac{\sqrt{\pi} M H_2}{\sqrt{Q}}, \tag{2}$$

where:

$$Q = Q(a/c) = 1 + 1.464(a/c)^{1.65}, \tag{3}$$

$$M = M(a/c, a/W) = (1.13 - 0.09(a/c)) + \\ + \left(-0.54 + \frac{0.89}{0.2 + (a/c)}\right)(a/W)^2 + \\ + \left(0.5 - \frac{1}{0.65 + (a/c)} + 14(1 - a/c)^{24}\right)(a/W)^4, \tag{4}$$

$$H_2 = H_2(a/c, a/W) = 1 - (1.22 + 0.12a/c)(a/W) + \\ + (0.55 - 1.05(a/c)^{0.75} + 0.47(a/c)^{1.5})(a/W)^2, \tag{5}$$

For the point at the surface the stress intensity factor is:

$$Y_s = \frac{\sqrt{\pi} M H_1 S}{\sqrt{Q}}, \tag{6}$$

where:

$$H_1 = H_1(a/c, a/W) = 1 - (0.34 + 0.11a/c)(a/W), \tag{7}$$

$$S = S(a/c, a/W) = (1.1 + 0.35(a/W)^2)\sqrt{a/c}. \tag{8}$$

Note 1: The stress intensity factor coefficients are valid only for $a/c \leq 1$. They can be used for $a/c$ ratios slightly greater than 1 with slight loss of accuracy.

Note 2: The term in the brackets of Eq. (1) is the flexural strength [MPa] of the beam with the surface crack. It is often useful to compare this value with a range of values of the flexural strength of test specimens without a precrack, in which fracture occurs from the natural fracture sources in the material.

Note 3: Precrack is Vickers indentation at the load (10 – 200) N, dwell time 45 s.

## MATERIALS AND EXPERIMENTAL PART

Ceramic samples were made from the wet ceramic plastic mass for manufacturing the high voltage insulators from alumina porcelain. Mineralogical composition of the mass was 36 wt. % of kaolin, 30 wt. % of $Al_2O_3$, 12 wt. % of clay and 22 wt. % of feldspar.

Green blank with diameter of 300 mm was prepared from the wet mass, described above, by the vacuum extruder in ceramic plant PPC Čab, Slovakia. Prismatic samples were cut from the wet blank then dried in the open air and then grinded and polished. The final dimensions of samples were 5 mm × 5 mm × 20 mm. The sets of 5 samples were fired at different temperatures 400, 500, 600, 700, 800, 900, 1000, 1100, 1200, 1250, 1300, 1400, 1500 °C. The heating and cooling rate was 5 °C/min without soaking at the highest temperature.

After firing, the four flat sides of samples were polished with sandpaper rotating machine with an angular frequency 600 rpm for 5 minutes. The sandpaper number was 800. The final dimensions of the samples were close to 3 mm × 4 mm × 20 mm. The structure of material is highly porous with a net of open pores. The porosity varies from 32 % for green ceramics to 37 % at 500 °C and decreases to 5 % within interval 1000 °C – 1200 °C [11].

To make a precrack, an indentation was performed by a hardness tester Zwick-Indtentec 5030 SKV [12] in the middle of the sample side under the load 10 N – 200 N with the dwell time of 45 s, see Fig. 2. The diagonals of indentation were parallel with sample sides. Indentations of the samples were then colored with red dye penetrant ARDROX F6R for 10 – 60 minutes and then cleaned with towel to remove excessive penetrant.

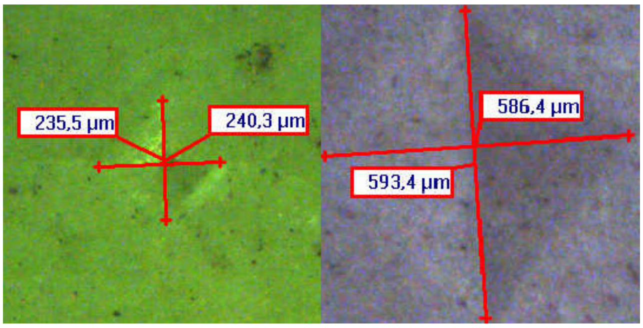

**Fig. 2.** Picture of the indent at 1250 °C (left picture) and at 400 °C (right picture)

A fracture toughness test was performed on the Instron dynamic testing system 8516 [12] under the loading rate of 10 N/s according to standard [10].

