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FRACTURE TOUGHNESS OF KAOLIN FIRED AT DIFFERENT TEMPERATURES

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Abstract: *The fracture toughness (FT) test was performed at room temperature on ceramic samples made from kaolin at temperatures of 400–1250 °C at a heating and cooling rate of 5 °C/min. The precrack was made by indentation under the loads 10–200 N, the dwell time was 45 s and the loading rate was 10 N/s. Results of the FT tests were in accordance with the changes in structure of the samples after the partial firings. The FT 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 (420–600 °C) and solid state sintering (700–950 °C) does not influence the value of FT. At temperature interval where we assume liquid state sintering (1000–1250 °C) we observe an exponential increase of FT up to 0.6 MPa·m$^{0.5}$. From comparison of FT, Young modulus and flexural strength, a correlation and proportionality of these mechanical properties follows.*

Keywords: Fracture toughness, Mechanical strength, Sintering, Dehydroxylation.

1. Introduction

Mechanical parameters are important characteristics of ceramic materials. Each ceramic product is mechanically stressed during technological processes as drying and firing as well as in actual service. For example it is known that increasing the temperature during a firing is only possible until the limit, when the thermomechanical stress is lower than the mechanical strength (Norton, 1970; Hanykýř, 2000).

Mechanical strength during firing was investigated in (Štubňa, 1992; Štubňa, 2011). Classical ceramics based on kaolinite and illite are presented in many fields of industry. Significant progress was made during the last few decades in increasing the mechanical strength of ceramic material.

Mechanical strength after firing at different temperatures was measured in (Štubňa, 2011). However, we did not find the results for fracture toughness (K_{Ic}) for kaolin in the literature in spite of the fact that kaolin plays a crucial role in such ceramics, e.g. in porcelain.

Ceramic materials are brittle. A lot of research is conducted to decrease the brittleness of advanced ceramics (Guo, 1996; Okabe, 2001). This brittleness may be judged indirectly by K_{Ic} which denotes material resistance to brittle fracture when a crack of critical size is present. The subscript *Ic* denotes the mode *I* crack opening under normal tensile stress perpendicular to the crack path. Brittle fracture is very characteristic for materials with low fracture toughness (Hertzberg, 1995). A related concept is the work of fracture (γ_{wof}), which is directly proportional to K_{Ic}^2 / E, where E is the Young's modulus of material (Dos Santos, 2003).

The aim of this contribution is to find out how K_{Ic} of the fired kaolin depends on the firing temperature.

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2. Theoretical Backround

For the surface crack method and the three-point-bending, 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} = fracture toughness [MPa·m$^{0.5}$], Y = stress intensity factor coefficient, P_{max} = breaking force [N], S_o = outer span [m], B = side-to-side dimension of the test specimen perpendicular to the crack length (depth) [m], W = top to bottom dimension of the tested specimen parallel to crack length (depth) [m], a = crack depth [m], c = crack half width [m].

A precrack is a Vickers indentation at the load 10–200 N and dwell time 45 s. Therefore, the stress intensity factor coefficient is

$$Y_s = \frac{\sqrt{\pi}MH_1S}{\sqrt{Q}} \tag{2}$$

where $H_1 = 1-(0.34+0.11a/c)(a/W)$ and $S = (1.1+0.35(a/W)^2)\sqrt{a/c}$, [ASTM C1421-10].

3. Experimental

Samples were made from kaolin, its chemical composition is in Tab. 1.

Tab. 1: Composition of Sedlec kaolin.

Component name	***LOI***	***SiO_2***	***Al_2O_3***	***Fe_2O_3***	***TiO_2***	***CaO***	***MgO***	***K_2O***	***Na_2O***
Weight %	***12.95***	***45.80***	***37.31***	***0.98***	***0.17***	***0.58***	***0.46***	***1.17***	***0.58***

Kaolin was crushed and sieved and a powder was mixed with water to obtain a plastic mass with 20 wt.% of water. Prismatic samples of the dimensions 5×5×20 mm were cut from the wet mass, dried in the open air for 5 days and then grinded and polished. The sets of 5 samples were fired at temperatures 400, 425, 450, 475, 500, 550, 600, 650, 700, 730, 760, 790, 820, 850, 880, 910, 950, 1000, 1050, 1100, 1150, 1200 and 1250 °C. The heating and cooling rate was 5 °C/min without soaking at the highest temperature.

After firing, the samples were polished with sandpaper No. 800 using a rotating machine. The final dimensions of the samples were close to 3×4×20 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 an interval of 1000–1200 °C (Štubňa, 1997).

To make a precrack, an indentation was performed by hardness tester Zwick-Indtentec 5030 SKV in the middle of the 4×20 mm face using a load of 10–200 N and dwell time of 45 s. The diagonals of indentation were parallel with the sample sides. After that, the indentations were coloured with red dye penetrant ARDROX F6R for 10–60 min.

A fracture toughness test was performed on the Instron dynamic testing system 8516 under the loading rate of 10 N/s according to standard (ASTM C1421-10).

4. Results and Discussion

The dependence of the fracture toughness on the firing temperature is shown in Fig. 1. The standard deviation of K_{Ic} varies from 2 % to 18 %.