## RESULTS AND DISCUSSION

The dependence of the fracture toughness on firing temperature is shown in Fig. 3. The standard deviation of $K_{Ic}$ varies from 2 % to 18 %.

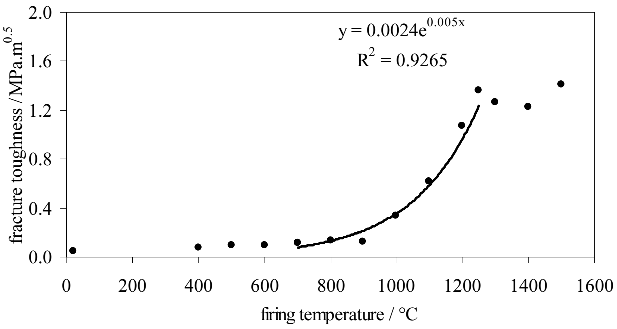

**Fig. 3.** The dependence of fracture toughness on firing temperature

Similar graphs, as pictured in Fig. 3, were also obtained for flexural strength and Young's modulus on samples made of quartz porcelain mixture (50 wt. % of kaolin, 25 wt. % of feldspar, 25 wt. % of quartz) measured at the same conditions [3, 13], see Fig. 4. Although the material composition of quartz samples is slightly different, the processes and their consequences during heating are the same in the alumina and quartz samples [1, 2]. Therefore, we can use the results presented in Fig. 4 for qualitative explanation of the fracture toughness results pictured in Fig. 3. The fracture toughness, Young's modulus and flexural strength have similar temperature dependences, as follows from Fig. 3 and Fig. 4, so they correlate together.

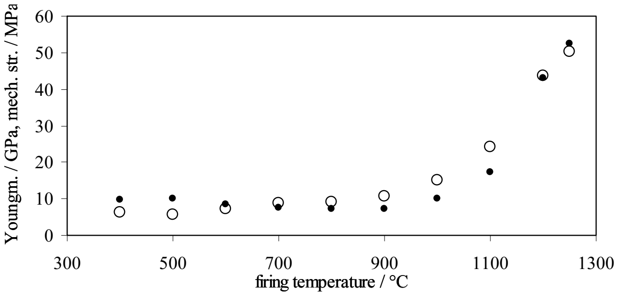

**Fig. 4.** The dependence of flexural strength (o) and Young's modulus (●) on firing temperature

For the samples fired at 20 °C – 500 °C no changes in structure occurred at these low temperatures [1, 2], therefore the mechanical strength and Young's modulus, Fig. 4, are nearly constant in this range. The same is valid for fracture toughness, as can be seen in Fig. 3.

For the samples fired at 500 °C – 700 °C, dehydroxylation, which weakens the kaolinite crystals, takes place. Metakaolinite created during dehydroxylation is very porous material with internal vacancies [14] and lowers the mechanical properties of the samples. In spite of that, we do not register a significant decrease of the mechanical properties within the temperature region of 500 °C – 700 °C, see Fig. 3, Fig. 4. We can assume that the solid state sintering prevails over the weakness of the metakaolinite crystals, so the crystal interfaces remain stronger after cooling up to room temperature.

For the samples fired at 700 °C – 1250 °C, there is exponential increase in the fracture toughness and also the mechanical strength and Young's modulus increases dramatically. The solid-state sintering continues up to 1150 °C where feldspar begins to melt and a liquid-phase sintering starts. The samples contain glassy phase, their density is higher, and this corresponds to higher values of the flexural strength measured at room temperature.

In the samples fired above 1300 °C closed pores are present [1, 2] which lower the mechanical strength and fracture toughness.

## CONCLUSIONS

1. Fracture toughness from 20 °C to 500 °C is almost constant and it varies between 0.1 and 0.2 MPa·m$^{0.5}$.
2. Dehydroxylation (420 °C – 600 °C) does not influence the value of fracture toughness.
3. At temperature interval where we assume sintering (700 °C – 1250 °C), we observe exponential dependence of the fracture toughness.
4. From comparison of the fracture toughness, Young's modulus and flexural strength follows a correlation and proporcionality of mechanical properties which agrees with theoretical assumptions.

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