Similar graphs, as pictured in Fig. 2, were obtained for flexural strength and Young's modulus on samples made from a quartz porcelain mixture (50 wt. % of kaolin, 25 wt. % of feldspar, 25 wt. % of quartz) measured at the same conditions (Štubňa, 2010; Štubňa, 2011), see Fig. 2. Although the material composition of these samples is different, the processes in them and their consequences during heating are mainly determined by the reactions and structural changes in kaolinite (Norton, 1970; Hanykýř,

2000). Therefore, we can use the results presented in Fig. 2 for a 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. 1 and Fig. 2, so they correlate together.

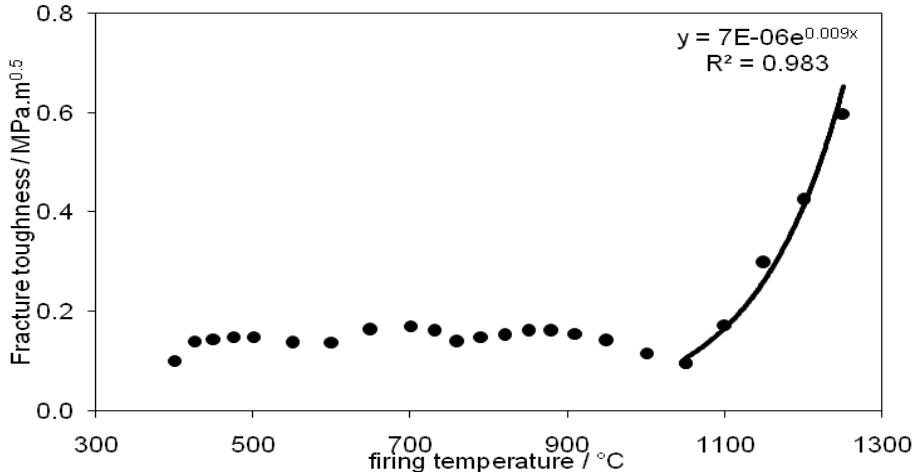

Fig. 1: The dependence of fracture toughness on firing temperature.

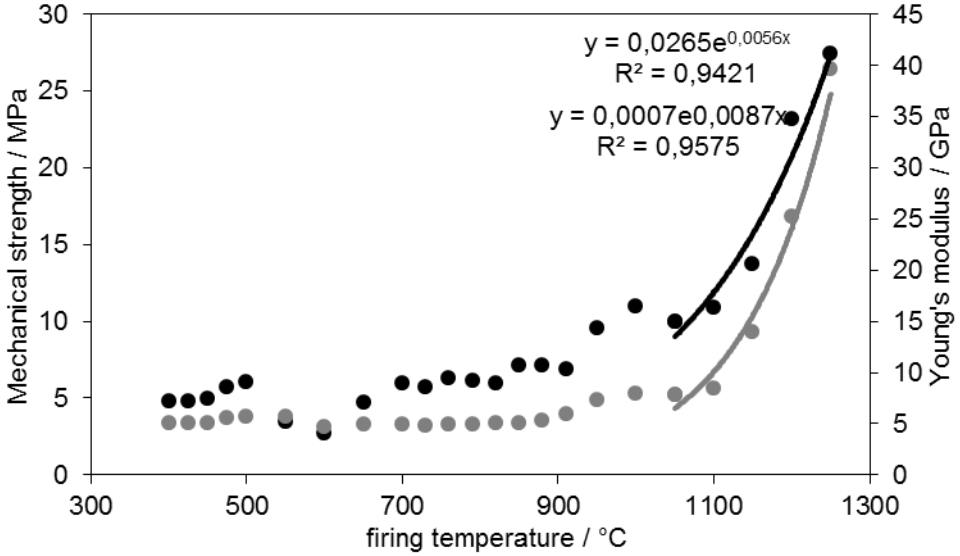

Fig. 2: The dependence of flexural strength (•) and Young's modulus (•) on firing temperature.

For the samples fired at 20–500 °C no changes in structure occurred at these low temperatures (Norton, 1970; Hanykýř, 2000), therefore the mechanical strength and Young's modulus, Fig. 2, are nearly constant in this range. The same is valid for the fracture toughness, as can be seen in Fig. 1.

For the samples fired at 500–700 °C, dehydroxylation, which weakens the kaolinite crystals, takes place. Metakaolinite created during dehydroxylation is a very porous material with internal vacancies (Freund, 1967) and lowers the mechanical properties of the samples. We observe a significant decrease of the mechanical properties within the temperature region of 500–700 °C, see Fig. 1, Fig. 2. We can assume that the kaolinite crystals are being rebuilt, so they become very defective during the dehydroxylation and this property remains even after cooling up to room temperature.

For samples fired at 1050–1250 °C, there is an exponential increase in the fracture toughness as well as the mechanical strength and Young's modulus increase dramatically. The solid-state sintering continues up to 1150 °C where feldspar begins to melt and a liquid-phase sintering starts. The samples contain a glassy phase, their density is higher, and this corresponds to higher values of the flexural strength measured at room temperature.

5. Conclusions

A development of the mechanical properties (fracture toughness, Young's modulus and flexural strength) of kaolin after the firing was experimentally studied at room temperature. It was found that:

1. Fracture toughness from 20 to 500 °C is almost constant and it varies between 0.1 and 0.2 $MPa \cdot m^{0.5}$.
2. Dehydroxylation has a small influence on the value of the fracture toughness.
3. In the temperature interval 1000–1250 °C, where intensive sintering takes place, we observe an exponential increase of fracture toughness.
4. From a comparison of the fracture toughness, Young's modulus and flexural strength follows a correlation and proporcionality.

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